ENEEP Member: The Slovak University of Technology Bratislava

Synthesis of implementation workflow and conditions for novel activities

February 2024

Contents

1. Administrative procedure

The STU administrative procedure valid for the implementation of new experimental activities at the laboratories of the Institute of Nuclear and Physical Engineering (INPE) reflects the available experimental equipment and is based on the work description provided by the interested party and a simple approval process.

The applicant is required to prepare a detailed description of the proposed experiment in collaboration with the head of the specific laboratory. This description must include all relevant technical data, a list of instrumentation, the measurement methodology, an estimate of the radiation exposure of the personnel, and relevant information from the literature review.

The work description is reviewed by the Radiation Protection Officer and, if approved, is then formally authorized by the director of INPE.

2. Operating limits and conditions for experiments

Activities carried out during normal operation in all laboratories have to be in accordance with the national legislation, explicitly with the Law 87/2018 on Radiation Protection and Decree 99/2018 of the Ministry of Health of the Slovak Republic on Ensuring Radiation Protection.

Utilization of new sources of ionizing radiation or radioactive specimens, which are not in the evidence of INPE laboratories, is allowed but only for the time necessary to carry out the planned experiments in laboratories located in the Controlled Area. Use of sources or specimens must be supported by the documentation, which includes the name of the owner, the activity and the certificate of the long-term stability, integrity or tightness. All sources or specimens have to be transported and stored in transport containers or experimental configurations, in which the rate of the ambient dose equivalent is smaller than 0.0025 mSv/h on the surface of the equipment.

Manipulation with the sources and radioactive specimens without transportation container or direct shielding is permitted only for the time necessary to carry out the planned experiments. Measures to minimize radiation exposure of the workers in the specific laboratory, adjacent laboratories and the vicinity of the controlled area must be taken and have to be documented in the support documentation of the experiment.

3. Technical data

The Institute of the Nuclear and Physical Engineering as one of the leading institutes of the Faculty of Electrical Engineering and Information Technology of STU has been active in performing non-destructive material testing, development of detectors, radiation shielding and computer simulation for several decades. Its laboratories located in their premises can be divided to ones for experimental science and numerical simulations. The most important laboratories in terms of ENEEP are the Laboratory of Reactor Physics, the Laboratory of Mössbauer Spectrometry, the Material Science Laboratory, the Laboratory of detector development, the Laboratory of dosimetry and radiation protection as well as the Supercomputer Applications Centre.

3.1. Laboratory of Reactor Physics

The laboratory of Reactor Physics consists of experimental workspaces for neutron emission rate, neutron diffusion length and Fermi age measurements with Pu-Be, Am-Be and Cf-252 neutron sources and apparatus for remote control and monitoring of experiments. Currently, a new "Labyrinth" experiment is being prepared for testing of materials relevant to deep geological repository of radioactive waste and spent nuclear fuel. The laboratory is equipped with a shielded source loading mechanism and IoT based monitoring grid, to minimize the doses obtained by the personnel and to be able to monitor the conditions of experiments from remote workplaces.

Features:

- measurement of neutron emission rate,
- measurement of neutron diffusion length and Fermi age,
- evaluation of shielding materials in Labyrinth,
- defectoscopy measurements,
- plastic nuclear track detectors, evaluated using AI,
- remote source handling,
- IoT radiation monitoring grid,
- crane to handle heavy loads.

3.1..1 Characteristics of the neutron emission rate measurement workplace

Available sources:

- Pu-Be ($1.1 \cdot 10^7$ n/s 4π),
- Pu-Be ($5.1 \cdot 10^5$ n/s 4π),
- Am-Be ($6.3 \cdot 10^6$ n/s 4π),
- Cf-252($5 \cdot 10^6$ n/s 4π).

Specifics:

- vessel with $MnSO_2 \cdot H_2O$,
- outer measurement circuit,
- manganese evaporation workplace,
- IoT solutions,
- remote measurement control,
- shielded neutron source loading,
- simulation model.

Detection

- 2.5“ HPGe,
- 3.5“ NaiTl (AmpTek).

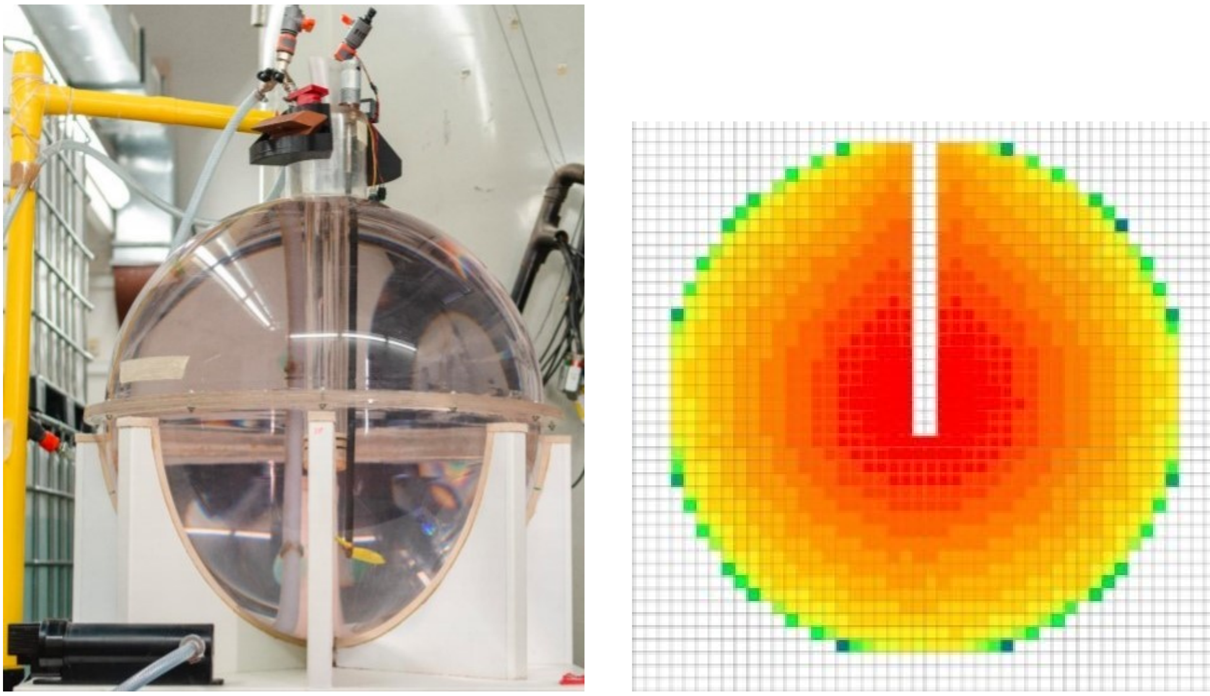


Figure 1: Neutron emission rate measurement at STU.

3.1..2 Characteristics of the neutron diffusion length and Fermi age measurement workplace

Available sources:

- Pu-Be ($5 \cdot 10^7$ n/s 4π),
- Pu-Be ($5 \cdot 10^5$ n/s 4π),
- Am-Be ($2 \cdot 10^5$ n/s 4π),
- Cf-252($5 \cdot 10^6$ n/s 4π).

Equipment:

- vessel for liquid moderators,
- various measurement positions,
- measurement in liquid or gaseous state of the moderator,
- use of soluble absorbers (H_3BO_3),
- temperature range: 20°C – 70°C,
- steam generator,
- IoT solutions,
- remote measurement control,
- shielded neutron source loading,
- simulation model.

Detection

- He-3 filled proportional neutron detector,
- activation foils (In, Cd, Ag, Au).

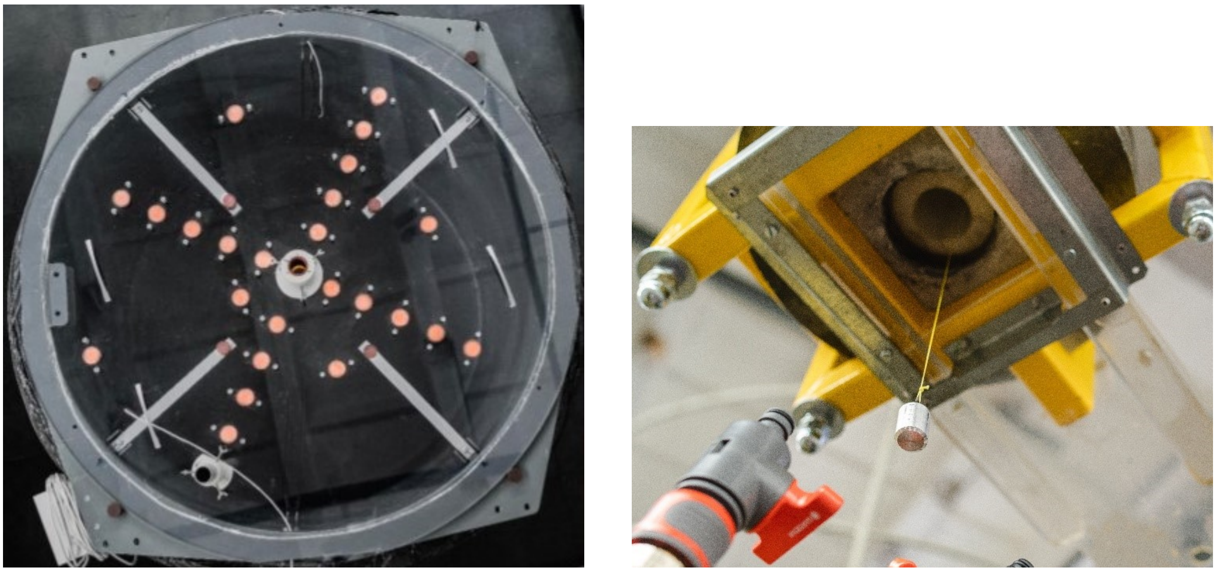


Figure 2: Neutron diffusion length and Fermi age measurement at STU.

3.1..3 Characteristics of the mini labyrinth workplace

Available sources:

- Pu-Be ($5 \cdot 10^7$ n/s 4π),
- Pu-Be ($5 \cdot 10^5$ n/s 4π),
- Am-Be ($2 \cdot 10^5$ n/s 4π),
- Cf-252($5 \cdot 10^6$ n/s 4π).

Equipment:

- labyrinth made from NETRONSTOP C5 shielding blocks,
- graphite or liquid moderators,
- adjustable size and position,
- measurement inside or outside the Labyrinth,
- IoT solutions,
- remote measurement control,
- shielded neutron source loading,
- simulation model.

Detection

- RadEye survey meter,
- NUDET neutron detector,
- He-3 filled proportional neutron detector.

Figure 3: The mini labyrinth experiment at STU.

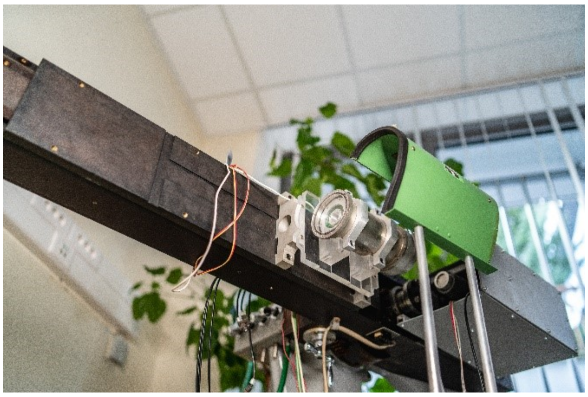
3.2. Laboratory of Mössbauer Spectrometry

Mössbauer spectrometry (MS) is a non-destructive method with a wide diagnostic potential, applicable to all materials containing iron. It allows performing accurate phase analysis of materials, as well as identification of the iron atom in various crystallographic positions. Mössbauer spectrometry can be applied in the field of studying the structure of metallic materials, alloys, steels, and magnetic materials in the study of the magnetic structure.

The application of MS in the field of mineralogy, geology, corrosion products, and materials for magnetic recording provides possibilities for accurate identification of iron oxides. It is also suitable for the analysis of amorphous non-crystalline substances.

INPE has experience with the analysis of samples from the fields of archaeology, biology, chemistry, thin films, and mineralogy including meteorites. It is not difficult to prepare samples for the MS measurements. For normal analytical purposes, a thin film or a few grams of powder sample is sufficient. It is possible to perform measurements on large samples.

The laboratory includes a Mössbauer spectrometer, which measures in transmission geometry (TMS = Transmission Mössbauer Spectrometry) and is also able to record conversion electrons (CEMS = Conversion Electron Mössbauer Spectrometry) or conversion characteristic radiation (CXMS = Conversion X-ray Mössbauer Spectrometry). TMS provides information on the volume of the examined sample, CEMS and CXMS scan the subsurface layers to a depth 200 nm and 5000 nm. At the same time, one can obtain information about the internal volume and surface states of the examined sample.



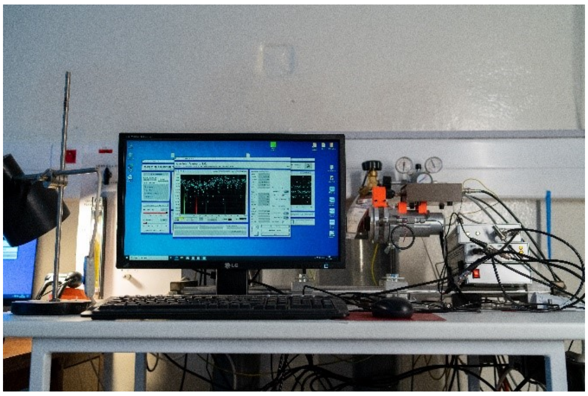


Figure 4: Mössbauer spectrometry at STU.

3.2..1 Characteristics of instruments at the INPE Laboratory of Mössbauer Spectrometry

Mössbauer spectrometer
Technical information

- Gamma ray detector with electronics (YAP scintillator, proportional counter)
- Data acquisition module

- Mössbauer velocity transducer with function generator
- Gamma-ray source
- Evaluation software

Features and possible applications

- The current configuration of the Mössbauer spectrometer can be used in transmission or backscattering geometry.
- In addition, the techniques of Conversion Electron Mössbauer Spectrometry (CEMS) and Conversion X-ray Mössbauer Spectrometry (CXMS) are also available.
- They scan surfaces of the investigated samples to the depth of $\sim$ 200 nm and 5000 nm, respectively.
- All experiments can be currently performed only at room temperature.
- The recorded spectra are evaluated by suitable evaluation software, their interpretation, however, requires experienced personnel.
- An inevitable condition for Mössbauer spectrometry experiments is a presence of the individual Mössbauer nuclides, e.g. Fe or Sn, in the investigated samples.

Mössbauer source
Technical information

- Type Co-57/Rh
  - 14.41 keV photon emission efficiency: $\geq$ 75 %
  - ISO classification: C54243
  - Temperature range: 4.2–700 K
- Type Sn-119m
  - 23.87 keV photon emission efficiency: $\geq$ 75 %
  - ISO classification: C54243
  - Temperature range: 77–700 K

Features and possible applications

- Cobalt type Mössbauer source is prepared by electrodepositing high purity carrier-free Co-57 onto a thin metal backing (matrix), which is rhodium in our case.
- Tin type Mössbauer source active part is based on calcium stannate [$CaSnO_3$] matrix synthesized from high specific activity (> 300 mCi/g) Sn-119m radionuclide. Active part is placed into titanium alloy holder with brazed beryllium window and sealed with laser welding.

3.3. Material Science Laboratory

Material Science Laboratory is equipped with several instruments which are listed and described in following tables. The aim of the laboratory is to provide non-destructive methods for analysis and comprehensive investigation of progressive materials. Instruments utilize nuclear methods like positron annihilation spectroscopy, x-ray spectrometry and nanoscale microscopy. An integral part of the laboratory is also instrumentation for sample preparation which includes optical microscopes, nanoindenter, high temperature furnace, plasma cleaner, rotary coater, precise saw grinder and polisher.

Figure 5: Material science laboratory at STU.

3.3..1 Characteristics of instruments for Positron Annihilation Spectroscopy

Positron annihilation lifetime spectroscopy
Technical information:

- Equipment
  - Positron source Na-22
  - 2x NaITl detectors Hamamatsu H3378 and 1x NaITl detector Scionix equipped Photonis XP2020Q PMT,
  - 2x HV supply Ortec 566
  - Digitizer Evm DRs4
  - PC
- Measurement time approx. 8 hours
- FWHM < 170 ps
- Positron life-time measurement up to 150 ns

Features and possible applications:

- Measurement of the positron life-time since emission till annihilation in investigated material (metal based alloys).

- Method gives information about size and intensity of the defects.
- Three detector configuration allows application of the method on alloys contained isotope Cobalt-60.
- Maximal dimension of the sample is 10 × 10 mm square shape with 1 mm thickens.
- For one measurement it is necessary to have two identical samples.

Coincidence Doppler Broadening Spectroscopy
Technical information:

- Equipment
  - Positron source Na-22
  - 2x HPGe detectors Canberra GC0519 with iPA preamplifier,
  - 2x HW supply Canberra 3125, NIM Bin
  - 14bit Digitizer Adlink PCI-9820
  - PC
- Measurement time approx. 24 hours
- FWHM < 1 keV

Features and possible applications:

- The methods give information about the momentum of the electron-positron pair in the process of annihilation.
- Based on the ratio of the annihilations of free electrons to bound electrons, it is possible to quantify the concentration of the vacancies and chemical composition of the sample.
- The maximal dimension of the sample is 10 × 10 mm square shape with 1 mm thickness.
- For one measurement it is necessary to have two identical samples.

3.3..2 Characteristics of instruments for material structure analysis

X-ray diffractometry
Technical information:

- D8 ADVANCE (Bruker).
- Co and Cu anodes with output power 2.2 kW.
- Equipped by goniometer with scan range of 360°.
- Applicable Bragg-Brentano or GIXRD configuration
- X-ray reflectometry

Features and possible applications:

- Qualitative and quantitative phase analysis of the samples.
- Crystallography analysis applicable for residual stress investigation.
- Extreme variability in the size of samples – from very small and thin up to bulky samples (1kg or several cm in diameter)
- Investigation of the sample structure as a function of the depth up to several µm.
- In case of thin layers development in electronics (sensors, batteries, solar cells, ...) the quality assessment of the layers can be carried out by X-ray reflectometry.

X-ray fluorescence spectrometry
Technical information:

- Mini X-ray source + X-123 spectrometer (AMPTEK)
  - Ag target, voltage 10-40 kV, current 5 – 200 μA; Be window with thickness 500 μm;
  - Si-PIN detector with detection area 6 mm^2, thermoelectric cooling and resolution 149 eV to 5.9 keV,
- Measurement time from second to minutes.
- Shielding box
- Defined geometry
- PC with evaluation software – MCA and XRS-FP quantitative analysis

Features and possible applications:

- Qualitative and quantitative analysis of sample composition (reliable identification of the elements with atomic number higher than 11).
- Compact geometry and simple sample preparation make the device applicable for in-situ measurements (industry, glass, ceramics, historical artefacts, biological/environmental samples, meteorites).
- The detection limit is approx. 10 ppm.
- Currently, the measurements are carried out in air environment, in vacuum or Helium atmosphere.
- The range of elements identification can be increased up to elements with atomic number 5 and less.

3.3..3 Characteristics of instruments for nanoscale microscopy

Atomic Force Microscopy
Technical information:

- AFM Veeco - Dimension Edge.
- Maximal scanning area 100 $\times$ 100 μm
- In z-dimension atomic resolution.
- The surface of the sample has to be flat, smooth, clean, dry and the roughness should be less than 1 μm.
- AFM is sensitive to dust, noise, air stream and temperature change.

Features and possible applications:

- Investigation of the magnetic domain on the surface of magnetic samples.
- Extreme spatial resolution in the z-dimension and ability to measure height and area of the surface.
- Resolution in z-dimension is less than 0.1 nm and in xy-dimensions is less than 1 nm.
- Applicable to amorphous and nanocrystalline alloys, thin ferromagnetic layers, soft materials like polymers (OLED displays), organic transistors, photonic crystals, polymer films.

Scanning Electron Microscope
Technical information:

- Hitachi SU3500
- Two modes of operation:
  - Secondary electrons magnification
    - Resolution 3 nm at 30 kV accelerating voltage.
    - Resolution 7 nm at 3 kV accelerating voltage.
  - Backscatter electrons magnification
    - Resolution 4 nm at 30 kV accelerating voltage.
    - Resolution 10 nm at 5 kV accelerating voltage.

Features and possible applications:

- Chemical analysis of the sample.
- Phase identification and separation.
- Strain analysis and grain boundaries characterizations.

3.4. Laboratory of detector development

The laboratory is focused on the complex development of semi-conductor detectors and measurement of the semi-conductor detector performance characteristics. The developed detectors are made from GaAs, SiC and Si materials. The laboratory of chemistry located at INPE is used for the preparation and construction of new detectors in the form of thin films on appropriate substances. The application of the new detector can be various and can be affected by the dimensions at which the detector is developed. The detectors can be used for the detection of alpha and beta particles, or even neutrons, if additional conversion layer is placed on the top of the detector. Therefore, these detectors with proper hardware and software solutions (in our case from the DVACAM company), can be also used as radiation cameras. The particular areas of their application can be non-destructive testing of samples, spectral imaging, mining and geology, x-ray diffraction and others. Currently, a new passive detector measurement technique is being developed at INPE. This technique utilizes passive polymer CR-39 detectors. After calibration, the CR-39 detectors can be used as additional measurement technique for estimation of dose at different shielding experiment and other relevant applications. To evaluate the tracks in the detector artificial intelligence and machine learning principles can be used.

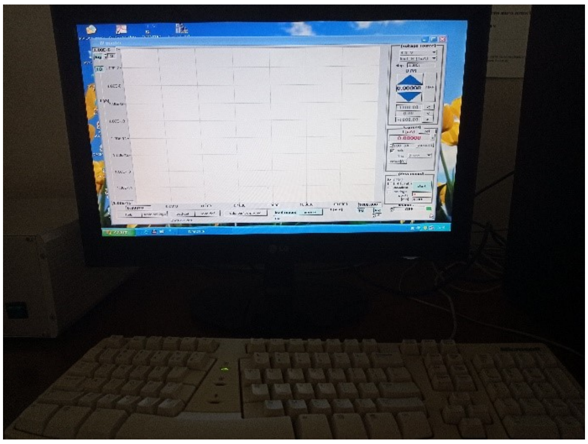


Figure 6: STU Laboratory of detector development.

3.4..1 Characteristics of the GaAs, SiC and Si detectors development

Measurement circuit of volt-ampere characteristic of semiconductors
Technical information:

- In-house developed circuit with unique software for automatic measurement of the volt-ampere characteristics
- Volt-meter and ampere-meter measurement of the volt-ampere characteristic
- Automatized analyser of measured data

Features and possible applications:

- The operation voltage is in the range of ±1000 V.

Figure 7: STU CR-39 detector measurement technique.

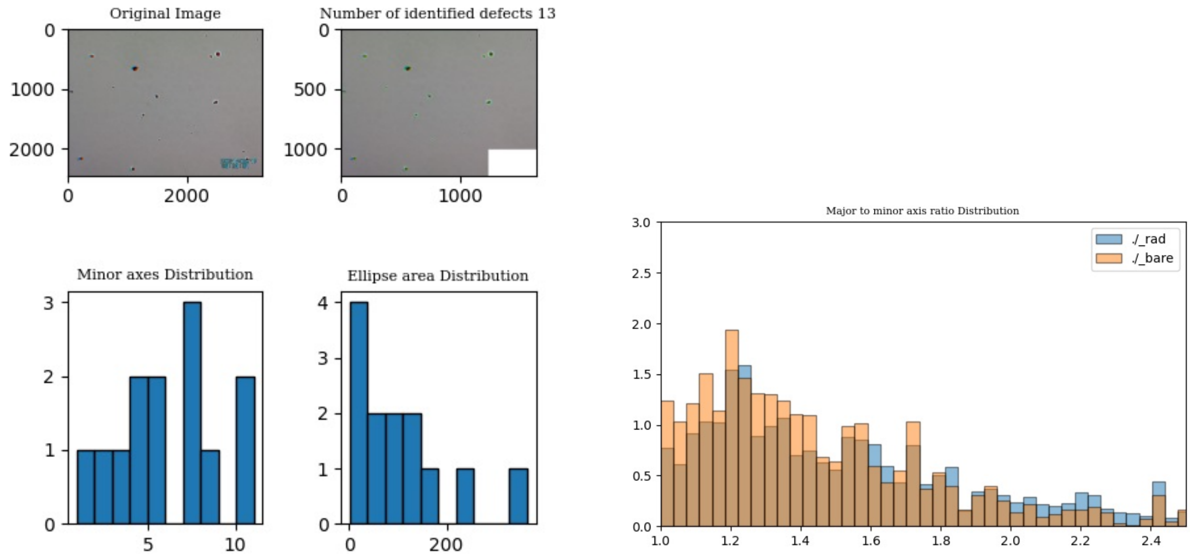


Figure 8: Track identified in CR-39 detectors using AI.

- The operation current is lower than 10 nA.
- The main application is to automatically measure the volt-ampere characteristics of the connected material with predetermined voltage steps.
- Any semi-conductor and diode can be measured.
- To measure the response of the radiation detector to different radiation sources, the calibration source can be placed near the detector during volt-ampere characteristics measurement.
- The measured data are stored in text file and graphical form.

Dark chamber

Technical information:

- Aluminium cover with dimensions: 420 × 225 × 105 mm

Features and possible applications:

- The purpose of the dark chamber is to eliminate the effect of generating false signals from the incoming ambient light in light-sensitive materials with a circuit including semi-conductor detectors.
- The design of the dark chamber is to tightly seal the measuring detector (with additional radiation source if necessary) from the light in the laboratory.

GaAs, SiC, Si detectors
Technical information:

- Semi-conductor detector can be developed with thicknesses: 50-500 μm
- Dimensions of the semi-conductor area: standard dimensions 1.5 $\times$ 1.5 cm (can be different for other applications)

Features and possible applications:

- The detectors are prepared in chemistry laboratory and thin layers of detection sensitive material are placed on the substrate by the spraying method.
- It provides a possibility to prepare different unique semi-conductor layers except GaAs, SiC and Si.
- GaAs detectors achieve 20 times higher linear attenuation coefficient in comparison with Si material in the energy region 5-100 keV (appropriate for measuring of low energy gamma rays).
- Si based detectors can be used in strong radiation environment for measurement of charged particles.

3.4..2 Characteristics of the measurement process with CR-39 passive detectors

CR-39 polymer
Technical information:

- Dimensions: 25 $\times$ 25 $\times$ 1.5 mm and 50 $\times$ 50 $\times$ 1 mm
- Possible dose measurement: 0.1-600 mSv
- Neutron energy detection range: 0.2-14 MeV
- Possible application of Radon measurement
- Density: 1.3 gcm^{-3}

Features and possible applications:

- Detection of ionizing particles, including neutron dosimetry.
- The detection process is based on the measuring of the track caused by knock-on protons from polyethylene radiator or heavy charged particles in CR-39 by ionizing radiation.

- Possible application of additional material/converter on the CR-39 polymer surface (e.g. to determine shielding properties of newly developed materials).

Thermostatic quenching bath
Technical information:

- Thermal power: 300 W
- Temperature range: 5-100°C
- Tank size: 160 × 160 × 130 mm

Features and possible applications:

- The purpose of the quenching bath is to fix and increase the size of the defects caused by ionizing particles in CR-39 polymer detectors.

Microscope for track detection
Technical information:

- Optical zoom level: 4-40x
- Digital zoom level: 50-2000x
- LCD display and SD card data storage

Features and possible applications:

- To be able to estimate the length and dimension of track, at least 50x zoom is necessary.
- Based on the shape of the trajectory it is possible to distinguish between the type of ionizing particle that induced the damage.
- The AI technology for identification of the dosimetry parameters is under development.

3.5. Laboratory of dosimetry and radiation protection

The laboratory of dosimetry and radiation protection deals with the measurement of environmental samples and other quantities, while the sensitive measurements has to be carried out in the low background chamber. It allows to measure the dose on the level of natural background. The application of these measurements is very wide from nuclear industry, chemistry, environmental protection to the estimation of the radionuclides in the water, air or food. The used detectors are alpha/beta counters iSolo with PIPS detector. In the case of the measurement of the radioactivity of water, the measurement method is based on sample evaporation. It ensures the concentration of radionuclides in dried sample to increase the measurement accuracy. In the case of air measurement, precise airflow through air-filter is estimated. In both cases, the beta activity of the dried sample and air-filter is measured. Additional measurements of environmental samples can be carried out by the HPGe detector placed in the low background chamber. To decrease the measurement uncertainty, the samples are stored in Marinelli beaker and placed on the top of the HPGe detector. Each device is using the in-house software for its measurement and the results can be stored on portable media, if needed.

Figure 9: Laboratory of dosimetry and radiation protection.

3.5..1 Characteristics of the laboratory of dosimetry and radiation protection

PIPS and iSolo detector
Technical information:

- Operating Temperature: 10°C to 40°C

- NiMH battery power for 10 hours
- Ethernet & USB interfaces
- Air filter analysis tool
- Radon measurement possible

Features and possible applications:

- Measurement of alpha and beta radiation.
- Portable device (no computer needed for operation).
- The air and dried water samples are inserted into the device through special socket (maximum sample diameter 101.6 mm).
- The dose rate of the measured samples is estimated by the datasheet provided to the detector due to the application of the energy thresholds during the measurement.
- Detected alpha emitters with energy 6-8.78 MeV.
- The possible application is to determine the radioactivity of the water and air samples taken in the environment.

HPGe detector

Technical information:

- High energy resolution
- Low sensitivity
- Liquid nitrogen cooling required
- Automatic safe shutdown in case of detectors heat up
- Software with spectrum analysis tools
- Voltage input range: 0-5000 V
- Output load capacity: 0 to 300 μA

Features and possible applications:

- Samples are placed in standard Marinelli beakers on the top of the detector.
- Possibility of continuous measurement (Nitrogen cooling is sufficient for approximately 2 weeks).
- The energy and efficiency calibration necessary for automatic estimation of dose quantities.
- Possibility to store raw data and to use auto-processing of the measured peaks in the spectra.
- In conjunction of the low background chamber, the detector is suitable for low dose measurements of the radioactive samples.

Low background chamber
Technical information:

- Low background steel shielding
- Wall size: 17 cm

Features and possible applications:

- The HPGe detector is placed in the low background chamber to lower the influence of background radioactivity for environmental samples.
- The chamber is divided into two sections where in one the sample is placed on the top of the HPGe detector and in the second section the cooling equipment for HPGe detector is placed.
- The detector is connected to the computer (allowing to save the measured data to portable medium).
- Possible application is to measure very low radioactive materials within the chamber.

3.6. Supercomputer Applications Centre

Supercomputer Applications Centre deals with computationally complex problems in the field of reactor core and shielding analyses as well as nuclear data treatment. The hardware equipment can be divided into three specialized computing systems. The first one is a 108 CPU cluster system designed for massive calculations with simulation codes MCNP and KENO for solving safety analysis for Slovak NPPS. For preparation of microscopic cross-section libraries and for Monte Carlo and deterministic calculations a 48 CPU computer is used with the total operational memory of 48 GB. The third system consists of two 64 CPU modules with 2 $\times$ 256 GB operational memory, designed for shielding analyses using the MAVERIC code. Features:

- shielding analyses using variance reduction techniques,
- criticality safety analyses,
- nuclear data processing,
- sensitivity and uncertainty analyses,
- similarity assessment,
- cross-section adjustment techniques,
- in-house calculation codes,
- job que management,
- ganglia cluster tools.

Available calculation tools:

- MCNP5, MCNP6,
- SCALE6 package,
- SERPENT,
- NJOY21,
- TRANSX,
- DIF3D,
- PARTISN,
- NESTLE,
- STUUP*,
- PORK*,
- SBJ*,
- TEMPIN*,
- FEMAXI.

* In-house calculation tool
Features:

- fixed source and criticality calculations using Monte Carlo codes,
- development of tailored multi-group and continuous-energy cross section library,
- propagation of uncertainties from nuclear data to integral parameters,
- similarity assessment of new reactor designs ,

- transient analyses of thermal and fast reactors,
- calculation of temperature distribution in the fuel pin, taking into account fuel burnup ,
- graphical post-processing of results.

Possible applications:

- safety analyses of commercial power reactors,
- design studies of promising reactor concepts,
- optimization of fuel burnup,
- nuclear data research,
- optimization of calculations,
- dose assessment of workers in the radiation environment,
- verification of experimental results,
- deep geological disposal of spent nuclear fuel and high-level radioactive waste,
- dosimetry,
- decommissioning of nuclear power plants.

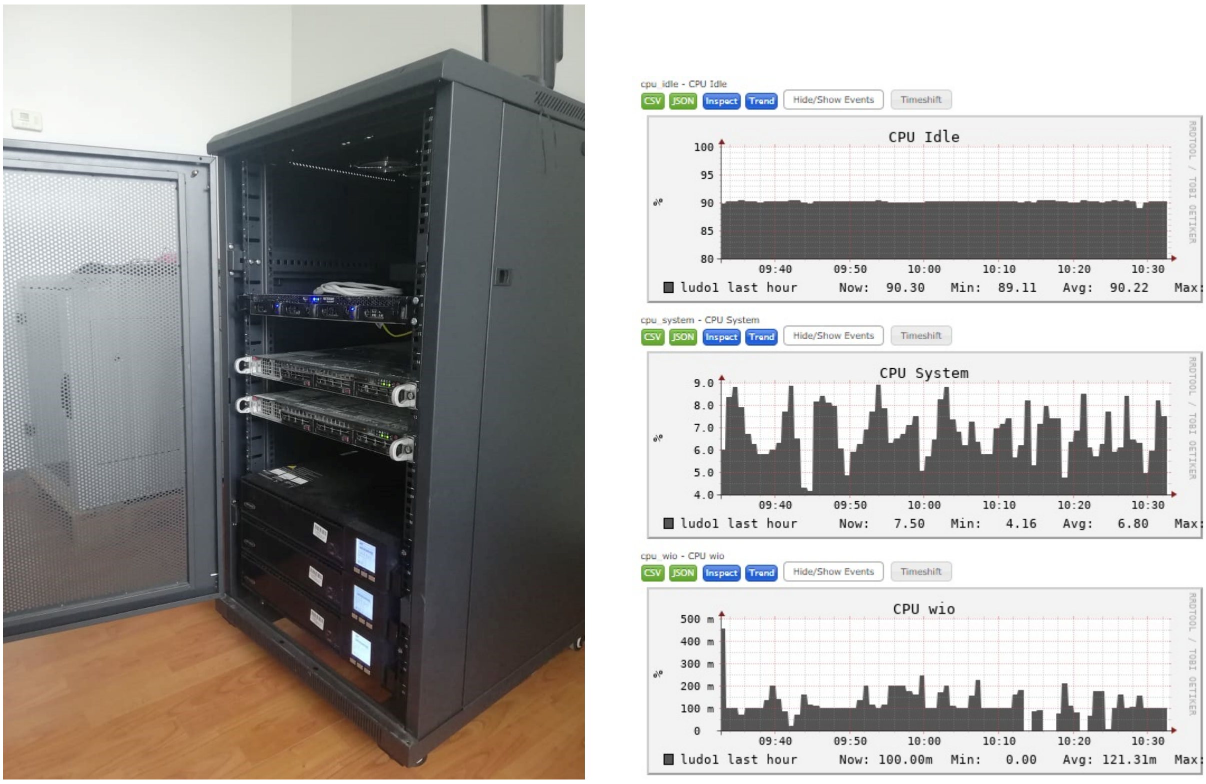


Figure 10: STU supercomputer system and cluster load.

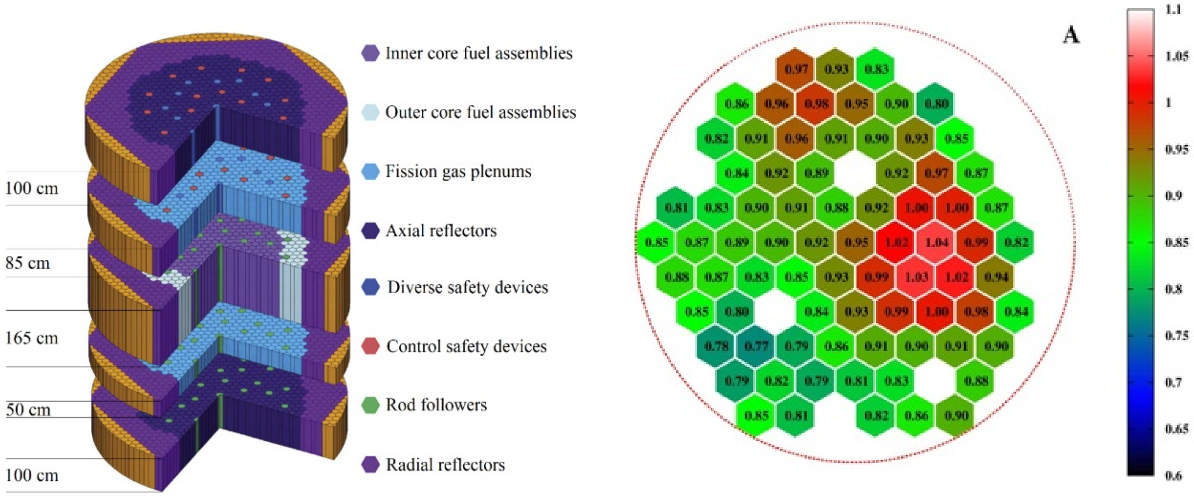


Figure 11: Calculations of fast reactors.

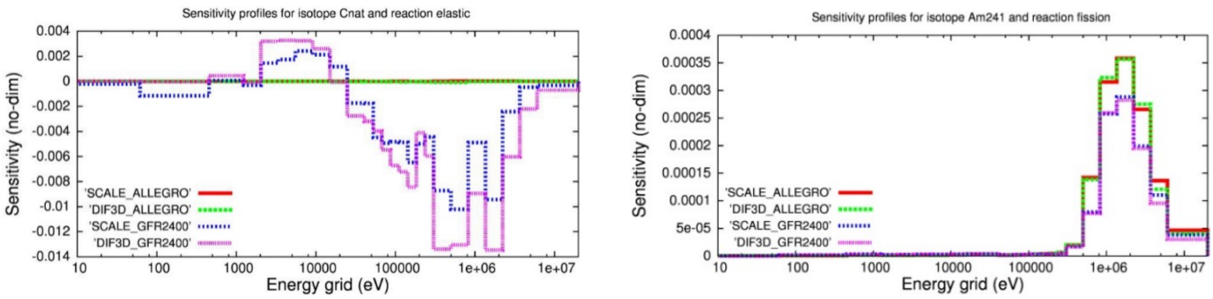


Figure 12: Calculation of sensitivity profiles of fast reactors.

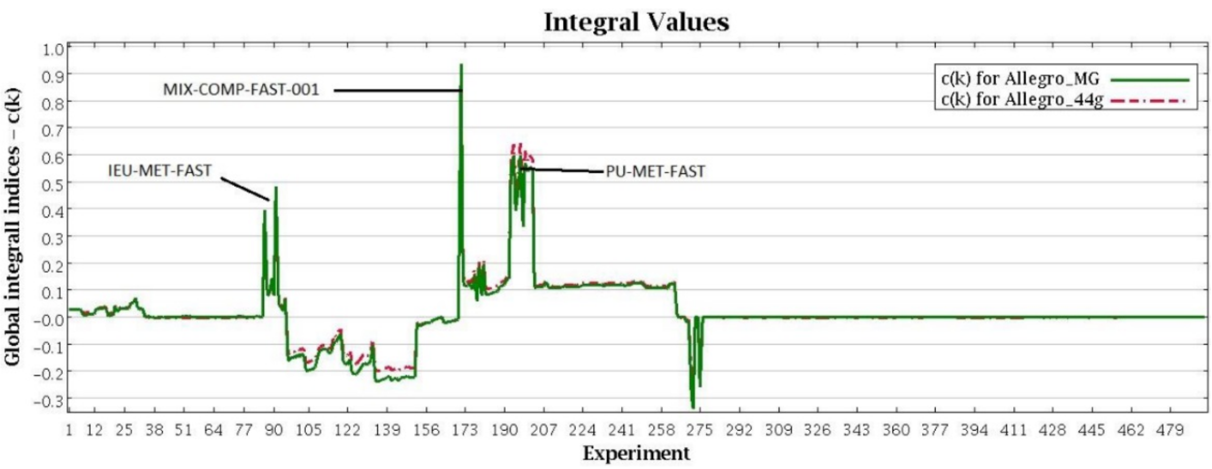


Figure 13: Similarity assessment of the Allegro reactor.