

**CZECHOSLOVAK SPECTROSCOPIC SOCIETY
TASK GROUP OF INSTRUMENTAL RADIOANALYTICAL METHODS**

**CZECHOSLOVAK CHEMICAL SOCIETY
TASK GROUP OF NUCLEAR CHEMISTRY**

ABSTRACTS

**CONFERENCE
ON INSTRUMENTAL
ACTIVATION ANALYSIS**

IAA 92

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ABSTRACTS
CONFERENCE ON INSTRUMENTAL ACTIVATION ANALYSIS
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ASPECTS OF QUALITY PRACTICE AND ACCREDITATION FOR A NEUTRON ACTIVATION ANALYSIS LABORATORY IN A UNIVERSITY ENVIRONMENT

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During a relatively short period of time quality practice has become an accepted lifestyle in industry and in large technological institutions. Competition becomes stronger; the answer seems to be "quality". Ongoing from 1992, many governmental authorities in Western Europe will have their analyses by preference be performed by laboratories which have been accredited according to the international standards EuroNorm (EN) 45001 or the ISO guide 25. Accreditation of laboratories with standardized quality systems facilitate mutual comparability of the results. Moreover these quality systems imply a high degree of traceability.

There are several challenges for introducing a quality system into an university laboratory. When studying the aims of a quality system it turns out that the situation in an academic environment is almost the opposite: seldomly know-how is documented, even instructions for operating equipment are missing. Often long after experiments have been completed and results have been reported, sources of errors may be discovered directly affecting the results, such as improper or lacking calibrations, malfunctioning equipment, errors in computer programs. In universities there is always time to repeat things; seldomly there is time spend to take necessary precautions to do experiments correctly in the first place.

In addition, university workers often arrogated themselves such a high degree of self-respect that laboratories are advertised as "highly experienced". Although the basic expertise of the professionals is not questionable, results of laboratory intercomparisons by these "experienced" laboratories are sometimes not as good as what would be expected. This indicates that apparently there is an insufficient control of the analytical process and probably unknown sources of error exist.

Characteristic for a quality system is that a high degree of documentation is required. This refers not only to written procedures, working instructions and instructions for operation of equipment, but also to important decisions taken during the analysis.

In order to maintain and improve the position of a neutron activation analysis laboratory in the competition with commercial and industrial laboratories employing alternative techniques, accreditation will play an important, and maybe vital role in the forthcoming years. At the Interfaculty Reactor Institute at Delft, The Netherlands, the organization of the neutron activation analysis laboratory and associated analysis systems have been re-arranged according to the criteria mentioned above. The written procedures and instructions together with aspects of the organization, equipment manuals, instructions for archiving and complaint management have been compiled in a "quality manual".

In this contribution the special aspects of this quality system and its accreditation for a routine INAA laboratory in a university environment is presented.

INAA OF BORIDE CERAMICS MATERIALS

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The two main problems is necessary to solve in case of INAA of materials containing high concentration of boron. There are problems with heating of samples during neutron irradiation and high self-shielding of neutrons in the volume of samples. The dependence of boron concentration and sample density on self-shielding of thermal and epithermal neutrons, the intersample influence on self-shielding and selection of suitable neutron monitors were studied. The analysis Zr-B and Ti-B ceramics as an example will be discussed.

ACCREDITATION OF RADIOANALYTICAL LABORATORIES

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Accreditation of analytical laboratories has become rather actual in Czechoslovakia. Accreditation is a formal recognition that an analytical laboratory is competent to carry out specific tests or specific types of tests. Accreditation is normally awarded following successful laboratory assessment and is followed by appropriate monitoring. Accrediting body in Czechoslovakia is Federal Bureau for Normalization and Measurement (Dept. of Accreditation). This body has issued a "Handbook for Accreditation" MPA 001-91 containing a set of requirements that should be fulfilled by the analytical laboratory to be accredited. The handbook and related documents are very close to international standards like ISO/IEC GUIDE No.25.

Accreditation is a process consisting of a collection of information, establishing of an assessment commission, direct assessment in the candidate laboratory, evaluation of results of an assessment and finally, decision about accreditation. An accrediting body usually establishes a specialist group to advice in assessment of technical competence of laboratories.

The requirements for accreditation include such criteria as organization, control and staff in a laboratory, quality assurance system, audits, instrumentation, calibration, methods and procedures, protocols and documentation, participation in interlaboratory tests etc. All these criteria are discussed with respect to special features of radioanalytical laboratories. Laboratories with good organization and well-designed quality assurance system can be granted by accreditation without drastic changes in laboratory conditions and management used.

NEW MICROTRON IN OPERATION AT FNSPE CTU, PRAGUE

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In November 1990 first internal electron beam was accelerated in new microtron MT 25, which replaced the previous MT 22, for more than 10 years in exploitation in the microtron laboratory and dismantled in January 1990. The main parts of the new accelerator having been manufactured and tested already, the assembling could start immediately. The most significant difference between the old and the new microtron, designed and developed in close collaboration with JINR Dubna, consists in the omission of the acceleration chamber, the acceleration taking place between the pole pieces of the vacuum tight iron magnetic yoke with magnetization coils accomodated inside [1]. The major advantage of this so called chamberless microtron is the complete removal of all for checking inaccessible vacuum joints and in easy access to the acceleration space by lifting the roof of the magnetic yoke enabling at the same time an uncomplicated exchange of the only rubber sealing in the microtron if damaged by radiation. For complete description of the new microtron see [2]. On this place only the main features are mentioned.

A new adjustable high stability DC supply up to 400 A at 12 V was developed for the main magnet, alimented from 50 c/s distribution network, which eliminated the old one fed from a 400 c/s rotary convertor, left for alimentation of HF generator only. Another current supply of the DC pulsed type, less power consuming, is in final state of development.

The HF system from the old microtron was used, however seriously modified by matching the wave guide line to the european standard and substituting the ferrite isolator for a circulator. The matching of pulsed magnetron HF generator to the resonant acceleration cavity was improved considerably and operation stability increased as a result of these modifications.

In order that the output electron energy could be varied in large limits, a new extraction system was developed. Although based on the same principle as that in JINR, an entirely different solution of the driving mechanism for the telescopic iron output channel has been adopted, enabling

to withdraw electrons tangentially from the 8. to the 25. orbit. Thus, the energy can be varied in 0,6 MeV steps from 5 to 15 MeV or in steps of 1 MeV from 8 to 25 MeV with the use of two types of interchangeable resonant cavities.

In the initial period of operation 7 μ A internal target electron current from the 17. orbit was obtained immediately, which raised successively up to 17 μ A. These assembling of the external electron guide lines started in the first half of 1991. This transport system comprises two magnetic dipoles, one of them as an integral part of the internal extracting system and deflecting the beam from all selecteable orbits to a fixed direction, the second deflecting the beam to three experimental posts for electron, bremsstrahlung and neutron irradiations. The transverse dimensions of the beam passing through evacuated 50 mm ID tubes must remain within internal openings of all deflecting and focusing elements and are controlled by the main quadrupole doublet inserted between the two dipoles. Additional two quadrupole doublets are available for the control of the spot size and beam divergence on the target when placed on the final sections of the electron guides. Before having been installed, all deflecting or focusing elements were submitted to precise measurements of magnetic field distribution from which their optical parameters were calculated. The coincidence with design parameters was found to be satisfactory. The initial set of magnetic current values for all possible energies and beam paths was calculated by special program before starting the experiments with real beam. The initially supposed beam positioning system along the transport line had to be omitted because of the lack of funds in the last years. Thus, the tuning of the whole transport system became very difficult and one had to proceed by successive adding of individual components which proved to be a very tedious procedure, during which the microtron had to be filled with air and high vacuum restored again many times. The performance of the whole transport system in the most difficult configuration, represented by the 4 m total path length leading to the neutron irradiation post, with two deflections and 30 mm guide tube ID at the end, was tested with electrons from the 14. and 24. orbits. The currents in optical elements giving the best results were close to those calculated before.

An unexpected decrease of the electron current from higher orbits was observed probably due to some magnetic field inhomogeneities not supposed before. The real magnetic field configuration, especially the required

homogeneity could not be tested because the lack of a precise magnetometer. The field has to be corrected experimentally by correction coils, which again is a time consuming procedure.

Several converters have been prepared for measurements of neutron yield from gamma-n reactions in Pb, U, D₂O and in their combinations. The Pb and U converters are water cooled. Calculations of neutron yields from Pb and U converters in combination with D₂O or Be converters on the base of monochromatic cross sections data were performed as well, to enable a comparison with the results of the measurements, which started end of November.

References.

- [1] M.Vognar, A.G.Bělov, Č.Šimáně, V.N.Pokrovskij: Electromagnet for electron accelerator, CSFR Patent 239494, PV 5538-84.
- [2] Č.Šimáně, M.Vognar, R.Černý: Internal Research Report SPZV, III-9-1/08, FJFI CTU, 1991.

PROGRAM QA SUPPORTING QUALITY ASSURANCE LABORATORY SCHEME

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There is a continuous effort in our institute to have a professional QA computer code allowing to handle all sample results fast and precisely. The first prototype of such a complex algorithm was designed for gamma spectroscopic laboratory and IBM PC compatible computer. There are several procedures that have to be done simultaneously.

At first the actual results are given in numerous formats and all the data have to be converted into one standard data base structure so the question of the chosen data base standard arises first. In this first version designing special interfacing utilities for each system in use has been overcome.

The second part of the development of operating environment. The central data base is controlled by Microsoft multiuser relational data base system on the PC workstation. Some outputs are performed by far calls of other programs (for example, utility for drawing graphs). The next version will probably be driven by Microsoft, but more of the utilities will be done calling routines written in Turbo Pascal.

The hardware and software conditions allowing to run the QA program are following:

min. 530 KB free RAM memory, MS DOS 3.3 or higher, PC AT 286

The theoretical background is described in previous papers, some of them are listed below.

References:

- [1] L.Kolář, J.Obrusnik: Long Term Stability of Blank Sample Results in the Quality Assurance System of the NAA laboratory. Proc. Conf. IAA 89, Klučnice.
- [2] L.Kolář, J.Obrusnik, J.Faltejsek: Quality Assurance Program in the NAA Laboratory. Proc. Conf. IAA 87, Klučnice.

COMMERCIAL MARKETING OF INAA SERVICES

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At present, research reactor establishments and associated laboratories for instrumental neutron activation analysis (INAA) are developing strategies to justify the existence and continuation of their expensive facilities particularly when in an university environment. Part of such strategy might be to focus the potentials of INAA laboratories to problems of economical and social importance. Thus, not only the scientific and technical community of a country but rather the public as a

whole may benefit from the facilities.

Many laboratories deal with the difficulty that their research staff is hardly trained or experienced in sales promotion and marketing. Moreover, there are no textbooks on INAA or e.g. IAEA documents which can be used as a guide in this. "Selling" services requires understanding of commercial communication, how to do homework and what not to do. In addition, much is asked of the organization of an INAA-group, since in commercial services customers expect results in time. It is not a simple task for university workers to adapt themselves such a fully different lifestyle, but practice has shown that it can be done. At I.R.I. commercial INAA is now being carried out for many years. Presently, 4 analysts are involved in this together with a full-time administrative support. All salaries are fully accommodated for by the income from the analyses, whilst also investments in the equipment can be done. Each analyst has a capacity of appr. 1000 analyses per year. The service department has an active marketing policy.

When commercial applications for university facilities and expertise is foreseen, it is advisable to consider the set-up of a "business-plan". Such a business plan may comprise

- identification of customers
- collecting background information
- contacting customers
- cost analysis
- evaluation of competitors
- organization of work

This contribution outlines how to find opportunities for INAA services, and various aspects of a business-plan for INAA.

The methodology of INAA, the variety of materials for analysis, not the details of possible research programs will be discussed. Numerous examples of applications may be found in review articles such as the bibliography section of the Journal of Radioanalytical and Nuclear Chemistry, and the bi-annual review section "Nuclear and Radiochemical Analysis" of Analytical Chemistry. Additional information may be obtained from the proceedings of various topical conferences e.g. the bi-annual series "Environmental Contamination" or "Heavy Metals in the Environment".

ON DETECTION LIMITS IN RBS ANALYSES

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Rutherford back-scattering technique (RBS) based on elastic scattering of MeV charged particles is commonly used for non-destructive examination of thin surface layers [1]. Because of known Z^2 dependence of the elastic scattering cross section and relatively low background, the RBS method is especially suitable for the determination of heavy element contaminants on light substrates [1]. The RBS detection limit for particular element is adversely affected by underlying background caused by particle scattering on bulk sample atoms. In the case of heavy atoms spread on lighter substrate, the main background is a pile-up of much more frequent pulses from the sample bulk [2,3]. In this study, the effect of the particle channeling in monocrystalline backing on the pile-up background was examined with due regard for heavy element detection limits.

The samples under investigation were prepared by vacuum evaporating of minute amounts of different elements on standard (111) monocrystalline Si wafers. The RBS analyses were accomplished using 1.3 and 2.0 MeV α -particles from NPI electrostatic accelerator. The experiments were performed at 5×10^4 counting rates and the typical α -particle fluences were about 100 μC . Scattered α -particles were detected with windowless photodiode PIN (Hamamatsu S 3590-02) using Canberra electronic equipment (CI 2003BT, CI 2024, ND 581). The spectrometer energy resolution for α -particles was 12 keV (FWHM).

In the channeling geometry, the RBS yield from the of Si bulk atoms decreases by about two orders of magnitude in comparison with that of random geometry. This leads to corresponding decrease of the pile-up background in higher energy region and in turn to substantial improvement of the heavy atom detection limits. In the table, the real detection limits for three characteristic elements obtained in the channeling arrangements are compared with those achievable in standard random geometry:

Element	Detection limit ($\times 10^{12}$ at.cm ⁻²)	
	Random geometry	Channeling geometry
S	80	30
Zn	14	2.5
Pb	1.4	0.4

The improved detection limits are of interest not only for semiconductor technologists but they may be utilized also in another cases where the analysed substance can be deposited onto a monocrystalline backing.

- [1] W. Chu, J. W. Mayer, M. A. Nicolet, Backscattering Spectrometry, Academic Press, New York, 1978.
- [2] V. Hnatowicz, V. Havránek, J. Kvítek, Nucl. Instr. and Meth., B53 (1991) 337.
- [3] P. Oberschatsiek, V. Schule, R. Gunzler, M. Weiser, S. Kalbitzer, Nucl. Instr. and Meth., B45 (1990) 20.

ANALYTICAL POSSIBILITIES OF PIGE AT INP

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PIGE (Particle Induced Gamma-ray Emission) is a suitable method for the determination of light elements like F, B, Li, Na, Al, Mg, Si and P. In our laboratory at INP a new PIGE chamber has been developed as an attachment to the PIXE chamber. Intensity of the proton beam has been monitored by proton scattering from Ni foil and gamma-rays produced in a sample have been counted by means of a HPGe detector with a relative efficiency 30% and resolution (FWHM) 1.8 keV placed at 90° to the ion beam.

Elements contained in construction materials in chamber walls and apertures, especially light ones, could cause an enhancement of "chamber

background". Therefore, an alignment from thin (100 μm) Ni foil has been placed to inner walls of the PIGE chamber and also to the apertures originally made from copper. Background level has been decreased 30 times for Al gamma-rays and copper lines have been removed from the spectra completely. To diminish the influence of X-rays produced in the sample a 1 mm Cd foil has been placed between HPGe detector and PIGE chamber.

The PIGE setup has been tested on analyses of thick samples (tablets) of fly ash and coal. To determine fluorine, tablets prepared from reference fly ash (SRM 1633a) doped with known amount of F have been used as standards. Pure glucose has been used as a binding material. Gamma-ray spectra obtained in 10 min measurements have been evaluated by means of the PEAK program (Nuclear Data). Detection limits for 3 μC fluence of 3 MeV protons calculated according to Currie [1] are summarized in table 1.

Table 1

Detection limit of PIGE method for the determination of light elements in reference fly ash (SRM 1633a) and coal (SRM 1632a)

Element	F_{γ} (keV)	Detection limit (ppm)	
		fly ash	coal
F	110	80	40
F	197	40	15
F	6129	45	15
Na	440	30	70
Na	1635	300	240
Al	844	750	500
Al	1014	450	350
P	1266	350	200
Si	1273	1.4%	1.4%
Li	478	40	20
Mg	585	4500	2000

The detection limits are favourable for the determination of F, Al, Li and Na, in some types of samples other elements like Si and P could also be determined especially with the use of more energetic protons. Concentration of F found by PIGE in reference fly ash samples have been determined with inaccuracy in the range of 5-10% relative.

RECOIL IMPLANTATION OF RADON USED FOR CALIBRATION OF MONITORING EQUIPMENT

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The usual procedure for calibration of radon monitoring equipment is based on the introduction of a known amount of radon into the measuring chamber and on reading the response. For this purpose standard radium solution in a bubbler can be used. From this bubbler after a given time of accumulation, radon is transferred with a convenient carrier gas into the measuring chamber. In a similar way dry emanating radium sources can be used.

We suggest here a different procedure for such a calibration which is based on the introduction of a certain amount of radon into the surface of a tin alloy foil by exposing it in the vacuum in close proximity to a thin Ra 226 source. From this source Rn 222 atoms are ejected by recoil and implanted into the surface layer of the foil. The absolute activity of the injected radon can be measured e.g., with a silicon semiconductor detector. From implanted specimens alpha particles of Rn 222, Po 218 and Po 214 are emitted, which are clearly resolved in the spectrum, as can be seen in figure. This is because the median thickness of the depth distribution of the implanted activity is only $5.5 \mu\text{g}/\text{cm}^2$. After absolute determination of radon activity this gas can simply be released into the measuring chamber. This is achieved by melting the tin foil at 300°C in a small electric oven either directly in the measuring chamber or in a small auxiliary vessel, from which radon can be transferred into the chamber with a carrier gas. The advantage of this method consists in the possibility of basing the calibration data directly on the absolute

activity measurement of the introduced radon. Implanted foil specimens with a wide range of radon activities can be obtained from the same radon source (typically 100 kBq) by varying the implantation time exposure

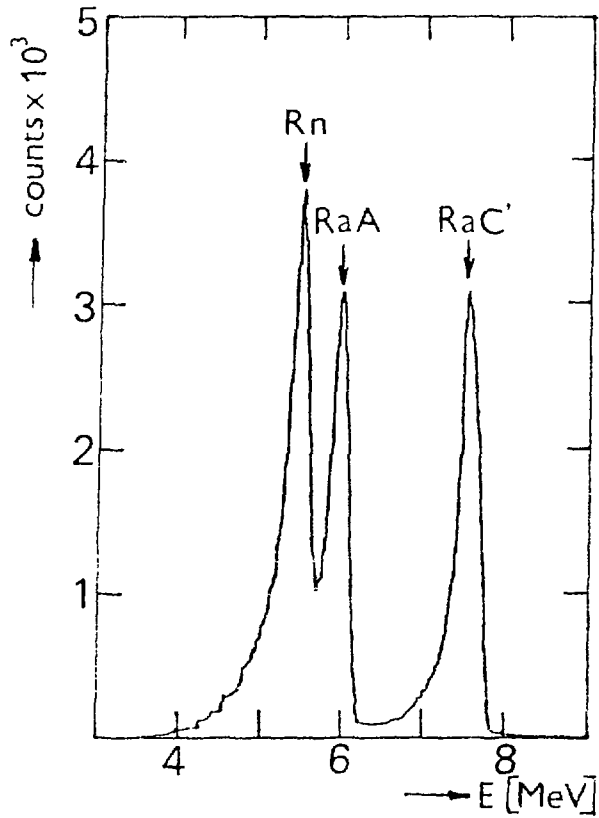


Figure: Spectrum from the alpha decays of Rn 222, Po 218 and Po 214 nuclei implanted into the tin foil.

IMPROVED EXPERIMENTAL ARRANGEMENT AND SEMI-ABSOLUTE CALIBRATION OF PIXE METHOD AT INP.

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The new experimental arrangement of the PIXE target chamber has been realized with the aim to reduce X-ray background and improve reproducibility of the results. Now all components of the sample carousel, including the sample frames, are made from plexiglass and defined geometry is ensured. The samples are kept on 12 μm thick mylar foil which was chosen for its low trace element content. In this way a considerable reduction of Zn, Fe and Cu background lines have been achieved. The collimation system and beam flux monitor were also rebuilt. The 200 nm thick gold beam diffusion foil is placed at the distance of 50 cm from the sample. The beam diameter is defined by a carbon collimator placed in front of the sample. The collimator outer surface is covered by amorphous gold layer and the protons backscattered on this layer are registered on a surface barrier detector. The number of registered protons is proportional to the proton fluence.

Semi-absolute calibration of PIXE method was performed using a set of thin single element standards. Part of this standards was prepared at INP and calibrated by RBS method. The measured X-ray yields of the thin standards have been employed for the determination of the free parameters of the Si(Li) detector efficiency curve, the thicknesses of additional absorbing layers between the sample and the detector and the thicknesses of the various filters used for the reduction of irrelevant spectrum regions. These data together with theoretical energy dependent X-ray emission cross sections, X-ray attenuation coefficients and semiempirical proton stopping powers enable us to determine the element content in the thin and thick samples with the accuracy of about 10%.

ACTIVATION OF SURFACE LAYERS OF SOLID BY ALPHA RECOILING RADIOACTIVE NUCLEI AND ITS USE FOR MATERIAL WEAR STUDIES

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A frequently used technique for studying the wear of materials consists of incorporating radioactive tracers into specimens and measuring activity removed by the wear process. In the solid specimens radiotracers can be induced, e.g. by neutron activation, or they can be implanted. A very simple technique for tracer implantation consists of using alpha recoil of conveniently selected nuclei from natural radioactive series. We have used recoil implantation of Rn-222 and its shortlived products in a study of dental wear in standardized tooth brushing simulation experiments with different tooth pastes. Atoms of Rn-222 and of its progeny were implanted into tooth specimens by exposing them in vacuum in close proximity to a thin Ra-226 source for 4 hours. Wear measurements were based on activity loss of solid short-lived radon decay products (rather than of Rn itself) - in spite of the necessity of taking into account the rapid activity decay. These corrections can be made by comparing the specimen activity decrease data to standard activity decay data recorded with an aluminium standard implanted simultaneously with the tooth specimen. Sensitivity of this implantation procedure in wear studies can be estimated from a calibration depth distribution of Rn-222 in aluminium, obtained with an electrochemical stripping technique, which is shown in Fig.1. As can be seen layer removal as low as $2 \mu\text{g}\cdot\text{cm}^{-2}$, which is about 7 nm in aluminium should be easily detectable. On the basis of these results the following hard tooth tissue wear studies were performed. The brushing machine represents a container, a carrier for a tooth specimen and a special moving carrier for a brush. The

carrier for the specimen is fastened to the bottom of the container which is filled with a dentifrice slurry of the given tooth paste. The moving carrier enables uniform motion of the bristles of the brush forward and backward (double stroke) over the specimen surface. Tension at each brush head is 220 grammes. After the chosen number of double strokes the motion is stopped, the specimen is removed, washed and dried with the help of filter paper. The activity of the specimen surface is measured before and after brushing. The activity of the standard Al-foil which is activated simultaneously with the specimen in the activating device is measured too and it is used for decay and implanted Rn activity corrections. A typical plot of the decrease of the activity of short-lived products in a tooth specimen (corrected for decay and implanted Rn activity) recorded during a simulated "tooth brushing" experiment is shown in Fig.2.

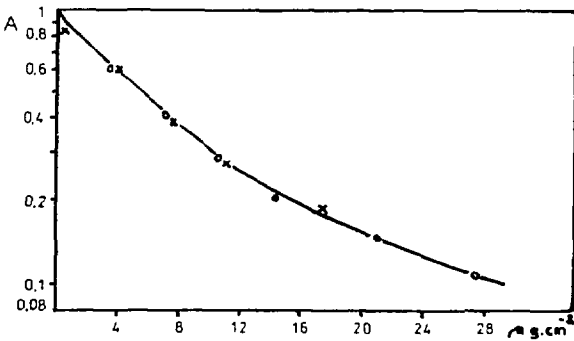


Fig.1
A - aluminium surface relative activity

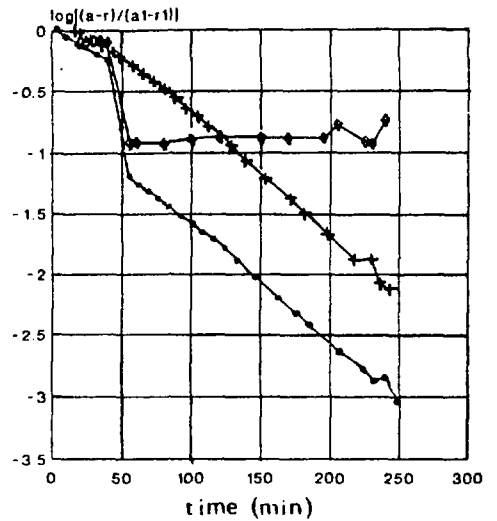


Fig.2
 -■- specimen + standard -○- standardized curves (upper curve before brushing lower curve after brushing) a-activity of the specimen, r = radon activity, a1,r1 initial activities

USE OF THE ANTI-COMPTON SPECTROMETER FOR THE SELECTIVE SUPPRESSION OF SOME ISOTOPES IN COMPLEX GAMMA-SPECTRA

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Analyzing gamma-spectra of complex radioactive specimens we, often meet a problem that we have to identify a very low intensity gamma radiation from one isotope on the background of a strong gamma radiation from another isotope. Placing the radioactive specimen inside an anti-Compton spectrometer we can, in special cases, suppress the radiation from the unwanted isotope.

The anti-Compton spectrometer consists of a central detector (as a rule a HPGe or Ge(Li) crystal) which is surrounded by a large scintillation detector (as a rule a NaI(Tl) or BGO crystal). Both detectors are connected by an anti-coincidence scheme.

In the conventional arrangement, the radioactive specimen is placed outside the spectrometer. In this case the Compton continuum and both the single and double escape peaks are suppressed, whereas the full energy peaks retain their intensity. Usually, the maximum suppression of 5 - 10 is reached for ^{60}Co .

There exists an alternative arrangement of the source-to-detector geometry, when the radioactive specimen is placed closely to the Ge-detector inside the scintillator. In this case, those parts of the gamma spectrum corresponding to the transitions being in cascades (including the full energy peaks) are suppressed in addition to the conventional suppression of the Compton continuum. In the case of a two-member full (100%) cascade, the magnitude of this additional suppression of one of the cascade members is given by the expression,

$$1/1-\varepsilon_2$$

where ε_2 is the absolute registration efficiency of the scintillator for the other cascade member. The resulting suppression is the product of the conventional and additional suppressions.

In the anti-Compton spectrometer of the Institute of Nuclear Physics in Řež, the maximum suppression of ^{60}Co is little more than 6-fold in the outer geometry now. The absolute registration efficiency of our scintilla-

tor for both the 1173 keV and 1332 keV gamma rays is about 80%. The maximum suppression of ^{60}Co in the inner geometry is as high as 34-fold. For ^{88}Y (898 keV - 1836 keV cascade), the suppression reaches the value of 40. For ^{22}Na (two annihilation gamma quanta in coincidence with 1275 keV), the suppression is up to about 100.

Non cascading transitions are not suppressed additionally. Hence, if we have to identify an isotope with non cascading radiation (e.g., ^{54}Mn , ^{58}Co , ^{65}Zn , ^{137}Cs , ^{197}Hg , ^{198}Au et al.) on the background of another isotope with cascading radiation (e.g., ^{22}Na , ^{56}Co , ^{60}Co , ^{75}Se , ^{88}Y , $^{108\text{m}}\text{Ag}$, ^{124}Sb , ^{133}Ba , $^{152,154}\text{Eu}$, $^{166\text{m}}\text{Ho}$ et al.), the use an anti-Compton spectrometer in the above described arrangement might be quite useful.

EXPLORATION OF RADIOACTIVE CONTAMINATION OF RIVER-BOTTOM SEDIMENTS IN BOHEMIA - PROJECT "LABE"

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Radioactive contamination of river-bottom sediments from the catchment area of the river Labe has been investigated in the framework of the research project "Labe". Both gamma- spectrometric measurements of medium sized (some 50 - 60 g batches) samples and the measurements of their total alfa and beta activities have been performed. Some 80 samples were analyzed in 1991, some 40 more are currently being analyzed. The network of sampling places covers homogeneously the catchment area of Labe river in Bohemia. The specific activities of both naturally occurring (^{226}Ra , ^{228}Ra , ^{228}Th , ^{40}K) and man-made (^{137}Cs , ^{134}Cs , ^{60}Co) radionuclides were determined. Based on the results of these analyses geochemical maps of radioactive contamination of river bottom sediments in Bohemia have been constructed.

Most of the sources of contamination turned out to be obvious and well known.

Generally, the radioactive contamination of river-bottom sediments by the main fall-out radionuclides ^{137}Cs and ^{134}Cs was found to follow their distribution in soils after the 1986 Chernobyl NPP accident. The contamination by ^{226}Ra and its daughters is mostly localized in the two well-known regions - Ploučnice and Ohře rivers. This contamination is the result of mining and milling of uranium ores in Hamr-region and of the effluents from the historical Jáchymov-region with its radioactive springs, respectively. A region of slightly enhanced contamination by ^{226}Ra has been found in southern Bohemia, its source has not been definitely determined yet. The distribution of ^{228}Ra and ^{228}Th is rather homogeneous over the catchment area investigated. The single spot of higher contamination found at Davle (Vltava river) will have to be at first verified, its possible source (mining and milling activities at Příbram) will be investigated later.

NEUTRON DEPTH PROFILING BY LARGE ANGLE COINCIDENCE SPECTROMETRY

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Neutron depth profiling (NDP) is one of the most powerful nondestructive techniques for the depth profiling of some light element in solids [1]. Two most important reactions $^6\text{Li}(n_{\text{th}}, ^4\text{He})^3\text{H}$ and $^{10}\text{B}(n_{\text{th}}, ^4\text{He})^7\text{Li}$ having thermal neutron cross sections of 940 and 3837 b respectively, are commonly employed for the determination of lithium and boron with practical detection limits of 10^{-6} in a standard experimental arrangement. The detection limits may be improved by 5-6 orders of magnitude using coincidence technique in which the energies of both charged reaction

products emitted in opposite directions are measured simultaneously [2].

In this work, the NDP coincidence device comprising two large area semiconductor detectors arranged in close geometry and two dimensional data handling was realized for the first time. In this experimental set-up, the particles emitted from the sample under different angles are registered simultaneously. The samples in the form of thin plastic foils were irradiated in the thermal neutron beam from 5.5 m long neutron guide (5×10^6 $n_{th} \text{ cm}^{-2} \cdot \text{s}^{-1}$). The neutron beam was collimated to the $0.5 \times 50 \text{ mm}^2$ cross section and the reaction products were registered by means of two $10 \times 10 \times 0.5 \text{ mm}^3$ PIN diodes (Hamamatsu S3590-06) placed at the distance of 1.5 mm from the sample. The effective solid angle subtended by each detector was $0.35 \times 4\pi \text{ sr}$ with respect to the sample centre. The detector signals were processed in two identical spectrometric chains producing also the timing pulses for a coincidence module (1 μs time resolution). The coinciding events were stored sequentially in a buffer memory of a PC AT computer and occasionally rewritten to floppy disks. The data evaluation comprising the relevant data selection and the transformation of the measured two-dimensional particle energy spectra into the depth profile of the analyzed atoms, was performed off-line on PC AT computer using specially designed software.

The feasibility of the device was checked using several thin standards prepared by deposition of small amounts of Li and B onto thin plastic foils. The data on real detection limits and the depth resolutions were obtained. The method can advantageously be used for the depth profiling of Li, B and some other elements present in minute concentrations.

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NEUTRON ACTIVATION ANALYSIS OF THE U.S. NIST SRM-1515 APPLE LEAVES AND SRM-1547 PEACH LEAVES

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Neutron activation analysis (NAA) has a prominent position in certification of element contents in reference materials (RMs) because of high potential for precision and accuracy of determination of many elements even at ultratrace level. Using both modes of NAA, i.e. instrumental NAA (INAA) and NAA with radiochemical separation (RNAA), 28 elements were determined in the new botanical NIST SRMs Apple and Peach Leaves which were analyzed in our laboratory in the frame of the ASTM nuclear methods intercomparison. Bowen's Kale and NIST SRM-1571 Orchard Leaves were analyzed as control samples.

For INAA, about 150 mg samples and synthetic multielement standards were packaged in polyethylene disks and irradiated in a thermal neutron fluence rate of $6 \times 10^{13} \text{ n.cm}^{-2}\text{s}^{-1}$ for 10 hours. Neutron flux monitors (iron disks weighing about 50 mg) were inserted between each four samples and/or standards in a column to be able to correct the axial neutron flux gradient. Gamma-spectrometry measurement of the samples and standards was carried out after decay times of 4 to 5 days and 1 month for 1 hour (counting geometry 7 cm) and for 2 hours (counting geometry 2 cm), respectively, using high efficiency and high resolution HPGe detectors. Great care was devoted to reproducible positioning of the samples and standards towards the detectors. The elements Ba, Br, Ca, Ce, Co, Cr, Cs, Eu, Fe, K, La, Lu, Na, Nd, Rb, Sc, Sm, Sr, Tb, Th, Yb, and Zn were determined by the INAA procedure.

For RNAA, the samples and synthetic standards were sealed in silica ampoules and irradiated as for INAA, but for 20 hours. Each vial carried an iron wire weighing about 35 mg which served as neutron flux monitor. Two RNAA procedures were used. The first consisted of sample decomposition after 3 days of a decay time in a mixture of $\text{H}_2\text{SO}_4 + \text{HNO}_3 + \text{HClO}_4$, addition of KI and ascorbic acid, and simultaneous extraction of the elements As(III), Cd, Cu, Mo, and Sb(III) by 0.025 mol/l zinc diethyldithiocarbamate ($\text{Zn}(\text{DDC})_2$) in CHCl_3 from 2-4 mol/l H_2SO_4 . The Cd-In pair was stripped in

equilibrium from the organic phase by 6 mol/l HCl. Separation yields exceeded 95% and were controlled by ^{77}As and ^{109}Cd radiotracers. The separated fractions were counted on a cap of the HPGe detectors for 1 hour. The second RNAA procedure for Hg and Se determination was based on sample decomposition after 14 days of a decay time in conc. HNO_3 in teflon lined steel bombs heated at 150°C for 4 hours, Hg extraction by 0.01 mol/l $\text{Ni}(\text{DDC})_2$ from 2 mol/l HNO_3 and subsequent precipitation of metallic Se by ascorbic acid from 1-2 mol/l HClO_4 . Separation yields exceeded 97% as found in tracer experiments. Counting times of the fractions amounted to 2-4 hours.

Excellent agreement was found between our results and certified and/or literature values for Bowen's Kale and Orchard Leaves RMs. More importantly, excellent agreement was also found between the majority of our values and intercomparison results and NIST certified values, the latter values being assumed to be close to the "true" values, for Apple and Peach Leaves SRMs which were analyzed as samples of unknown composition. The only problem concerned Cr determination by INAA due to poor counting statistics and Nd interference, unusual for botanical samples. Thus, it can be concluded that highly accurate INAA and RNAA procedures were developed in our laboratory which can compete in their reliability with those being used for certification of RMs in several leading analytical laboratories. Other data gained during the intercomparison and NIST certification process have indicated that the new Apple and Peach Leaves SRMs may very well turn out to be the most useful certified botanical RMs ever produced.

INVESTIGATION OF LEVELS OF SOME TRACE ELEMENTS MEASURED BY NAA IN BLOOD SERUM OF PATIENTS WITH THYROID DISEASES

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Essential trace elements play the role of effectors in most biochemical and physiological pathways of organism. They play their role in active reaction center, in allosteric center or as binding components of macro-

molecular complexes. Changes in metabolism of trace elements and their concentrations in various tissues and body fluids take place not only for supplementation of deficit elements, but also for diagnosis and control of medical treatment.

From this point of view we started investigation of levels of Zn, Se, Fe, Co, Rb, Cs, and Sc in blood serum of patients with thyroid diseases - carcinomas, adenomas, and hyperthyroidism. Blood was obtained immediately before thyroid surgery. Each thyroid was histologically evaluated after removal. Serum was obtained with the use of acid-washed polyethylene vessels and lyophilized. Trace elements were measured by the method of neutron activation analysis as described elsewhere [1]. Comparative values were obtained by the analysis of sera of 120 probands from the district of central Bohemia [1]. IAEA reference material II-4 (animal muscle) was used for quality assurance of NAA. Routine statistical methods were used for evaluation of results and comparative studies.

In the contrary to level changes of trace elements in thyroid tissue [2] there were only few significant changes in the levels of elements in the case of blood serum. The most dominant changes took place in the case of scandium, not only between healthy persons and patients but also among various groups of thyroid diseases. Changed levels were obtained also in cases of Zn, Cs, and Rb. As for Co, Fe, and Se there were nonsignificant or no differences.

These results were surprising mainly in the case of Se because of great differences in the content of Se in diseased thyroids [2] and Glatte et al. [3] published prediagnostic epidemiological study, which demonstrated higher possibility of thyroid malignancy in the cases of low content of Se in blood serum during the last 7 years prior to diagnosis of malignancy. Higher differences in Rb-levels were also expected because of great differences in the concentrations of this element among diseased thyroids [2].

As a result, from investigated elements only Sc seems to be different enough to serve as diagnostic tool, mainly in the group of malignancies. The changes of the other investigated elements are too small and for individual cases insignificant. The question is whether evaluation of whole spectrum of trace elements would not give us also some valuable information.

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STUDY OF AIR POLLUTION ORIGINATING FROM COAL COMBUSTION BY INAA
AND QUALITY ASSURANCE OF THE ANALYSES

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Air pollution monitoring is an important part of environmental studies in Czechoslovakia. It has been estimated that about 70% of solid emissions released into the air in Czechoslovakia originate from coal combustion. Instrumental neutron activation analysis (INAA) is a very suitable method for elemental analysis of solid emissions owing to sufficiently low determination limits for many elements, high potential for accuracy of element determination even at the nanogram level and below, and multielemental character which is especially valuable for receptor modelling.

Using both short- and long-time irradiation of samples and standards in a thermal neutron fluence rate up to $6.10^{13} \text{ n.cm}^{-2} \text{ s}^{-1}$ and subsequent

gamma-ray spectrometry with high efficiency and high resolution HPGe detectors, up to 36 elements were determined in fly ash and aerosol samples. Results have been utilized for studying the mechanism of pollution and to identify the types of pollution sources.

The large data sets on elemental composition of fly ash samples from several lignite-fired power plants from the Northern Bohemia and the Central Slovakia, and also of some aerosol samples, were used for evaluation of crustal enrichment factors. The enrichment factors (EFs) show that large quantities of several elements such as As, Br, Cs, Cu, Hg, In, Sb, and Se are released into the atmosphere from lignite combustion. Increase of EFs of some elements, namely As, Sb, and Se with decreasing size of fly ash particles was found by analysis of fly ash samples from electrostatic precipitator (ESP) hopper, ESP inlet, ESP outlet of a power plant, and of aerosols collected in the vicinity of the power plant. The mean composition of aerosols collected both in the vicinity of power plants (impact stations) and in rural, locally unpolluted regions (background stations) was also evaluated and origin of aerosols was studied by receptor modelling using factor analysis. For receptor modelling, the five basic types of emissions were considered: soil weathering, lignite combustion, oil combustion, road traffic, and marine aerosol. The highest relative contribution of lignite combustion to the aerosol composition in impact stations was demonstrated. The data obtained in this work were used in several international programmes on air pollution monitoring such as ECE EMEP, ECE HEMET, WMO BAPMON, etc.

Considering the high cost of pollution control and monitoring, great care was devoted to quality assurance (QA) of analysis to provide data of guaranteed and desired quality. Computerized QA system based on concurrent analysis of matrix-based reference materials (RMs) was employed. Of various fly ash RMs available, the NIST SRM 1633a Trace Elements in Coal Fly Ash and Czechoslovak RM IRANT ECH Coal Fly Ash were chosen as control samples for fly ash analysis. For aerosols, the NIST SRM-1648 Urban Particulate was analyzed as a control sample. Inference on accuracy of our results for aerosol samples can also be made from interlaboratory comparison on analysis of air particulate filters prepared recently within the IAEA Co-ordinated Research Programme "Use of Nuclear and Nuclear-Related Techniques in the Study of Environmental Pollution Associated with Solid Wastes".

DETERMINATION OF CADMIUM IN PLASTICS CONTAINING OF FLAME-RETARDING AGENTS BY INAA

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INAA of cadmium in the level of 50 ppm in plastics containing flame-retarding agents is problematic. High concentration of bromine (up to 10 %) or antimony (up to 0.16 %) respectively forms high ballast activity after the irradiation and especially fast determination of cadmium is complicated. The optimum way of cadmium determination was carried out and the following table brings protocol of analysis.

PROTOCOL OF INAA OF PLASTICS CONTAINING

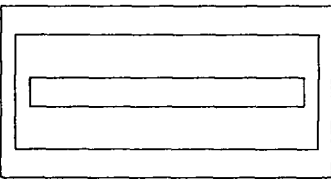
Limit of detection = 50 ppm

criterion of LD = $3 * \text{SQRT}(3 * \text{FWHM} * C)$

C - single channel content, FWHM - in channels

Detector ORTEC - Rel. efficiency 96.3%, FWHM 1.82 keV/1332 keV

Peak-to-Compton ratio 97.1 : 1

			FAST ANALYSIS	LONG ANALYSIS
				$t_{IRR} = 1 \text{ min.}$ counting 245 keV of ^{111m}Cd
P L A S T I C	NOT PVC	up to 1.4% Br	$t_D \leq 5 \text{ min.}$ $t_C \geq 1 \text{ hour}$	$t_D = 14\text{-}21 \text{ days}$ $t_C \geq 2 \text{ hours}$
		up to 10% Br	LD > 50 ppm	
	PVC	up to 0.2% Sb	$t_D \leq 5 \text{ min.}$ $t_C \geq 1.5 \text{ min.}$	$t_D \cong 14 \text{ days}$ $t_C \geq 1 \text{ hour}$
		up to 10% Br	LD > 50 ppm	$t_D \cong 14 \text{ days}$ $t_C \geq 2 \text{ hour}$
	up to 0.2% Br	LD > 50 ppm	$t_D \cong 7\text{-}14 \text{ days}$ $t_C \geq 1 \text{ hour}$	

t_D - decay time, t_C - counting time, t_{IRR} - irradiation time

USE OF INAA FOR TESTING OF SUITABILITY OF SOILS IN TŘEBOŇ REGION FOR PRODUCTION OF NUTRITIONALLY HARMLESS ALIMENTARY MATERIALS

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In consideration of a contract between the firm DEMETER (Germany) and a former state farm Třeboň the location of farm Chlum was chosen to produce harmless alimentary materials by rational biological farming, which is a less orthodox way of so called "alternative farming".

According to preliminary tests of some toxic substances in soils of this area (heavy metals, PCB pesticides residua) it is possible to say that the ecological conditions correspond with the demands of the contract. The farm manager asked the research centre of the Agricultural Faculty SBU to work out the optimal technologies of production. This Faculty employees considered the data insufficient and therefore they asked the Institute of Nuclear Physics in Řež for cooperation in the area of soil analysis with use of the neutron activation analysis.

In the area there were demarcated three squares (each 100 m^2) for samples collection. In each square 100 samples were taken in a square network of humus accumulated horizon of sod-forming soil (loamy-sand soil).

After the sample adaptation in laboratory the analyses of soil organic matter were made (humification grade, humic acids and fulvic acids, color quocient $Q_{4/6}$, functional groups analysis, and molecular weight of isolated humic acids and their infrared spectrometry). The sorptive-ion exchange characters (ion exchange capacity and selectivity coefficient for chosen model ion exchanges) were made too. The characters of soil biological activity were tested at the non-adapted samples (transpiration coefficients, test of cellulolysis, test of anonization, and test of nitrifying). The analyses were completed by the neutron activation analysis of siftings 0.1 mm soil samples - seven of each square.

For the reactor irradiation the samples of cca 200 mg weight were prepared and put into aluminium boxes. After activation the material was poured into welded PE bags and their actual weight was specified.

The irradiation passed for 6 hrs in the active core of reactor in Řež at neutron fluence rate $6 \times 10^{13} \text{ n.cm}^{-2}\text{s}^{-1}$. The gamma spectra were detected by HPGe detector with 22 % efficiency.

The above mentioned conditions of irradiation and measurements enabled to obtain for all samples the data of concentration of 29 elements as follows:

As, Ba, Br, Ce, Co, Cr, Cs, Eu, Fe, Ga, Hf, K, La, Lu, Na, Nd, Rb, Sb, Sc, Sm, Ta, Tb, Th, Tm, U, W, Yb, Zn, Zr.

On account of high uranium concentrations it was necessary to make significant corrections due to fission products which made quite impossible to determine e.g. Mo and Te and had strong effect on determination of Ce, Nd, and Zr.

The results showed that squares A and B have extremely high content of C_{ox} (5.5 - 8.04 %), high content of humic acids with low Q 4/6, for this soil type quite inadequate rate HA:FA (1.63 - 1.90), relative low humification level (18 - 25 %), and very high ion exchange capacity (32.6 - 49.5 mg.kv./100 g). The samples from both those squares have high content of some metals, especially uranium (in square B 14.9 - 51.9 mg.kg^{-1}). Here exists significant correlation of U with Br, Sc, and heavy rare earth elements as well as strong correlation with As, Ba, Co, Cr, Sb, W, Zn, and light rare earth elements.

The significant anticorrelation of U with potassium and sodium is interesting from the practical point of view. Uranium depends on Fe in two factors and there are two sources of uranium in tested soils. One of them is uranium from soil forming substrate (nearby are the uranium mines Včelnice). The second one is at squares A and B produced composts in which probably the sediments from uranium ore enrichment plant MAPE near České Budějovice were used by error.

The square C has normal values of C_{ox} (91.48 - 2.55 %) as well as other characteristics of soil humus and ion exchange processes. This square samples contain only 2.2 - 4.8 mg.kg^{-1} of uranium and the concentration of other evaluated elements is significantly lower too. It was found that no composts were produced and scarcely applied here.

Regarding to the values of elements, especially uranium, found by neutron activation analysis the use of locality Chlum is quite unsuitable for producing the alimentary harmless materials.

DETERMINATION OF CONTENT OF PHOSPHORUS AND SULPHUR IN WASHING POWDERS

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Compelling ecological aspects induce the public to use ecologically suitable products. One of the products examined from this viewpoint is washing powders. Fast assessment of ecological suitability of these powders was performed using an X-ray fluorescence method permitting a classification of the washing powder as to its content of phosphorus or sulphur, as well as a possible quantitative determination of these elements. Phosphorus and sulphur were assayed with the aid of energy-dispersive X-ray fluorescence spectrometer EDAX 9500 from Philips. Samples of washing powders from ordinary retail shops were not specially processed except for pelleting. Several phosphate-free imported washing powders were analyzed as controls. The calibration curve was determined through additions of the elements under study. Homogenization of the added components in the matrix was performed using a vibrating ball mill. Under these conditions the two elements can be assayed in concentrations higher than 0.1 % / w/w /.

XRF AND INAA STUDIES OF COMPOSITION OF PREHISTORIC BRONZES FROM BOHEMIA

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The knowledge of material composition is an important tool for archaeological investigations and it is layed an increasing attention to it. Some results from the early middle age were presented by us last year. At present, the main task is to study nonferrous metalurgy of the bronze

age (now supported by CAS grant), but analyses from other historical periods (e.g. celtic bronzes) are also made for the comparison.

Analytical methods used are XRFA (excitation source ^{241}Am) and INAA. More than 1000 artifacts were studied by means of XRF, about 200 of them by INAA, too. If the corrosion is not very strong, the area of several mm is polished to the metallic core to avoid the surface effects. In other cases only an approximate qualitative estimate of the composition is made. Following elements are being determined: Ag, Sb, As, Pb, Sn, Ni, Fe, Zn, Cu (with detection limits from 0.05% to 0.2%, but in many cases these limits are dependent on the overall composition, e.g., the interference of As and Pb).

After the XRFA, some of the artifacts are selected for INAA. Small samples (1 to 10 mg) were obtained by drilling. More reliable and sensitive results are then obtained for As, Sb, Ag, Ni and almost in all cases also for Co and Au. In many cases Fe, Se and Zn are determined. The trace determination is very difficult due to usually high contents of Sb and Ag.

The investigation is concerned mainly to artifacts from hoard finds, because they are related, they are from approximately the same time. We assume, that from deviations of compositions it will be possible to determine statistical criteria describing the most probable composition:

- a) for artifacts from one workshop (with the same shape and designation);
- b) for artifacts with different designation but in the same find;
- c) for artifacts between finds of the same region or age.

Derived regularities could be used as the basis for the proposition of evolutionary development in the composition of alloys.

From the preliminary results it is possible to derive following relatively important information:

- 1) Individual historical periods have some characteristic composition of bronzes with respect to the presence or absence of some of these elements: Sn, As, Ag, Sb, Pb and Ni, but the deviations may be very wide. It is possible to confirm that copper with high contents of As, Ag and Sb (up to several %) was widely used also in Bohemia in the early bronze age (similarly to [1]), and tin was relatively rare (the real bronze). However, in comparison with Gemeinlebern, up to now no high nickel content has been found in our analyses.

- 2) 38 ingots were also analysed (from Velvary, Svinárky, Třtěno, Rataje, Kšely, Klobuky and Plzeň-Jíkalka). Only in three of them the

content of tin was higher than 0.2%. This fact is a clear evidence, that, predominantly they are not products of remelting of old artifacts, but of primary copper raw materials, usually with admixtures of iron and nickel.

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RBS STUDY OF OXIDATION PROCESSES IN ION IMPLANTED POLYPROPYLENE

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Ion implantation of polymers has attracted growing attention for fundamental reasons and for potential technological applications as well [1]. By the ion implantation, electrical conductivity, optical properties and mechanical properties of polymers can effectively be changed. Ion implantation also leads to the molecular fragmentation, the crosslinking and the formation of unsaturated groups and free radicals.

The polypropylene (PP) foils implanted with I^+ and F^+ ions at the implantation energies 50, 100, 150 keV and 200 keV to the fluences of 10^{14} - 10^{15} cm^{-2} were examined using standard RBS technique. The samples were measured repeatedly in order to follow possible post-implantation changes. Meanwhile, the samples were kept in darkness, at room temperature on the air.

The surface of the PP samples implanted to higher doses was found to be heavily damaged and enriched in carbon. The fact that no F atoms were detected in the samples implanted with F^+ ions indicates the fluorine atoms are not bound in the substrate and they diffuse rapidly outside. On the contrary, the mobility of I atoms in PP is much lower. In repeated experiments, only mild diffusion of I atoms was observed with diffusion coefficient of $D=2.7 \times 10^{-18}$ $cm^2 \cdot s^{-1}$.

All implanted PP samples exhibit a strong oxidation proceeding from the sample surface to the bulk. The oxygen penetrates into radiation damaged

layer and it is fixed on radiation induced defects or chemically bound. No oxygen was detected on the pristine, unimplanted PP. The oxidation process cannot be described as a regular diffusion. In the samples implanted with F^+ ions, the oxidation proceeds steadily and a homogeneous oxidized layer is built up. The oxidized layer/pristine bulk interface is sharp and it moves to the sample interior with a mean velocity of about $4.0 \times 10^{-5} \text{ nm.s}^{-1}$. Curiously enough, the oxidation involves not only the radiation damaged layer but it proceeds in the same manner into pristine PP. So that, the radiation defects induced by the ion implantation seem only open the PP surface for oxidation.

Quite different oxidation dynamics was observed on the PP samples implanted with I^+ ions. The oxygen atoms penetrating through the sample surface are captured mainly in the narrow region of prevailing nuclear defects near the very end of the implanted ion trajectory. The measured oxygen content is proportional to the calculated number of vacancies (TRIM code) produced during the implantation and it exceeds the total number of implanted iodine atoms 20-40 times depending on the implantation energy.

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DETERMINATION OF CADMIUM IN PLASTICS BY XRF

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Cadmium is generally considered to be a hazardous element for man and his environment. In a rapidly increasing way governmental authorities are setting limits to the use of cadmium in plastics. Experiments in measuring of levels of cadmium in plastics were carried out utilizing radioisotope XFA as an analytical technique. A comparison of various excitation sources was carried out and an influence of matrix structure was studied. XRF appeared to be usable for fast determination of cadmium in plastic materials because of the absence of a destruction step, multielement results and an adequate sensitivity.

MODERN WAYS OF SPECTROMETRY - HARD AND SOFTWARE ASPECTS OF RADIATION DETECTION

Bernhardt Pögelt

Intertechnique Deutschland GmbH, Poing bei München, BRD

With more than 30 years experience as conceptor and manufacturer, in the field of nuclear detectors **INTERTECHNIQUE** has developed a very close cooperation with many research laboratories and industries.

INTERTECHNIQUE is a French company with a German daughter, the **INTERTECHNIQUE DEUTSCHLAND GmbH**, and includes special know-how, developments and technics for all things related to quantitative and qualitative analyzing of natural and artificial radiation.

INTERTECHNIQUE as thus remained one of the only industrial manufacturers of Si(Li) detectors for charged particle spectroscopy at room temperature and applied to research or industrial monitoring systems. The Si(Li) technology, where a thick volume is achieved by drift of lithium ions through silicon provides very rugged and reliable devices transmitted and improved over many years. A wide range of areas, thickness (up to several millimeters) and designs are available.

Today we have the longest experience - more than ten years - in the techniques of ion implanted passivated planar silicon detectors. We were the pioneers and the first (starting with registered name of **ENERTEC**, Lingolsheim near Strasbourg) to transfer the semiconductor technology to silicon, and later, to germanium detectors.

From the detection of γ and X-rays with high resolution we develop and produce high purity germanium detectors. Different applications will be shown to explain the wide range from standard configuration up to special designs like the **EUROBALL**, **EUROGAM**, etc.

Together with our special low level lead shields (activity < 3 Bq/kg) we are able to configure gamma spectrometry systems with extremely low background count rate:

Pb-210	46.5 keV:	< 1.0 counts/1000 s
Th-234	92.5 keV	< 2.0 counts/1000 s
U-235	185.7 keV	< 1.0 counts/1000 s
Cs-137	661.6 keV	< 0.1 counts/1000 s

Low level radioactivity measurements with germanium detectors are rapidly gaining popularity, particularly since the crystal size allows greater counting efficiencies to be reached. These measurements are required for environmental protection (reactors, nuclear waste storage, nuclear tests and health physics).

In september 1990 **INTERTECHNIQUE** and the **TENNELEC-NUCLEUS** Incorporation signed an agreement for international distribution. So we can offer also a wide spectrum of electronic units in NIM format and norms, data acquisition boards for PC germanium crystals and more.

A modern spectroscopy system for γ - as well as for α - and β -spectroscopy today is mostly PC-based. Using the data acquisition board which includes

- * 100 MHz Wilkinson ADC
- * up to 8192 channel memory (battery buffered)

we developed in Germany the family of our INTER... programs (INTERALPHA, INTERGAMMA, INTERPC, INTERWBC, INTERWINNER and special applications). Easy to handle we apply sophisticated methods to deconvolute peak areas, to separate different peaks which are close together in the range of detectors resolution but coming from different nuclides.

Especially in the field of alpha spectroscopy a good probe preparation is required. But not in any case it is necessary to apply a high resolution spectroscopy system. With a grid chamber for α spectrometry we can get an efficiency of 100 % for a 2- π -geometry without any preparation. Only the detectors resolution is lower.

In the field of environmental protection also the analysis of probes with an expected extremely low activity in the alpha and beta range is required. We can introduce a "Low activity and low background counting system" based on gas flow counters, lead shield, guard counters (for protection from cosmic rays and environment) and PC-controlled.

Both product groups - grid chambers and low-level-counters - are developed by the French **NUMELEC** company which is since more than one year a part of our **INTERTECHNIQUE** family.

This small overview should show the wide spectrum of possibilities to configure a spectroscopy or counting system for an also wide range of applications.

AN ADVANCED SPECTROSCOPY ACQUISITION AND ANALYSIS PLATFORM

Edward Fisher

Canberra/Packard International

I. Introduction

Traditional spectroscopy acquisition/analysis platforms are dedicated systems comprised of "front-end" electronics (e.g. detector, amplifier, ADC, etc.), multi-channel data acquisition modules, and a set of computer hardware and software components. Such spectroscopy computing systems are typically designed around an underlying operating environment (e.g. VMS, DOS), and are tailored for the specific acquisition hardware being controlled and spectral data being analyzed. In general, major modifications are required to this type of platform when new acquisition hardware is to be supported, data analysis criteria or reporting requirements change, etc.

By employing an advance, general-purpose architecture in the design of a spectroscopy acquisition and analysis platform, modifications and enhancements are easily implemented. This adds up to development cost savings in terms of resources applied and time spent as well increased capability and flexibility when tailoring systems for specific customer requirements.

II. Design Goals and Underlying Principles

The motivation behind this spectroscopy platform development effort was multi-dimensional.

- (1) A response was needed towards the industry trends towards instrumentation platforms based on open-system and/or industry standards in areas such as hardware platforms (e.g. Intel 80x86, RISC processors such as the R4000, VAX processor family); operating systems (e.g. OSF, POSIX/UNIX, DOS, OS/2); user interface environments (e.g. X/Motif, Windows, Presentation Manager) and networks (e.g. Ethernet, DECNet, Lan Manager, Novell NetWare).
- (2) The need for an underlying architecture which would provide the foundation for a future family of related, interoperable products.
- (3) The architecture must be designed so that its functions could easily be extended and ported as required.

To achieve the stated design goals, an object-oriented design approach

was used; this involved encapsulating "common" spectroscopy-related functions (e.g. spectral data access, hardware control commands, analysis algorithms) into a set of program objects which could then be used to build specific spectroscopy applications.

III. System Architecture Overview

The basic model for the system architecture is based on the client/server paradigm. This model allows an application to be partitioned into "client" and "server" components which can execute together in one computer or be distributed across computers over a network. An InterProcess Communication (IPC) subsystem provides the connective layer between the components. The IPC layer has been designed in such a manner that neither the client nor the server are aware of the underlying communication mechanisms employed. Note that the client and server components are depicted as "sitting on top" of the underlying operating system and user interface environments; this is to show that these components have been implemented to be as independent of the underlying system environment as possible.

RADIOACTIVE WASTE MEASUREMENT. THE MODERN TECHNIQUES

Jan Verplancke

Canberra/Packard International

The choice of the optimum instrument to measure nuclear waste is not an obvious task. The right choice depends on many variables, such as the types of isotopes to be measured, the matrix in which these isotopes are to be found, the detection limit and the accuracy wanted, the number of drums to be measured, etc...

Basically one can distinguish waste assay systems based on gamma measurements and systems based on neutron counting.

In the gamma based systems, Canberra offers complete, turnkey systems to characterise fission product waste (the Waste Assay System) and trans-uranic waste (the Segmented Gamma Scanner), as well as a system (the Q2 system) with which one can accurately define whether the waste contains

man-made activities or only naturally occurring activities. Examples of automation and customisation of these instruments will be given.

In a number of cases, transuranic waste can not accurately be measured with gamma measurements. For these cases, Canberra offers a variety of neutron counting systems, both using the passive neutron coincidence counting technique as well as the active neutron interrogation technique.

These techniques will be discussed in detail. Examples and results will be presented.

SAMPO 90

Gamma Spectroscopy Software

Features

- Complete Quantitative and Qualitative Analysis of Spectra from HpGe Detectors
- Automatic Operating Mode - Hands-off Operation for High Volume Sample Counting Labs
- Menu Driven Interactive Mode for Hands-On Operation
- Interactive Peak Fit Lets You Interact with the Analysis - and Verify Results
- Linear and Non-Linear Fit for Optimal Multiplet Deconvolution and Precision Fitting
- Analyze Extremely Complex Spectra with up to 32 Peaks per Multiplet
- Energy, Efficiency Calibrations
- High Low Tailing Calibration for Problem Detectors
- Sophisticated EGA/VGA Color Graphics
- Compatible with Canberra AccuSpec and System 100 Personal Computer Multichannel Analyzer Product Lines
- Ability to Read a Variety of Data File Formats
- Minimum Detectable Activity (MDA) Calculation and Reporting

Description

SAMPO 90 is a research grade Gamma Spectroscopy software package that is also highly capable for high volume sample counting labs. It provides exceptional analysis results on all HpGe spectra, even those with highly complex multiplets. The software is fully compatible with Canberra System 100 and AccuSpec personal computer based MCAs and with Canberra SATURN Systems.

SAMPO 90 is based on sophisticated SAMPO analysis algorithms and techniques, the standard for gamma ray spectroscopy for over 20 years. For the first time, SAMPO 90 brings these sophisticated capabilities to both the research and volume sample counting environments.

SAMPO 90 is intended for the spectroscopist who requires the maximum flexibility in the analysis of complex pulse-height spectra. Increasingly this includes routine, volume sample counting laboratories in applications such as environmental monitoring, radiochemistry and health physics, where accountability is important.

For most routine sample counting, SAMPO 90's automatic mode provides hands-off counting procedures. For those occasional samples where routine analysis isn't good enough, SAMPO 90 gives the spectroscopist the ability to interact with the analysis process via an interactive peak fit function. In this manner, the experienced spectroscopist can apply his judgement to the analysis - and back up that judgement with sound statistical results. For the researcher, this capability makes SAMPO 90 an essential tool. With interactive shape calibration, linear and non-linear peak fit and interactive display manipulation, SAMPO 90 is unsurpassed in its flexibility in performing complex gamma analysis.

Analysis and reporting features include background subtraction, automatic interference correction, weighted mean activity and MDA.

HISTORY

The name SAMPO 90 is derived from its early predecessor, SAMPO - Advanced Gamma Spectrum Analysis Software - originally developed in 1969 for the mainframe computers at Lawrence Berkeley Laboratory and at CERN, the European Nuclear Research Center. SAMPO has been top rated in international comparisons and has been acknowledged by the International Atomic Energy Association (IAEA) as being the best gamma spectroscopy program available.

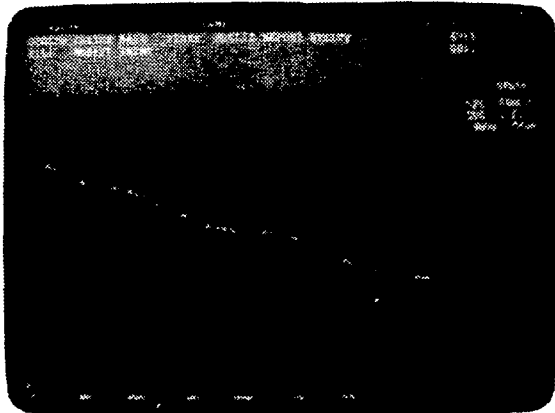


Figure 1 - Full Spectrum Display.

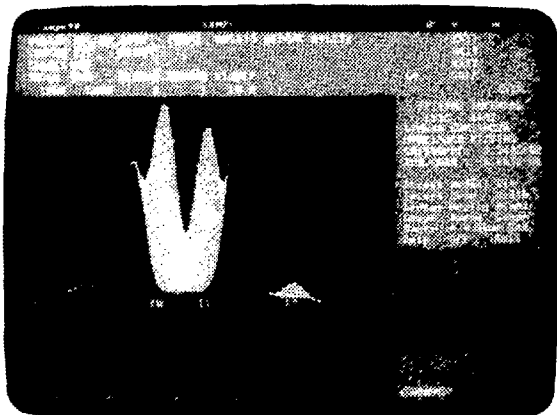


Figure 2 - Multiplet Deconvolution Screen Showing Residual Graphs.

SAMPO 90 is an enhanced personal computer gamma spectroscopy program that utilizes the core algorithms of SAMPO, but has been extensively redesigned to incorporate user-friendly menus, interactive color graphics, automation for production operations and additional features.

AUTOMATING PRODUCTION SAMPLE COUNTING

SAMPO 90 is a research grade spectroscopy tool that is also highly capable for high volume, routine sample counting operations in radiochemistry, health physics and environmental monitoring. Such facilities require "hands-off" counting capabilities - fully automated data collection, analysis and reporting procedures that can be executed with little or no operator intervention.

WHAT IS IT INTERTECHNIQUE?

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That means development and manufacturing of:

- * High purity Germanium detectors
 - n-type, p-type, planar, coaxial
 - standard and special windows and mechanics
 - low level material
 - standard and special cryostats
- * Ion implanted Si-detectors
 - standard configuration
 - strip detectors, position sensitive
- * Lithium drifted Si-detectors for X-ray or beta spectroscopy
- * Lead shields in standard and low level configuration
- * NIM-electronic, also products from the TENNELEC-NUCLEUS-Inc. (Oxford Instruments)
- * User friendly software for measuring control and spectra analyzing
- * Low-level α - β -counting systems
- * Alpha grid chambers

THAT'S INTERTECHNIQUE

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**If you have any questions or we can do something for you,
please don't hesitate to contact us in Germany.**

7423/UHS-S

16K ULTRA HIGH-SPEED SPECTROSCOPY ANALOG TO-DIGITAL CONVERTER

- 16K ADC
- 4 μ S conversion time
- Integral non-linearity
 $\leq 0,02\%$ over the top 99,8% of range
- Differential non-linearity
 $\leq \pm 0,3\%$
over the top 99,8% of range

DESCRIPTION

The SILENA Mod. 7423/UHS-S is derived from the successful Mod. 7423/UHS ADC of proven use with hundreds of units sold and operating all over the world.

The SILENA Mod. 7423/UHS-S is an ultra high-speed, highly reliable, digitally corrected, subranging analog-to-digital converter.

The excellent differential linearity characteristics of the ADC result from the incorporation of a special circuit that minimizes the ADC differential non linearity and from the use of the sliding scale method devised by Prof. Emilio Gatti (Nuclear Instruments and Methods Vol. 24, p. 241 (1963), patented in Europe and U.S.).

SILENA's exclusive design eliminates all undesirable effects (peak broadening, etc.) due to ageing effects in the DAC used for the sliding scale corrected ADC.

The Mod. 7423/UHS-S makes an extensive use of integrated circuits: only a few discrete components and no hybrid microcircuits are employed.

Utilizing new advanced concepts, the Mod. 7423/UHS-S achieves an exceptional level of performance and reliability, combined with easy maintenance and servicing.

The Mod. 7423/UHS-S is designed for use with SILENA systems. However, because of its modular design, it can be easily used with any existing data processing systems, and in particular with digital computers.



SILENA
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