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No. NP-T-5.3

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APPLICATIONS OF  
RESEARCH REACTORS

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IAEA NUCLEAR ENERGY SERIES NO. NP-T-5.3

# APPLICATIONS OF RESEARCH REACTORS

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# FOREWORD

One of the IAEA's statutory objectives is to "seek to accelerate and enlarge the contribution of atomic energy to peace, health and prosperity throughout the world." One way this objective is achieved is through the publication of a range of technical series. Two of these are the IAEA Nuclear Energy Series and the IAEA Safety Standards Series.

According to Article III.A.6 of the IAEA Statute, the safety standards establish "standards of safety for protection of health and minimization of danger to life and property". The safety standards include the Safety Fundamentals, Safety Requirements and Safety Guides. These standards are written primarily in a regulatory style, and are binding on the IAEA for its own programmes. The principal users are the regulatory bodies in Member States and other national authorities.

The IAEA Nuclear Energy Series comprises reports designed to encourage and assist R&D on, and application of, nuclear energy for peaceful uses. This includes practical examples to be used by owners and operators of utilities in Member States, implementing organizations, academia, and government officials, among others. This information is presented in guides, reports on technology status and advances, and best practices for peaceful uses of nuclear energy based on inputs from international experts. The IAEA Nuclear Energy Series complements the IAEA Safety Standards Series.

The purpose of the earlier publication, *The Application of Research Reactors*, IAEA-TECDOC-1234, was to present descriptions of the typical forms of research reactor use. The necessary criteria to enable an application to be performed were outlined for each one, and, in many cases, the minimum as well as the desirable requirements were given. This revision of the publication over a decade later maintains the original purpose and now specifically takes into account the changes in service requirements demanded by the relevant stakeholders. In particular, the significant improvements in equipment and technology available for such utilization applications are considered.

This publication is of particular benefit to those seeking to increase the utilization of their facilities and to assist with the strategic planning required prior to the installation of new equipment or modification of an existing facility, or even for the construction of a new research reactor. This consideration becomes particularly relevant where the owners and operators of these facilities must demonstrate either the financial or the strategic value of their facilities to the relevant stakeholders.

The applications presented represent a variety — from those that are possible at any power level of research reactor, such as training, to those that require higher power and more specialized reactors with expensive experimental facilities, such as transmutation doping and radioisotope production.

The publication has been expanded to include considerations on strategic planning and user and customer relations. The simplified research reactor capability matrix which was originally developed has been updated accordingly and is now presented in Annex I. This assists in the determination of the various applications that may be appropriate for a particular power level reactor.

The IAEA wishes to acknowledge the assistance of all the experts who contributed to the updating and revision of this publication. The IAEA also thanks C. Piani (South Africa) for assistance in reviewing and editing this publication.

The IAEA officers responsible for this publication were D. Ridikas of the Division of Physical and Chemical Sciences and N.D. Peld of the Division of Nuclear Fuel Cycle and Waste Technology.

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# 1. INTRODUCTION

Research reactors are sophisticated devices for basic and applied research in the fields of particle and nuclear physics, radiochemistry, activation analysis, materials sciences, nuclear power and nuclear medicine. They also serve as powerful instruments for the production of high technology commodities, such as a wide variety of radioactive isotopes, as well as radiation modified materials important for microelectronics, space programmes and other technologically advanced areas. These reactors also enable the testing of various types of nuclear fuel and the study of radiation resistance of new materials. As a source of high intensity fluxes of neutrons, gamma quanta, neutrinos and other types of radiation, research reactors play an irreplaceable role in science, industry and medicine. Since many are based at universities and dedicated research institutions, research reactors have also established a considerable legacy in the education and training of science and engineering students and nuclear power plant staff.

Owners and operators of many research reactors are finding that their facilities are not being utilized as fully as they might desire. This can generally be attributed to a complex multitude of reasons. Most notably, many existing reactors have aged significantly, and either require increasing attention and funds for refurbishment and maintenance, or are no longer capable of performing innovative research. Furthermore, neutron intensities at many facilities are lower than accelerator based spallation neutron sources, and some analysis methods have been rendered obsolete by alternative technologies or more modern equipment at recently established research reactors. Some reactors lack precise and determined direction following the fulfilment of their designed mission. Other critical issues facing the international research reactor community include the attrition of experienced staff without adequate replacement and a lack of appropriate financial policies regarding access to, and charges for, a facility's services. At the same time, escalating pressure from stakeholders to ensure that reactors generate some form of financial income to offset operational and maintenance costs has also become increasingly relevant, although the fact that a research reactor exists and is available does not guarantee that potential users will proactively seek to take advantage of the facility.

Many research reactor owners and operators now recognize that there is a need to develop a strategic plan for long term sustainability, including the marketing of their facilities [1.1–1.3]. An important initial stage in preparing such a plan is to evaluate current and potential capabilities of the facility. An overview of current activities and applications at research reactors is provided in Table 1.1.

The purpose of this publication is to assist in such an evaluation by providing some factual and advisory information on several peaceful applications of research reactors. With the help of this information, each facility owner and operator should be able to assess whether or not a new application is feasible at the reactor, and to what extent the relevant capability in that application might require development. To assist with such an evaluation, this publication has been broadly grouped into three categories: human resource development, irradiation and testing, and extracted beam work.

- (1) The human resource category includes public information, training and education, and can be accomplished at any reactor.
- (2) Irradiation applications and testing involve inserting material into the reactor, often in highly specialized irradiation rigs or loops, to induce radioactivity for analytical purposes, produce radioisotopes or induce radiation damage effects. Almost all reactors can be utilized for some irradiation applications, but as reactor flux increases, the range of potential uses also grows. In particular, high power reactors common to national laboratory facilities can support nuclear fuel testing and experiments in specialized in-core loops.
- (3) Beam work usually includes using neutron beams outside of the reactor for a variety of analytical purposes. Because of the magnitude of the flux required at some distance from the core, most beam work can only be performed by medium and high power research reactors.<sup>1</sup>

The presentation of the applications of research reactors generally follows the progression outlined above. The specific requirements for each application are typically addressed according to flux/power level, reactor

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<sup>1</sup> Unless otherwise specified in the text, miniature research reactors are under 100 kW; low power research reactors are less than 250 kW; and high power research reactors are above 2 MW. Medium power reactors fall between low and high power reactors.

TABLE 1.1. COMMON APPLICATIONS OF 273 RESEARCH REACTORS FROM AROUND THE WORLD

Type of application	Number of research reactors involved <sup>a</sup>	Number of Member States hosting utilized facilities
Teaching and training	172	54
Neutron activation analysis	125	54
Isotope production	94	45
Material irradiation	75	29
Neutron radiography	71	40
Neutron scattering	50	33
Transmutation (doping of silicon)	31	20
Geochronology	25	21
Boron neutron capture therapy, including research and development	23	13
Transmutation (gemstones)	22	13
Other <sup>b</sup>	126	31

<sup>a</sup> Out of the 273 research reactors considered, 248 were operational, 15 were in temporary shutdown, four were under construction and six were planned.

<sup>b</sup> Other applications include: calibration and testing of instrumentation and dosimetry; shielding experiments; reactor physics experiments; nuclear data measurements; and public relations tours and seminars.

**Source:** IAEA Research Reactor Data Base (March 2013).

facilities, external equipment, personnel, funding, time required to achieve implementation of applications, and — increasingly important — public and customer relation issues. Some flexibility in these topics has been adopted, as appropriate, for each application. For example, a special section, Section 14, has been added regarding customer relations. Of particular note is that cost estimates in this publication are given in 2011 US dollars, regardless of import duties and State variations.

## REFERENCES TO SECTION 1

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- [2] INTERNATIONAL ATOMIC ENERGY AGENCY, Specific Considerations and Milestones for a Research Reactor Project, IAEA Nuclear Energy Series No. NP-T-5.1, IAEA, Vienna (2012).
- [3] INTERNATIONAL ATOMIC ENERGY AGENCY, Implementation of a Management System for Operating Organizations of Research Reactors, Safety Reports Series No. 75, IAEA, Vienna (2013).

## 2. EDUCATION AND TRAINING

Every functioning research reactor facility, irrespective of its operational power, can be used for education and training. When reviewing the potential uses of existing reactors, this type of application is not to be dismissed as being a trivial or unprofitable mission. On the contrary, it needs to be thoroughly explored and utilized to the benefit of the facility as much as the university faculty commitments and facility staff revenue requirements allow. Educating the public to be more knowledgeable of nuclear affairs usually results in less opposition and potentially more support on related issues. Similarly, educating and training the scientific and industrial communities relates to a marketing exercise to entice future customers and users of available facilities.

Education and training programmes can encompass all facets of civil society, from primary students and the general public through public tours to university courses and power reactor operator training.

### 2.1. PUBLIC TOURS AND VISITS

Encouraging and ensuring the successful implementation of educational tours for the public, students of secondary and tertiary education, teachers and other interested groups have great potential for building support and quality usage of a reactor facility (see Fig. 2.1). In the short term, facility staff successfully demonstrating presentations, experiments and practical activities for visitors can develop student interest in science. In the longer term, the number of students graduating in nuclear science and technology can be increased if interest is generated at an early age. In addition, education, training and research applications of a reactor facility can increase as others become more aware that the facility is active and providing services.

Tours and visits may involve time commitments from a few minutes with one staff member to most of the day with several research reactor employees. Such activities can involve a simple facility walk through for a few visiting non-technical students to a full day visit by students from a physics class. When addressing the latter, or an audience of similar ability, lectures could be presented on reactor capabilities and usage. A reactor startup could be demonstrated, if allowed by the institution, and experiments could be performed, followed by one or more detailed tours. These experiments could include a rabbit system irradiation of a pure, single element nuclide (see Section 3), for example aluminium, for half-life measurements, or irradiation of a volunteer hair or nail clipping



FIG. 2.1. Open house at the Kansas State University TRIGA Mark II reactor facility (courtesy of Kansas State University).

sample to identify trace elements. Spectrometry measuring X ray fluorescence can demonstrate the identification of radioisotopes such as  $^{241}\text{Am}$  in jewellery or other volunteered samples. Other activities may involve dressing the visitors in anticontamination clothing, with sample training of survey meters to measure radiation dose rates associated with low activity radioactive materials such as thorium bearing gas lantern mantles, uranium bearing dishes or radium bearing watch and clock dials. The handling of simulated radioactive material also works well, and fluorescent materials such as motor oil or powdered laundry detergent can be used, since they scintillate under an ultraviolet light.

Students can therefore undertake careful handling and packing of such material in transfer containers, and then use an ultraviolet light to see how successful they were. They can also clean the area using standard decontamination techniques and evaluate the degree of success using a final check with ultraviolet light.

Key to successful public tours and visits is to:

- Know the group well enough to make presentations at the proper level;
- Answer questions directly and provide ample time for questions, answers and interaction;
- Not speak down to the group;
- Provide a mix of lectures and practical and experimental activities to maintain student attention;
- Provide enough experimental equipment to avoid students waiting to be involved;
- Provide written material, samples and brochures for students to take home.

The research reactor staff can assist through various practical exercises:

- (a) Conduct lectures describing the reactor and its capabilities and usage, making liberal use of any available models, such as those of fuel assemblies, research facilities or power reactor models, and including suitable viewing areas of the operation room or experimental facilities.
- (b) Administer demonstrations and practical exercises in the use of survey meters, self-reading pocket dosimeters and anticontamination clothing.
- (c) Provide a detailed walk through tour of accessible areas of the facility, emphasizing physical components and systems reviewed or referred to in the lecture.
- (d) Allow students to observe a reactor startup and the approach to full power, regulations permitting.
- (e) Perform an inverse square law measurement using a low level radioactive source and a simple detector, or perform a shielding experiment.
- (f) Conduct a half-life measurement using a radioisotope made in the reactor.
- (g) Show students how to prepare a sample for irradiation and have them prepare one or more such samples. One or more standards can also be prepared.
- (h) Irradiate a hair sample or nail clipping, and show students how to identify and even quantify elemental concentrations. If time and the number of students permit, the samples or standards can also be counted and analysed to obtain some readily identifiable elements such as aluminium, chlorine, magnesium, manganese, sodium and vanadium.
- (i) Involve students in other activities that may be available at the facility.

### **2.1.1. Flux/power level requirements**

Any power level is sufficient for this category of education and training. Even critical facilities can be successful in this area of application, although they might not be able to reap the full benefits a research programme can offer. Regardless of power level, bearing in mind that most facilities have regulatory restrictions regarding access to radiological areas, visiting groups from secondary education and non-technical university classes can undertake visits to learn about the reactor, its uses and capabilities, and to perform practical activities and experiments, thereby gaining an understanding of nuclear science and technology.

### **2.1.2. Reactor facility requirements**

The only requirement for performing meaningful, practical activities is to have a reactor. As indicated above, any power level is acceptable, and is only restrictive in terms of the complexity of the practical activities

that normally occur at the reactor facility. Consideration must be given to the operation of the reactor and access flexibility, and radiological safety and security requirements. In some States, an age limit is set for people entering radiological laboratories; constraints exist for pregnant women. Taking photographs is not allowed in several facilities. Should maintenance or other activities make the reactor unavailable, tours and visits may still be possible, with the exception of sample irradiations.

For visits to research reactors with neutron beam facilities, it is highly desirable to provide one or more areas where panoramic views of the neutron beam instrument suite can be seen by groups of between 2 and 16 visitors (see Fig. 2.2). Viewing areas may be equipped with displays such as poster boards or computer screens showing highlights of the scientific applications and experimental techniques.

### 2.1.3. Experimental equipment requirements

The tour agenda can be tailored to the experimental equipment in service at the facility. One of the key considerations for successful group visits is to ensure active group involvement in all, or most, of the activities. Therefore, when larger groups are involved, additional sets of experimental equipment need to be available for several smaller groups of ideally three or four students — this makes it easier to maintain student interest. For analysis of an activated sample, a scintillation detector and computer based multichannel analyser (MCA) is sufficient for a simple analysis demonstration. Germanium detectors are required for demonstrating X ray fluorescence. If two or more computer analysers and screens can be used, the students' interest is more likely to be sustained. Demonstrations and experiments with neutron beams are more difficult, and are usually restricted to university level science students.

### 2.1.4. Personnel requirements

A tour or visit of this type requires a reactor operations person with the skills to perform lectures and demonstrations. For groups of more than five or six, additional personnel are necessary to ensure the success of the practical hands-on activities. The size of the laboratory and the quantity of experimental equipment will generally limit the group size. It is thus recommended that groups larger than 30 be arranged into groups of no more than 15, according to regulations, to ensure active participation in the lectures and demonstrations.



*FIG. 2.2. View from the visitor's gallery of the neutron guide hall of the FRM II reactor (courtesy of Technische Universität München).*

### 2.1.5. Funding requirements

Existing equipment and materials should allow a facility to offer tours. Funding for additional equipment should not be required for the contamination control exercise described above, except for the ultraviolet light (around US \$100). Additional staff time may be necessary, especially if visitors prefer to come outside normal working hours. In addition, funding for extra sets of equipment, such as Geiger–Müller (GM) detector systems and self-reading pocket dosimeters, may be needed. Most of this equipment is generally not too expensive. More complex experimental equipment could incur significant costs, for example, a scintillation detector with an MCA can cost approximately US \$25 000.

### 2.1.6. Public relations and staff cooperation

Public relations and staff cooperation are very important for reactor utilization in education and training. Every effort must be made to provide interesting and informative lectures that make use of demonstrations, pictures, models and, preferably, hands-on activities. Individuals presenting such material and overseeing such activities must show enthusiasm. Successful first interactions ensure return visits and word of mouth advertising to friends and colleagues. The facility itself may also send out periodic notices describing the availability of the reactor for visits by universities, colleges, schools and other groups to build up a regular clientele of satisfied customers who themselves will further advertise. Finally, cooperation is required among facility personnel to accommodate occasional interest in a group visit outside normal working hours. Staff personnel must also show a collegial attitude during all visits and, most importantly, they must be respectful and encouraging towards all visitors. The reactor manager should promote this attitude.

## 2.2. TEACHING PHYSICAL AND BIOLOGICAL SCIENCE STUDENTS

Research reactor facilities that are associated with educational institutions have a natural audience for this application. However, even with physical and biological science classes taught near the reactor, the facility managers still need to approach the faculty and propose the use of their reactor. Non-university research reactors should not be discouraged from this application, as there will often be one or more educational institutions within a reasonable distance from the reactor facility.

Students in scientific disciplines such as biology, chemistry, geology, physics and even (veterinary) medicine can often be attracted to more sophisticated use of a research reactor when their initial contact is used to reveal the pertinent utilization opportunities available. Because of the broad range of applications, the initial lecture and facility tour to familiarize students and the visiting faculty with its capabilities must be presented by a knowledgeable individual dedicated to communicating and demonstrating the possibilities. These students and faculty may then be encouraged to seek more sophisticated use of the reactor, such as for a course project, or perhaps even for a funded research project.

### 2.2.1. Flux/power level requirements

A minimum flux of  $10^{11} \text{ cm}^{-2}\cdot\text{s}^{-1}$  is probably necessary to demonstrate possibilities in trace element analysis. Simple materials such as pure aluminium or calcium carbonate, elemental iodine and other materials may be irradiated, and the resultant radioactive half-life measured on an available counting system such as a GM counter, or a gas flow proportional counter, scintillation or semiconductor detector system. Such an introductory experiment is most appropriate following a lecture and tour of the facility. The tour should emphasize the capabilities and possible usage, especially as related to the disciplines involved. Prior to arrival, someone in the discipline — perhaps the class instructor or the students themselves — should be consulted to determine the type of application that would be of interest. This allows the reactor representative to refer to a potential use.

In this initial session, a simple application can be demonstrated. One of the most successful demonstrations is to explain the presence of trace elements in hair or nail clippings, and then to obtain two samples, one of which is cleaned with alcohol and the other not. The resultant spectra obtained on a germanium detector following sample irradiation are used to demonstrate the capabilities of instrumental neutron activation analysis (INAA) to identify

and to quantify trace elements in the hair samples. The presenter can explain this forensic application and extend the discussion to further possibilities for trace element analysis in other areas.

After the initial demonstration, specific discipline usage of the reactor will vary depending upon the course. For example, a course of general interest is radiochemistry, which can utilize the reactor for a series of experiments, including:

- Half-life measurements;
- Trace element analysis using INAA;
- Measurements of variation of detector efficiency with source location;
- Inverse square law measurements.

Students of anthropology, biology and geology are usually interested in the trace element analysis technique, while neutron and gamma irradiation effects on materials, as well as neutron transmission, are of interest to those attending materials science, physics and biological science courses. In the latter, a demonstration of the irradiation of biological material could include the effects of dose rate and integrated dose. A minimum flux of  $10^{11} \text{ cm}^{-2}\cdot\text{s}^{-1}$  is sufficient for these experiments. However, they become more realistic and effective with flux levels at, or above,  $10^{11} \text{ cm}^{-2}\cdot\text{s}^{-1}$ , at which significant trace element analysis projects can be performed.

At research reactors with neutron beam facilities, demonstrations and experiments are normally limited to advanced studies for university science and engineering students. Typically, such training is conducted over periods of between one and four weeks, and includes introductory lectures on scientific applications and experimental methods, performance of neutron beam experiments, and visualization and analysis of results with computer based analytical tools. Examples of simple experiments that exemplify the unique contrast of neutrons include neutron radiographs of objects such as fuel pumps or coffee percolators, powder diffraction of magnetic materials such as mild steel, and small angle scattering of porous media such as Vycor glass.

A student designed semester project is an important area of utilization. These projects can make excellent use of reactor facilities by employing techniques previously learned during courses. In particular, courses such as radiochemistry and radiation biology can make particular use of such facilities for semester projects. Some examples are dose rate characterization of experiment facilities, trace element analysis of water filters and detection of osmotic water movement through concrete, which requires a collimated beam. These experiments can all be accomplished with a flux of  $10^{11} \text{ cm}^{-2}\cdot\text{s}^{-1}$  or higher, and represent excellent establishments for producing students with an interest in performing research using a reactor.

### **2.2.2. Reactor facility requirements**

The only requirement to perform experiments involving activation and simple radiochemistry as described above is an irradiation system (e.g. a rabbit system) to facilitate sample irradiation. For short courses on neutron scattering, a lecture room with a capacity for 20–50 students and classroom type facilities equipped with networked computers are required for training in analysis of neutron beam experiments.

### **2.2.3. Experimental equipment requirements**

The experimental equipment required is normally available in the vicinity of a reactor facility. As more advanced experiments are performed, the equipment required may become more complex. However, it should be possible to obtain the equipment from existing research or service applications. There is usually no need to acquire new equipment for educational purposes.

### **2.2.4. Personnel requirements**

There are no additional operating personnel requirements since existing staff members can perform the work.

### 2.2.5. Funding requirements

Since all educational involvement uses existing reactor and experimental instrumentation and equipment, the only additional funding requirements are for staff overtime commitments and supplies (e.g. rabbit capsules).

### 2.2.6. Customer relations

Educational users (e.g. a university class with a faculty member) should receive encouragement and dedicated assistance and supervision of the reactor usage during their class visits. It is essential that their first experience is successful to encourage continued and expanded educational usage by faculty members and science and engineering colleagues who may learn of the success by word of mouth.

Another important aspect in encouraging reactor usage is to evaluate the needs of the faculties in science, engineering and other departments at the reactor's own educational institution and, where applicable, at other educational institutions. Capable upper level reactor facility personnel should perform such surveys periodically and volunteer to provide instructional input and laboratory experiences for interested faculties. An especially promising promotional technique is to provide seminars reviewing the reactor facility, its capabilities and usage, as well as its availability for use for a series of experimental activities in a variety of disciplines. This type of promotion using lectures and seminars can produce extensive increases in research reactor utilization.

## 2.3. TEACHING RADIATION PROTECTION AND RADIOLOGICAL ENGINEERING STUDENTS

Research reactor facilities that have nearby educational institutions teaching radiation protection and radiological engineering can readily avail themselves of the opportunity to present applicable training to students.

Education and training in radiation protection and radiological engineering for students and technicians is an activity that can be undertaken by reactor facilities of all power levels. However, the exercises can be more complex at facilities where highly activated materials are produced and potentially some radioactive effluents are possible. This application can be taught as individual classes (see Fig. 2.3) or used for training in an independent laboratory course where trainees receive practical experience in applying the principles of radiation protection. It may be possible to accomplish these activities with existing reactor facility radiation protection equipment and, in many cases, it can even be the actual activities required to be performed for regulatory requirements.



FIG. 2.3. Training at the North Carolina State University PULSTAR reactor (courtesy of North Carolina State University).

### 2.3.1. Flux/power level requirements

Although sufficient fluence must be available to activate samples to produce detectable activities, even very low power research reactors can often provide such fluences. The following set of experiments is typical among the training activities conducted for students and technicians, consisting of a series of lectures, demonstrations and associated practical activities and experiments.

#### 2.3.1.1. Typical experiments

- (a) Reactor pre-operational and operational surveys can be performed, including collecting and analysing air, water and swipe (smear) samples. The experiments can include the use of counting equipment such as gas flow proportional counting systems or NaI(Tl) and germanium detector systems to identify radioisotopes. If no real activity or contamination exists, the decay products of radon are detectable in high volume air samples.
- (b) The use of anticontamination clothing can be demonstrated (see Section 2.1).
- (c) Gamma and neutron radiation surveys utilizing different kinds of survey meters can be used to demonstrate instrument characteristics and sensitivity as the reactor power level is varied, beam ports are opened and closed, and shielding is modified. The sensitivity of these instruments can be demonstrated using gamma ray sources with various energies. Proper demarcation of areas need to be emphasized to demonstrate appropriate radiological controls.
- (d) Calibration checks can be performed on air particulate detectors, stack radiation monitor detectors, area radiation monitors and reactor coolant radiation monitors using standard sources. The sources can also be used to confirm calculated inverse square law predictions.
- (e) Scenario development and emergency evacuation drill simulation can be conducted, with response and evaluation provided by student teams. This is an excellent experimental activity to teach students to perform as an effective response team.
- (f) Radioactive sample monitoring and characterization can be performed with the development of applicable documentation so that the sample can enter a transportation cycle.
- (g) Air samples can be collected and analysed to characterize activity from  $^{41}\text{Ar}$  or other gaseous radionuclides in gaseous effluent. Evaluation of actual levels with respect to permissible levels can also be performed. Students can then develop resultant restrictions during hours of reactor operation or permissible power levels.
- (h) Radiation source leak checks can be performed, including the reactor neutron source and sealed gamma sources.
- (i) Creating the radioisotopes containing a single element by neutron activation (e.g. aluminium, calcium and iodine) enables measurements of individual half-life. In the case of a multiple element sample, the analysis can demonstrate how a decaying radioisotope can dominate the sample's half-life.
- (j) Gamma spectrometry on irradiated samples can be performed to demonstrate the process for identifying radioisotopes using photopeaks in the spectra. The discussion should include the applications of this technique to identify radioisotopes in various types of sample, including atmospheric, geological and forensic specimens.
- (k) Shielding effectiveness experiments can be undertaken where a beam port or other radiation source is accessed and shielding is placed to reduce the radiation field to meet the required radiation dose rates. This experiment works best if several types of portable reactor shielding material are available. As part of this experiment, students may learn to develop a radiation work permit for use in a radiation or contaminated area.
- (l) A frisker type handheld radiation meter may be used to survey low levels of contamination and demonstrate the use of a portal monitor. If a whole body counter is available, student monitoring can be a useful experiment. It can be performed with and without radioactive materials (e.g. lantern mantles) or other available unlicensed materials, concealed in or on clothing. The same can be performed with handheld survey meters, perhaps allowing one group to hide sources and another group to find them.

### **2.3.2. Reactor facility requirements**

The only requirement for the performance of meaningful, practical activities in this area is a research reactor of any power level. At higher power levels, more complex experiments are possible.

### **2.3.3. Experimental equipment requirements**

The training activities discussed earlier use readily available radiation protection materials and supplies, since the training is based on regularly performed radiation protection activities modified for training and education purposes. Extra quantities of materials (e.g. anticontamination clothing) may be required for larger numbers of participants. If the programme expands to a large number of students, additional survey instruments and other equipment may be necessary.

### **2.3.4. Personnel requirements**

Additional personnel are not generally required, but dedicated staff are necessary to create an enthusiastic atmosphere among the participants. However, if large numbers of students are involved, assisting personnel will be required to provide more personalized training.

### **2.3.5. Funding requirements**

Minimal additional funding above the normal operational budget is required. Funding for additional reactor operating hours and assisting personnel for large groups of students may be especially necessary if training is to take place outside normal working hours. The only essential extra equipment is an ultraviolet light for contamination detection and control simulation experiments and sufficient protective clothing for groups involved in hands-on radiological training.

### **2.3.6. Public relations and communication**

Facility availability for radiation protection training and education should be communicated to industrial organizations and academic and research institutions that may require personnel to be trained in radiation protection. Such training may also be offered to students in related academic disciplines, such as environmental science and chemistry. The training of high school science teachers needs to be a priority.

## **2.4. TEACHING NUCLEAR ENGINEERING STUDENTS**

Research reactor facilities that have nearby educational institutions teaching nuclear engineering can readily avail themselves of the opportunity to teach such students. However, this application greatly depends on the scientific infrastructure around the facility.

Four types of experimental reactor usage for nuclear engineering education are possible. The first type is reactor physics experiments to measure static and kinetic reactor parameters, including experimental facility characteristics. The second type is reactor utilization for experiments such as radioisotope production, neutron activation analysis (NAA) and extracted neutron beam experiments. Finally, the third and fourth types involve operator training for nuclear engineering students and future research and power reactor operators. Such training can provide these latter groups with experience in operations involving reactivity and temperature changes and their effects to demonstrate how theory and practice are related. The experiments are often divided into two groups: above and below the point of adding heat. The level of theoretical explanation for nuclear engineering students needs to be at a higher level than that for operator trainees.

### 2.4.1. Flux/power level requirements

A research reactor of any power level is suitable for training nuclear engineering students. It should be noted that more complex experiments and training courses can be conducted with a higher flux.

#### 2.4.1.1. Typical experiments

##### (a) Reactor physics experiments

- Measurements of flux spectra and variations at beam ports and thermal columns;
- Measurements of reactor kinetics parameters;
- Subcritical multiplication and shutdown margin measurements during the approach to criticality;
- Control rod or control blade calibration;
- Excess reactivity and shutdown margin measurements;
- Measurements of reactor period with correlation to one or all six delayed neutron groups;
- Measurements of temperature coefficients and heat-up rates;
- Calorimetric heat balance and nuclear instrument calibration;
- Absorber reactivity worth measurements;
- Measurements of hot channel factors, as allowed by facility design and operational limits and conditions, and effects of control rod position on nuclear instrument indications;
- Power decay and delayed neutron group measurements;
- Void coefficient of reactivity measurements.

##### (b) Utilization experiments

These applications would be demonstrated only if the experimental facilities already exist. They would not normally be developed specifically for educational purposes:

- Trace element analysis, radioisotope production and parent–daughter separation;
- Neutron radiography;
- Neutron transmission experiments using beam ports;
- Neutron scattering experiments using beam ports;
- Fermi chopper thermal neutron time-of-flight (TOF) experiments using a thermal column.

##### (c) Reactor operation experiments below the point of adding heat

These experiments are similar to the reactor physics experiments discussed above. However, the emphasis is now on operations and reactor control, as well as instrumentation systems and understanding basic physics measurements, prior to the addition of heat. The objective is to provide students with operations experience similar to that of a reactor operator:

- Withdrawal of control elements to demonstrate subcritical multiplication;
- Neutron source effects with subcritical multiplication;
- Startup to criticality with the source, followed by source removal effects;
- Approach to criticality or inverse multiplication experiments;
- Measurements of stable reactor period and criticality demonstration;
- Control rod calibration measurements;
- Low power measurements with observation of delayed neutron effects;
- Manual versus automatic control and the speed of response;
- Temperature and void coefficient of reactivity measurements;
- Effect of control rod position on nuclear instrument readings.

##### (d) Reactor operation experiments above the point of adding heat

These experiments are similar to the reactor physics experiments discussed above. However, the emphasis is now on heat adding operations together with reactor instrumentation and control (I&C) systems and understanding basic physics measurements. Once again, the objective is to provide students with operations experience similar to that of a reactor operator:

- Withdrawal of control elements to demonstrate subcritical multiplication;
- Approach to criticality experiments;
- Control rod worth measurements by rod withdrawal or rod drop methods;
- Measurements of temperature coefficients and heat-up rates;
- Automatic control and speed of response with observation of delayed neutron effects;
- Calorimetric heat balance and nuclear instrument calibration;
- Effect of control rod positions on nuclear instrument readings;
- Measurement of hot channel factors as allowed by facility design and operating limit conditions;
- Controlled cooling transient (cold slug) demonstration;
- Shutdown with immediate restart (hot restart operations).

(e) Reactor instrumentation and control systems

These experiments provide students with an insight into the typical instrumentation used for the safe control of the reactor during operational conditions:

- Explanation of reactor I&C systems;
- Calibration of nuclear channels;
- Calibration of temperature channels;
- Rod drop time measurements;
- Boron ionization chambers;
- Fission chambers;
- Self-powered neutron detectors (SPNDs);
- Logic circuits (two out of three systems).

#### 2.4.2. Reactor facility requirements

These experiments are possible on reactors of any power level with the required experimental facilities. However, they are most easily performed on a low power training reactor.

#### 2.4.3. Experimental equipment requirements

Neutron flux and neutron spectrum measurements require an assortment of neutron activation foils and threshold detectors (e.g. aluminium, cadmium, gold and indium), as well as counting facilities such as gas flow proportional counters or scintillation (NaI(Tl) or LnBr) and germanium detectors to the level of sophistication desired.

An approach to criticality experiments may require one or more neutron detection systems using boron trifluoride (BF<sub>3</sub>), <sup>10</sup>B or fission chambers, along with amplifiers and readout devices. All other experiments will normally utilize existing equipment.

#### 2.4.4. Personnel requirements

All experiments require a reactor operator. Some reactor physics experiments (e.g. a criticality experiment) require measurements and data collection in addition to those provided by the existing reactor instrumentation. In this case, a staff member is required to set up and to operate the equipment. The reactor operator or another individual must acknowledge the impact of the experiment upon operation and its results for the students involved.

Utilization experiments require an individual stationed in the area of the experiment. This individual can provide an introduction to the experiment and can oversee and direct the students' work.

Some operations experiments may be conducted with only a highly skilled reactor operator. Other experiments (e.g. an approach to criticality) might require other professional individuals.

#### 2.4.5. Funding requirements

Additional funding is usually not required for utilization and operations experiments. Reactor physics experiments that require instrumentation not available at the reactor might require some additional funding. The cost will depend on the level of sophistication desired in the experiment and may vary from US \$5000 to US \$40 000.

Flux and flux distribution or energy and spatial distribution measurements may make use of existing counting systems. However, if a new system must be procured, approximately US \$20 000–80 000 — depending on the choice of detector — is needed, and several thousand dollars may be required for the required activation foils and wires if not already available. For fine measurements of energy distributions common to graduate and doctorate research, special filtering and TOF equipment will add costs of US \$20 000–60 000.

### 2.5. NUCLEAR POWER PLANT OPERATOR TRAINING

Research reactors situated within a reasonable distance to the nuclear power plant are generally ideally suited to perform nuclear power plant operator training. However, many nuclear power reactor facilities are willing to send some of their reactor operator trainees for residential courses fairly distant from the power plant.

It should be recognized that the training of operators for nuclear power plants using research reactors has become less frequent in many States as plant specific power plant simulators have become more prevalent. Nevertheless, this activity continues in some industrialized countries and has the potential for considerable utilization in developing countries. This training usually consists of a set of experiments intended to provide hands-on experience in manipulation of reactor controls for the understanding of the principles of subcritical multiplication through full power operation.

In addition, nuclear power plant operators are usually assigned to a very specific task at their facility. Training or re-training at a research reactor offers the opportunity to gain further insights and improve their background in their daily work at the nuclear power plant (see Fig. 2.4).



FIG. 2.4. Remote view of the TRIGA reactor core displayed on a high definition camera system used for reactor operator training (courtesy of the Jožef Stefan Institute).

### 2.5.1. Flux/power level requirements

Low power level reactors may be utilized to perform the experiments presented in Reactor operation experiments below the point of adding heat, Section 2.4.1.1. Higher power level reactors may be utilized to perform the experiments presented in Reactor operation experiments above the point of adding heat, Section 2.4.1.1.

### 2.5.2. Typical training courses

Every opportunity is to be taken to communicate internationally that a particular research reactor facility is available for power plant operator training. This is especially important in developing countries where the construction of power reactors is anticipated, as it takes some time to build up the personnel resources and infrastructure required in the nuclear field.

In addition, a research reactor facility may be used to offer its training possibilities to States embarking on a nuclear power programme, intending to establish either a nuclear research centre or a nuclear power plant. In this case, a highly specialized training course lasting six to eight weeks may be developed, covering the following topics as examples:

- (a) Administrative and organizational topics:
  - Regulatory requirements;
  - Code of conduct;
  - Research reactor management;
  - Staffing requirements;
  - Site requirements;
  - Waste management;
  - Public information;
  - Physical security;
  - Emergency procedures;
  - Nuclear project planning, and implementation and control;
  - Decommissioning planning and implementation.
- (b) Reactor related topics:
  - Research reactor overview;
  - Research reactor utilization;
  - Introduction to atomic and nuclear physics;
  - Reactor physics;
  - I&C systems;
  - Thermal hydraulics;
  - Maintenance and inspection programmes;
  - Fuel management;
  - Fuel cycles;
  - Water chemistry;
  - Radiation monitoring;
  - Radiation protection;
  - Personnel monitoring;
  - Environmental monitoring.
- (c) Practical courses on:
  - Reactor physics and kinetics;
  - I&C systems;
  - Radiation protection and dosimetry;
  - Fuel management.

Such courses can be offered on a commercial basis. The revenue may be used to hire highly qualified lecturers or to provide for the use of dedicated reactor operation time and the institute's overheads.

### 3. NEUTRON ACTIVATION ANALYSIS

NAA is a method for the qualitative and quantitative determination of elements based on the measurement of characteristic radiation from radionuclides formed directly, or indirectly, by neutron irradiation of the material [3.1]. The most suitable source of neutrons for such an application is usually a nuclear research reactor. NAA can be performed in a variety of ways depending on the element and the corresponding radiation levels to be measured, as well as on the nature and the extent of interference from other elements present in the sample.

Next to education and training, NAA is the most widely used application of research reactors. Almost any reactor in excess of 10–30 kW is capable of providing a sufficient neutron flux to irradiate samples for selective applications of NAA. The costs of setting up an NAA facility is relatively low compared with the costs of neutron beam instruments. Since many of the uses of trace element determination can be directly linked to potential economic benefits, NAA is to be regarded as a key component of most research reactor strategic plans.

#### 3.1. METHODS OF NEUTRON ACTIVATION ANALYSIS

NAA is based upon the conversion of stable atomic nuclei into radioactive nuclei by irradiation with neutrons and the subsequent measurement of the radiation released by the decay of these radioactive nuclei. Among the several types of radiation that can be emitted, gamma radiation offers the best characteristics for the selective and simultaneous determination of elements, although one method of NAA, delayed neutron counting (DNC), is based on the measurement of delayed neutrons emitted during radioactive decay.

INAA is the most widely used method of NAA. Radionuclides may be produced from all elements present in the sample, albeit at sometimes greatly variant production rates. This mixture of radioactivities can typically be analysed by two different methodologies:

- (1) The resulting radioactive sample is chemically decomposed and the total number of radionuclides is split up by chemical separation into many fractions with a few radionuclides each. This is called destructive or radiochemical neutron activation analysis (RNAA).
- (2) The resulting radioactive sample is kept intact, and the radionuclides are determined by taking advantage of the differences in decay rates through optimized measurements at different decay intervals, utilizing equipment with a high energy resolution for gamma radiation. This is INAA, also called non-destructive NAA. Sample preparation for INAA is depicted in Fig. 3.1.

Several modes can be distinguished within the INAA methodology. In NAA, reactor neutrons are used, or more precisely, often a combination of thermal and epithermal neutrons. When mostly epithermal neutrons are used for irradiation, the method is called epithermal neutron activation analysis (ENAA). This method is advantageous if the epithermal activation rates of the element of interest are much higher than those of interfering elements. NAA with only fast reactor neutrons is, in principle, possible, but seldom practised. Cyclic INAA is a method for enhancing the signal to background ratio of a radionuclide with a short half-life, to be measured in the presence of other radionuclides with much longer half-lives. The sample is irradiated for a short time, quickly transferred to the counting station and again irradiated. This process is repeated for an optimum number of cycles. Large sample NAA is a method in which samples much larger than the common test portion, such as intact artefacts, are analysed, and can vary from several grams to kilograms, even without subsampling.

DNC is a form of NAA exclusively practised for the detection of the fissionable elements thorium and uranium. Some of the short half-life fission products decay by beta emission to excited levels of a daughter radioisotope that subsequently reaches a stable ground state by immediately ejecting a neutron. One primary example of this process is the yield of  $^{87}\text{Br}$ , a significant fission product of  $^{235}\text{U}$ , which undergoes beta decay to  $^{87}\text{Kr}$  followed by neutron emission to stable  $^{86}\text{Kr}$ . Detection of these delayed neutrons is a highly specific technique for measurement of amounts of thorium or uranium. The energies of the delayed neutrons vary between 250 and 910 keV. Similar physical principles are valid for the fission of  $^{232}\text{Th}$  and  $^{238}\text{U}$  by fast neutrons, but the fission cross-sections for these reactions are roughly three orders of magnitude lower than those for the fission of  $^{235}\text{U}$  with thermal neutrons.



FIG. 3.1. Sample preparation for instrumental neutron activation analysis (courtesy of the Malaysian Nuclear Agency).

It is possible that interference from the other elements present in the sample is so severe that the element of interest cannot be reliably determined by INAA. Under such circumstances, RNAA could be the preferred method to follow.

Chemical separations are often employed in conjunction with NAA, and may be applied to separate the element of interest from the matrix prior to the irradiation. Since an ‘interference free’ test portion is thus obtained, any other analytical technique may also be used. Sometimes, NAA is preferred because of the technique’s superior sensitivity, and the method can be classified as preconcentration NAA. If the element of interest is either replaced or combined with another element that can be determined by NAA with better sensitivity, the method is referred to as derivative NAA.

The measurements in INAA are often optimized on the basis of three categories of radionuclides, depending on the half-life of the radionuclide produced:

- (1) Short lived radionuclides (with a half-life around 1 s–10 h);
- (2) Medium lived radionuclides (with a half-life around 10 h–5 d);
- (3) Long lived radionuclides (with a half-life longer than 5 d).

The requirements for the detection and measurement of each type of radionuclide are presented below.

### 3.1.1. Flux/power level requirements

The amount of an element to be detected in NAA is linearly proportional to the irradiated amount, the neutron flux (to some extent) and the detector full energy photopeak efficiency. However, there is an inverse exponential relationship with the half-life of the radionuclide. The minimum detectable amount is based on the signal to background ratio in the gamma ray spectrum. Hence, although a minimum neutron flux of  $10^{10} \text{ cm}^{-2}\cdot\text{s}^{-1}$  can be used for the determination of some elements in some matrices, a neutron flux greater than  $5 \times 10^{11} \text{ cm}^{-2}\cdot\text{s}^{-1}$  is desirable for the measurement of trace elements in, for instance, geological matrices. The typical neutron flux in low power level reactors varies in the range of  $(2.5\text{--}10) \times 10^{11} \text{ cm}^{-2}\cdot\text{s}^{-1}$ , while the flux is in the range of  $(0.1\text{--}10) \times 10^{13} \text{ cm}^{-2}\cdot\text{s}^{-1}$  for medium to high power reactors. Higher neutron fluxes may be required to determine elements in some samples, such as biological tissue. Because of the relation between the minimum perceivable

induced activity and half-life, short and medium half-life radionuclides may be produced in detectable amounts by NAA using low flux reactors. However, many long half-life radionuclides require either a medium to high neutron flux level or a long irradiation time at a low flux to attain sufficiently high induced radioactivities to enable detection within reasonable measurement times.

### 3.1.2. Reactor facility requirements

#### 3.1.2.1. Neutron energy spectrum

The thermal neutron cross-section for most elements is much larger than the epithermal and fast neutron cross-section. In most reactors, irradiation positions are established in the reflector, whether comprising water, graphite or beryllium, outside the core itself or in the beam tubes. In such facilities, the ratio of the thermal flux to the epithermal flux is often larger than a factor of 15, and even in light water moderated reactors, it may approach a factor of 100. In some reactors, especially medium and high flux reactors, facilities exist inside the core itself, in which a high fast neutron flux can also be experienced.

The application of NAA, and especially the elements to be determined or the suppression of elements causing interfering radioactivities, defines requirements with respect to the neutron energy spectrum. For example, the element nickel is often determined with the  $^{58}\text{Ni}(n, p)^{58}\text{Co}$  reaction, which requires epithermal and fast neutrons. A very well thermalized facility thus reduces the sensitivity for that element. On the other hand, determination of the element magnesium is more readily performed in an extremely thermalized facility, since interfering reactions, such as  $^{27}\text{Al}(n, p)^{27}\text{Mg}$ , are largely reduced. A facility with a pure epithermal neutron spectrum can be achieved by covering the irradiation positions with a cadmium sheet one millimetre thick, thus removing all thermal neutrons from the spectrum. This is necessary to suppress reactions such as  $^{23}\text{Na}(n, \gamma)^{24}\text{Na}$  that are mainly induced by thermal neutrons. These conditions reduce the background in gamma ray spectra and increase the signal to background ratio for radionuclides that are also produced by epithermal neutrons. Most of the high power and some of the low to medium power level reactors have an epithermal flux greater than  $10^{10} \text{ cm}^{-2} \cdot \text{s}^{-1}$  and can be used for ENAA.

A thermal column with a well thermalized spectrum is optimal for large sample NAA, as no correction is necessary for self-moderation of the neutrons in the sample. The larger sample mass compensates for the lower flux in the column. However, regular irradiation facilities can also be used, and the correction for self-moderation depends on the composition and size of the sample. Although for samples with a mass of up to several grams, this correction is typically rather small.

For NAA via DNC, any thermal flux that enables fission of thorium or uranium is sufficient.

#### 3.1.2.2. Flux homogeneity, stability and reproducibility

It is very important to determine the neutron flux and the ratio of thermal to epithermal flux in all irradiation sites in terms of homogeneity along the longitudinal and axial gradients. The stability and reproducibility during the operation time of the reactor (e.g. due to xenon poisoning and the position of control rods due to fuel burnup) must also be determined. Samples or groups of samples sandwiched between flux monitors are usually the best approach to obtain reliable quantitative results.

For INAA, it is desirable that the neutron flux is as stable as possible during the irradiation of the sample and standard, and it must be reproducible from one run to another. For studying short lived radionuclides by cyclic INAA, it is essential that the flux is both highly stable and reproducible. In these cases, flux monitoring is normally possible.

#### 3.1.2.3. Sample transfer system

Irradiations for conventional NAA are usually performed using a pneumatic or rabbit sample transfer system, normally available at the reactor, in which use is made of plastic containers called rabbits and sample encapsulation (see Fig. 3.2). In some cases, a hydraulic system is employed. For ENAA, samples can be irradiated in specially built pneumatic sites fitted with thermal neutron shields (e.g. boron, cadmium and gadolinium). Although these shields are sometimes movable, this may have significant effects on the reactivity of the core. The samples may also be wrapped in one of these shields and placed in a conventional rabbit system, but the induced radioactivity

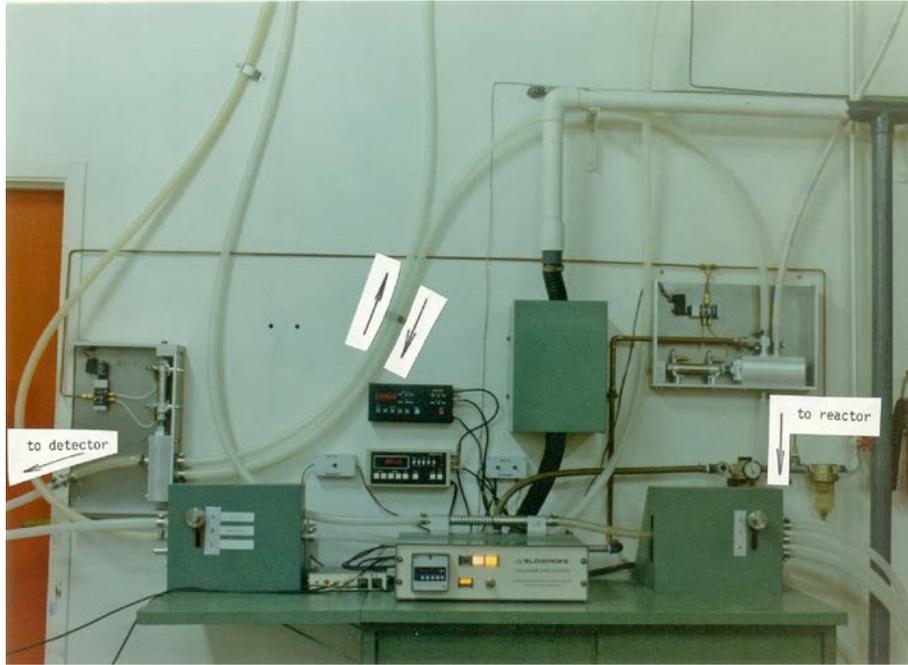


FIG. 3.2. Automated pneumatic rabbit system for short measurements with large batches of samples (courtesy of École Polytechnique de Montréal).

in the cadmium itself or the heat production in the boron shield in such a rabbit can cause serious limitations to usability and irradiation time.

Because of the limited irradiation resistance of plastic, exposure to high neutron fluences greater than  $10^{14}$ – $10^{15}$   $\text{cm}^{-2}$  requires other facilities for NAA, in which samples encapsulated in sealed quartz or aluminium vials can be irradiated. Such facilities are often manually loaded, and may be positioned either in the reflector or inside the core, in a dry or wet tube. Such facilities are often employed for the RNAA method.

NAA based on short lived radionuclides, and especially for half-lives of the order of tens of seconds, requires a rabbit system with a transfer time of one to two seconds. It is essential that this system is installed as close as possible to the reactor. The sample capsule must sometimes be removed from the rabbit prior to measurement, but occasionally the induced radioactivity can be measured without opening the rabbit. This also facilitates cyclic NAA. A difficulty often arises in the  $^{28}\text{Al}$  contamination of the rabbit resulting from wear inside the aluminium transfer tube. The use of carbon–carbon composites for the zone near the core and plastic tubing in the inactive zone has been shown to eliminate this problem. Consideration must be given to the type and level of radiation in the surrounding areas, so that the gamma ray spectrum of a sample is not influenced by the background radiation. The main background radiation inside reactor buildings is often due to  $^{41}\text{Ar}$  in the air surrounding the detector, either by release from the reactor pool water or directly from the transfer system if compressed air is used. This  $^{41}\text{Ar}$  cannot easily be removed by shielding without introducing backscattering effects. Furthermore, the  $^{41}\text{Ar}$  in the air of the sample container results in background interference.

A fast rabbit system can also be used for DNC, as the half-lives of the delayed neutrons vary in the range of 0.4–55 s. DNC requires a counting station different from that for normal NAA, and systems are often equipped with a divider for sample transfer to different measurement stations for flexibility in the use of the facility. Since the entire analysis requires typical irradiation times of at most one minute, decay times of one second and counting times of at most thirty seconds, most facilities are equipped with automated loaders so that a high throughput can be obtained.

The sizes of the vials that can be used for INAA depend on the sizes of the rabbits, while the rabbit sizes and the installed rabbit systems are interdependent. Large sample irradiations are usually done outside the immediate vicinity of the reactor core (e.g. in the thermal column), whose sizes vary depending on the reactor type.

### 3.1.3. Space requirements

Space appropriate for the samples being analysed is required for sample preparation, which comprises sample size reduction, drying, weighing and encapsulation. Contamination must be avoided for all samples, and thus conflicting activities, particularly during preparation of geological samples or mineral residues and handling of biological material, must be well separated. Contamination can also be minimized by performing sample preparation in a laminar flow hood or a separate fume cupboard. In some cases, a clean room may be more appropriate. A separate balance weighing room, with the balance placed on a special table, is also very convenient. Facilities must have the means to monitor and, if possible, control temperature and humidity.

A counting room for gamma ray spectrometers and a room for the storage of irradiated samples are also required. The sizes of the rooms depend on the extent of activities. In the design and assignment of rooms for counting systems, the floor must be able to support the weight of the lead shields. Filters may be required to minimize noise and ground loops in the electricity mains supply. Counting room temperature must also be monitored and preferably controlled. If the NAA facility is outside the reactor building, a ventilation system with appropriate filtration and connection to stack is necessary to prevent the spread of contamination and activity.

The measurement of radionuclides with half-lives of less than a few minutes and the cyclic NAA procedure require space for the spectrometers at the receiving end of the fast pneumatic transfer system.

### 3.1.4. Experimental equipment requirements

#### 3.1.4.1. Counting equipment

At least one gamma ray spectrometry system is needed for NAA, such as the counting station in Fig. 3.3. This system consists of a germanium semiconductor detector connected through its integrated preamplifier to either a modular or integrated analogue signal processing system comprising a high voltage power supply, a spectroscopy amplifier, an analogue to digital converter (ADC) and a pulse height MCA. A digital signal processor (DSP) system and an MCA can accomplish the same functions. Personal computers (PCs) are equipped with plugin units and the associated software by which the MCA is modulated. Dead time correction units, whether integrated in a DSP or available as 'loss free counting' units, are indispensable for measurements of radionuclides with short half-lives, in which a significant change of the count rate occurs during measurement.

Various forms of germanium detector configurations are suitable for NAA, such as horizontal and vertical cryostats. Coaxial and well type detector crystals are used in practice. The latter type provides the highest photopeak efficiencies, but at the cost of complications in the spectrum analysis. A detector with high resolution (FWHM <1.8 keV) — providing a near Gaussian peak shape (FWHM/FW0.1M <1.9), high relative efficiency (>35%) and a high peak to Compton ratio (>60:1) — is desirable. The germanium detector cryostat is mounted in a liquid nitrogen dewar that must be refilled at least once a week.<sup>1</sup> A recent trend favours new dewar types with either significantly lower liquid nitrogen consumption or electrically cooled detectors. The latter types are important if a laboratory is not guaranteed a regular liquid nitrogen supply.

Detectors need to be surrounded by lead shields to reduce the influence of the natural gamma ray background and to minimize cross-interference if more than one detector system is placed in a counting room.

Compton suppression systems are useful to reduce the Compton background, typically a maximum factor of 8–9, resulting from scattered high energy gamma rays. This can improve the minimum detectable amounts or the uncertainty in the detectable amounts, as well as reduce spectral interferences in certain cases. This gain is not the same for peaks in all ranges in the gamma ray spectrum, and is typically optimal only in the energy range of 400–800 keV.

Gamma ray spectrum analysis and interpretation software is needed. Laboratories can develop this themselves, purchase it in conjunction with MCAs or obtain it through the IAEA.

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<sup>1</sup> It is not a good practice to rely on the dewar's 'guaranteed' holding time of three weeks, since, in many cases, after two weeks there might be a high risk that the cryostat is insufficiently cooled, causing damage to the germanium crystal.



FIG. 3.3. Neutron activation analysis counting laboratory (courtesy of the Nuclear Research Center, Egypt).

The counting system for DNC consists of a moderator assembly of typically water free oil, paraffin or polyethylene surrounding the sample or receiving end of the rabbit system. Neutron detectors such as  $\text{BF}_3$  counters can be inserted inside this assembly. Standard electronics are required for the differentiation of pulses resulting from neutron interactions from those resulting from interactions with gamma radiation. An example of a counting system can be seen in Fig. 3.4.

A survey meter is necessary for the counting and the radioactive sample storage rooms. Personnel monitoring is required for each of the experimenters. Access to an oscilloscope (100 MHz is sufficient) is also very useful for setting up the equipment and troubleshooting.

#### 3.1.4.2. Sample loader and sample changer

If a large number of samples are analysed for radionuclides with medium half-lives, long half-lives and very long half-lives, a sample changer (see Fig. 3.5) can be used to increase the efficiency of operation and to increase the throughput capacity, as samples can also be counted at night and during weekends. A changer can also be used to provide a more reproducible counting geometry. Many facilities have designed and constructed their own sample changers for a modest cost. The cost for a commercial sample changer to be used with a conventional gamma ray spectrometer can be US \$80 000 or more. A sample changer for use with a Compton suppression system is at present not commercially available.

It should be noted that additional software may have to be developed for automation of the data processing if more samples are being counted and analysed.

#### 3.1.4.3. Laboratory equipment and utensils

For sample preparation, NAA laboratories require standard laboratory equipment such as:

- Rotary mill or pulverizer for sample size reduction and homogenization;
- Oven for drying, with a typical temperature range up to  $150^\circ\text{C}$ ;
- Freeze drier for drying biological tissue or samples in which the analyte is volatile;
- Microbalance with a resolution in the range of 0.01–0.1 mg;
- Micropipettes and volumetric glassware.



FIG. 3.4. Neutron activation analysis counting laboratory at the RSG-GAS research reactor in Serpong, Indonesia (courtesy of BATAN).



FIG. 3.5. Automatic sample changers in the laboratory for instrumental neutron activation analysis (courtesy of Delft University of Technology).

Calibrated radionuclide sources, either sets of single radionuclides or mixed sources, are required for gamma energy calibration and detector efficiency calibration.

Standards are required for element calibration. These calibrators may be derived from either single element chemical compounds, such as oxides or pure metals, or certified reference materials including single element

solutions. They serve both for calibration by the reference or single comparator method. Similarly, chemical compounds or metal foils may be the basis for neutron flux monitors.

Other utensils may include encapsulation materials, such as vials, plastic bags or quartz ampoules, and distilled water or ethanol, for example, for cleaning.

Of course, the special standardized software programs for analysis of spectra obtained from detectors are highly desired.

### 3.1.5. Personnel requirements

Although, in principle, one person can easily perform NAA, at least two people are required to operate an NAA facility. Ideally, one person would have a background in chemistry and the other a background in nuclear physics. Both people must be trained in NAA principles, including sample preparation, detector calibration and flux measurement, spectrum analysis and interpretation, and monitoring of sources of error. The latter is a basis for the indispensable implementation of appropriate internal quality assurance and quality control (QA/QC) procedures. Technical services need to be available to support certain evolutions such as development and maintenance of sample changers. The actual number of people required depends on the amount of work that has to be done. In some reactor facilities, students and experimenters are trained to irradiate and count their own samples while the reactor is in operation.

It is relatively easy for reactor facilities to insert, irradiate and remove NAA samples from the reactor and for experimenters to then count and to analyse these themselves. There are many users who do not have the background or training to perform NAA, nor have any desire to learn, but who nevertheless would like to make use of the technique. To this end, a research reactor facility may well gain a significant number of additional users if it provides a complete NAA service involving sample preparation, irradiation, counting and data reduction, with just the analytical results returning to the customer. Such an effort will clearly require at least one or two additional staff, depending on the total sample throughput. Appropriate QA/QC has to be operational to ensure the degree of accuracy of the results and to enhance international acceptability.

### 3.1.6. Funding requirements

It is assumed here that a pneumatic irradiation system exists in the reactor and that additional facilities, other than those described above, are optional.

At the end of 2011, germanium detector prices ranged from US \$30 000 to 100 000 for detectors with 30–100% relative efficiency. A well type detector typically costs in the order of US \$50 000. Analogue signal processing units, consisting of a high voltage supply, spectroscopy amplifier and ADC, cost around US \$15 000, and the cost of the newest DSP is approximately US \$13 000–20 000. A commercial lead shield costs around US \$25 000. A Compton suppression system with a 30% germanium detector, a scintillation guard detector and the associated electronics costs around US \$200 000–250 000, depending on whether a NaI(Tl) scintillator crystal or a bismuth germanate (BGO) scintillator crystal is used.

An additional indication of typical costs for support equipment is provided as a guideline:

- Sample changer: US \$50 000–80 000;
- Lead irradiation shield: US \$15 000–30 000.

Other peripheral equipment that may be considered includes:

- Electrically cooled detectors: US \$20 000–35 000 more expensive than liquid nitrogen cooled ones;
- Microbalance (five decimal): US \$20 000;
- Freeze drier: US \$80 000;
- Oven: US \$3500;
- Micropipette: US \$500;
- Sample size reduction machines: starting at US \$6500;
- Software: US \$15 000–25 000, depending on the extent and number of PC licences.

For a DNC counter, the moderator tank must be designed and built in-house. Depending on the experimental complexity, several (typically six to eight)  $\text{BF}_3$  counters may be needed. Costs will obviously relate to the final facility set-up, and can be as much as several tens of thousands of US dollars. Typically, associated electronics costs approximately US \$30 000.

### 3.1.7. Customer relations

Customers interested in NAA may originate from industries such as mining or agriculture, trade companies, government agencies, medical centres, universities and research institutions, and may have diverse needs relating to products and services available. Sometimes, an entirely distinct perception of what NAA has to offer as an analytical technique exists, and customers consequently only consider its applications when all other techniques have failed. In addition, customer expectations of quality do not always correspond to the same understanding of trueness and precision for the analysis results. Although it is expected that a facility provides good performance with respect to such trueness and precision, in many cases the analysis results need to be simply ‘good enough’, according to customer requirements. This is often a difficult approach for academic facilities — that is, to optimize analyses to provide a sufficient service rather than the best and often most expensive measurement capabilities possible.

In certain cases where quick turnaround times are required, adaptation of INAA measurement techniques using modern counting equipment with higher counting rates is possible. Although analyses are traditionally performed one to three weeks after irradiation, reasonably good results can be obtained some ten to twelve days after irradiation (e.g. when the  $^{24}\text{Na}$  background has reduced substantially). Similarly, satisfactory results can be obtained after a three day decay rather than waiting a full week after irradiation, all contributing to a quicker and higher throughput with associated improved customer satisfaction. Similar consideration applies to precision, for example in many cases, a 5% counting statistics precision is sufficient, and, in fact, a 20–30% precision may suffice. For example, an indicative result of 0.1, 1, 10 or 100 mg/kg of a certain element may be readily obtained after a few minutes of counting, even though the uncertainty of the measurement is still of the order of 50% or higher.

Although INAA provides multi-element determination up to 50 to 60 elements, customer needs may be limited to only one to three elements. The resultant tailoring of the analytical protocol of irradiation, decay and counting times enables a faster and more cost efficient service. On the other hand, customers from universities who initially request a large number of elements (e.g. for multivariate analysis applications) may reduce the requested number of elements rapidly when fully charged for the analyses. Typically, external customers offer their samples in small batch sizes and seldom in large quantities (e.g. hundreds) at a time.

DNC is a valuable technique to determine thorium and uranium. The short duration of the analysis procedure makes the facility very suitable, once the rabbit system is equipped with a sample changer, for analysis of large series of samples, for example in the frame of exploration studies in geology and mining. Typical sensitivities that can be achieved are 5  $\mu\text{g}$  for thorium and 50 ng for uranium in a 5–10 g sample.

It must be borne in mind that the reliability of providing a good service does not only depend on the analytical quality, it also depends on the availability of facilities, particularly when compared to alternative non-nuclear applications (e.g. X ray fluorescence spectroscopy, automatic absorption spectroscopy and inductively coupled plasma spectroscopy).

The bottom line is that paying customers expect sufficient levels of quality management and laboratory accreditation, ensuring that analyses can be reliably reproduced and, if required, that the laboratory be able to defend its results in court.

## REFERENCE TO SECTION 3

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## 4. PROMPT GAMMA NEUTRON ACTIVATION ANALYSIS

Prompt gamma neutron activation analysis (PGNAA) is an elemental analysis method similar to INAA (see Section 3). While INAA uses the radioactivity emitted by radionuclides produced during neutron irradiation for analysis, PGNAA uses the prompt gamma rays emitted during the process of neutron capture.

### 4.1. APPLICATIONS OF PROMPT GAMMA NEUTRON ACTIVATION ANALYSIS

PGNAA is being increasingly used, limited only by the scarcity of suitable neutron beams. The list of measurable elements emphasizes the low  $Z$  and high abundance elements such as boron, carbon, chlorine, hydrogen, nitrogen, phosphorus and sulphur in organic and geological materials and the high cross-section elements such as boron, cadmium, gadolinium and samarium. The analysis for boron and hydrogen is especially important because of the paucity of other reliable analytical techniques for trace levels of these elements. PGNAA is extremely sensitive for the quantitative determination of boron compared with destructive chemical techniques, particularly since boron is such an important element found in a wide range of applications, from meteorites to human tissue.

#### 4.1.1. Flux/power level requirements

The prompt gamma method ideally requires a well thermalized or, preferably, a cold neutron beam with a flux greater than  $10^8 \text{ cm}^{-2}\cdot\text{s}^{-1}$  over an area of several square centimetres. However, with careful attention to reactor induced gamma ray and fast neutron background, productive systems have been operated with a flux as low as  $10^6 \text{ cm}^{-2}\cdot\text{s}^{-1}$ .

#### 4.1.2. Reactor facility requirements

A beam tube is required to perform PGNAA. A tangential tube is preferred to a radial tube for a better thermal to fast flux ratio and neutron to gamma ray flux ratio, and thus an improved signal to background ratio. Figure 4.1 shows the thermal neutron PGNAA facility at the National Institute of Standards and Technology, which uses a vertical beam from the reflector.

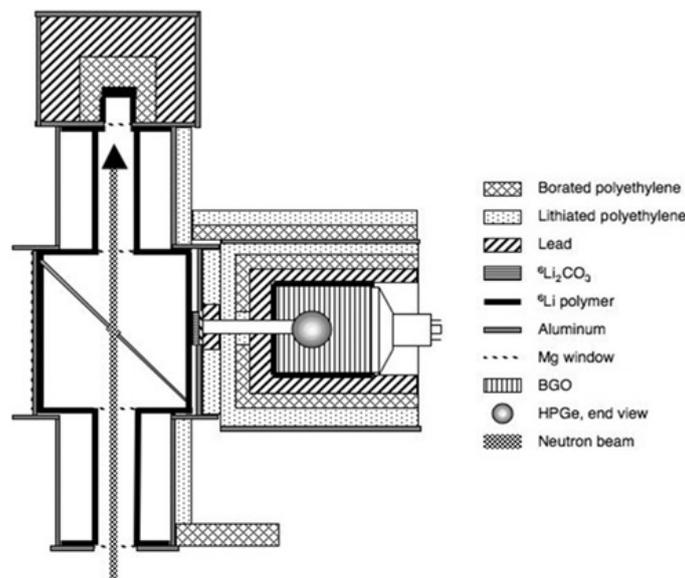


FIG. 4.1. Thermal neutron prompt gamma neutron activation analysis facility (courtesy of the National Institute of Standards and Technology).

The most effective PGNAA systems are operated with neutrons provided by a neutron guide because they — particularly bent guides — deliver a much cleaner flux to the sample, giving this improved signal to noise ratio.

Most practitioners prefer a PGNAA facility at the end of the neutron beam as shielding against scattered radiation from other beam users. Neutron focusing devices have been developed that allow for position sensitive detection of elements; typical resolution is about 0.1 mm at full width at half maximum (FWHM).

#### 4.1.3. Experimental equipment requirements

Similarly to NAA, a high resolution gamma ray spectrometer using an n type germanium detector is required, which should include a computer based MCA with software to control data acquisition and to analyse spectra. As capture gamma rays have energies up to 11 MeV, at least 16 000 data channels are required. The detector must be well shielded to minimize neutron activation of the detector itself and to reduce the gamma ray background components. The specification for an n type detector is based on the lower sensitivity for damage by scattered neutron absorption. The shielding against these stray neutrons, which may cause serious damage to the germanium detector, is best done with enriched  ${}^6\text{Li}$  containing compounds such as  ${}^6\text{Li}_2\text{CO}_3$ ,  ${}^6\text{LiF}$  or  ${}^6\text{Li}$  silicate glass, as no capture gamma rays are produced when neutrons are absorbed. Boron and cadmium compounds are also efficient neutron absorbers, but they produce prompt gamma rays upon neutron capture.

In addition, a Compton suppression system is a desirable option, although this is not necessary with larger germanium detectors.

Targets may be irradiated in air, but when low amounts of hydrogen or nitrogen must be determined, a sample changer either evacuated or filled with helium gas is preferred.

Measurement times depend on both the amounts to be measured and the intensity of the neutron beams, and may vary from minutes to days. The neutron fluence has to be monitored during the irradiation, which can be done, for example, by activating a suitable target and counting the induced activity. In principle, it is possible to design a sample changer for PGNAA facilities, especially when the reactor operates continuously.

#### 4.1.4. Personnel requirements

The establishment and operation of a PGNAA facility requires a nuclear physicist or chemist. Once functional, a well trained technician can perform routine operations, as the major part of the work is gamma ray spectrometry. Cooperation and coordination with other analytical groups, especially those using INAA and neutron scattering, will enhance the operation and utilization of the PGNAA facility. Mechanical engineers are desirable for the design of filters, collimators, the sample chamber and the shielded detector assembly. If a cold source is used, a person experienced in the design, installation and operation of such a source is required.

#### 4.1.5. Funding requirements

The costs for establishing a PGNAA facility vary depending on the effort expended to create a high quality neutron beam and efficient neutron and gamma ray collimation and shielding. Some typical costs in Europe and the United States of America are given for various aspects. Depending on how costs are allocated, the design, construction and installation of a PGNAA facility at an existing neutron beam tube or guide may cost US \$200 000–400 000, including:

- Thermal beam filters and collimators: around US \$5000–10 000.
- A germanium gamma ray detector with electronics: around US \$60 000.
- The sample station and detector shield: around US \$20 000.
- A PC based MCA: around US \$20 000.
- Advanced computer gamma analysis software: around US \$3000–5000.
- A pair spectrometer or Compton suppression system: around US \$150 000.
- A neutron focusing device. Focusing neutron lenses consist of a large array of bend capillary glass tubes (polycapillary lenses). They are built by a few suppliers on demand only, and hence a price estimation is difficult, although a budget of at least US \$50 000 may be required.

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## 5. RADIOISOTOPE PRODUCTION

Radioisotopes have a wide range of applications in various fields, including nuclear medicine, industry, agriculture and research. They are produced mainly in research reactors and accelerators, involving several interrelated activities such as target fabrication, irradiation, transportation of irradiated targets to processing facilities, radiochemical processing or encapsulation in sealed sources, quality control, and transportation to end users. The production of radioisotopes in reactors is based on neutron capture in a target material (i.e. either by activation or generation of radioisotopes from fission of the target material by bombardment with thermal neutrons). Research to develop new radioisotopes for diagnostics and therapy in nuclear medicine, non-destructive testing and radiotracer industrial applications, as well as for radiotracer studies in scientific research, provides excellent participation opportunities for research reactors and cyclotrons.

Radiopharmaceuticals, such as those extracted from generators (see Fig. 5.1), are substances that contain a radioisotope and which have the ability to perform the role of a marker in medical diagnostic or therapeutic procedures. Globally, the number of medical procedures involving the use of radioisotopes is growing, with an increasing emphasis on radionuclide therapy using radiopharmaceuticals for the treatment of cancer. These socioeconomic benefits, as well as the effective quality control processes and cleaner environments provided by supporting technology, strengthen national, regional and international capabilities of the reactor.



FIG. 5.1. Examples of radioisotope generators (courtesy of the Institute of Atomic Energy POLATOM).

Significant changes have taken place in many aspects of radioisotope production during the last few years, in particular, regarding the situation with research reactor facilities. Many research reactors worldwide are ageing rapidly, and have either ceased operation or are due for shutdown and replacement by new ones, or are being subjected to costly modernization in the near future. On the other hand, considerable experience and knowledge have been gained in the production of important radioisotopes as well as research by medical investigators. The discoveries of new nuclear medicine procedures for diagnostics, therapy and palliation have had a remarkable influence on the introduction of many new radioisotopes. The resultant collaborative research of nuclear medicine investigators, reactor operators and processing laboratories has led to success in establishing several new and challenging technologies for radioisotope production using research reactors.

Radiochemical separation techniques, including ion exchange and solid phase extraction chromatography, as well as high performance liquid chromatography techniques with on-line radioactive detectors, are commonly used for the separation of useful radioisotopes from nuclear reactor activated targets. The production of radioisotopes in a nuclear reactor relies on selective fluxes available in the various sized reactors, from low to high thermal fluxes. A wide range of radioisotopes for medical and industrial applications that can be made available is given in Annex II.

Radioisotope production from nuclear reactions on targets may often require further complex processing if highly active sources are produced (e.g. for medical applications). This often involves significant investments in hot cell facilities and associated processing equipment. In many cases, it will not be necessary to provide such processing facilities at the reactor site, but rather it will be sufficient to sell the irradiated targets to processing companies. In that case, only the equipment for packaging and the expertise for the transportation of dangerous goods according to international regulations will be required from the reactor operator.

Consequently, research reactors remain an essential source of radioisotopes in several fields including medicine, industry, agriculture and research. Recently, the Maria reactor in Poland and the LVR-15 reactor in the Czech Republic have undertaken qualification programmes for  $^{99}\text{Mo}$  production to diminish the risk of a supply shortage for the daughter radioisotope  $^{99\text{m}}\text{Tc}$ , which is used in approximately 80% of diagnostic medical scans. In addition, specifications for the testing procedures for ensuring quality end products form an essential part of any radioisotope production programme.

## 5.1. RADIOISOTOPE PRODUCTION IN RESEARCH REACTORS

Normally, three parameters are important for final radioisotope products:

- (1) Specific activity: The number of radioactive atoms per given volume or mass. The specific activity of produced radioisotopes depends on the physical and chemical properties of the target as well as on the irradiation conditions, and is presented in greater detail in the following sections. In particular, for widely used radioisotopes, an essential irradiation parameter is the neutron flux density available in the irradiation channel.
- (2) Chemical purity: The contamination by stable nuclei, which should not exceed the limit specified by the end user.
- (3) Radiochemical purity: The contamination by other radioactive nuclei, which should also not exceed the limit specified by the end user.

## 5.2. METHODS OF RADIOISOTOPE PRODUCTION

### 5.2.1. Radioisotopes produced in nuclear reactors

Target materials to be irradiated are encapsulated in suitably designed irradiation containers and then loaded using proper tools in predetermined locations in the reactor core or reflector for irradiation. In most pool reactors, the core is readily accessible, and the loading and unloading of targets can be carried out from the top of the pool using simple devices or remotely operated installations such as pneumatic or hydraulic rabbit systems. The irradiated targets are then chemically processed, packaged and loaded into appropriate shielding containers and transported to radioisotope processing laboratories. This process is illustrated in the global supply chain of radioisotope  $^{99}\text{Mo}$  depicted in Fig. 5.2.

### 5.2.2. Flux/power level requirements

Research reactors can be classified in the context of radioisotope production into three main categories according to the available thermal neutron flux: low flux reactors, medium flux reactors and high flux reactors. Generally, the neutron flux level determines the types of radioisotope and the related specific activities that can be produced in a particular research reactor. The commercial production of most radioisotopes requires research reactors with higher neutron fluxes. Lower flux facilities typically only produce lower activities of radioisotopes for local needs, which nonetheless could be of some commercial interest.

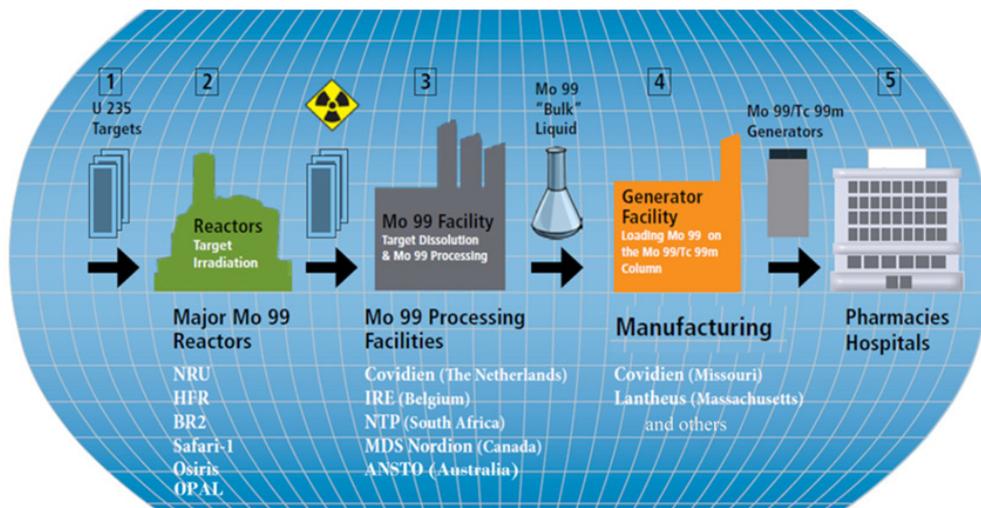


FIG. 5.2. Global supply chain of  $^{99}\text{Mo}$  for  $^{99\text{m}}\text{Tc}$  generators (adapted from an image from Covidien).

The irradiation process for the production of radioisotopes generally involves well defined levels of flux, as well as accurate irradiation exposure times; the combination of these two parameters ultimately provides the necessary neutron fluence. In low flux reactors ( $<10^{12} \text{ cm}^{-2}\cdot\text{s}^{-1}$ ), the resultant fluence available is also often low because of limitations on the reactor's operational periods (e.g. only for one shift). In addition, one shift operation limits the isotope production of intense sources to short lived radioisotopes such as  $^{41}\text{Ar}$ ,  $^{198}\text{Au}$ ,  $^{38}\text{Cl}$ ,  $^{64}\text{Cu}$ ,  $^{56}\text{Mn}$  and  $^{24}\text{Na}$ . Typically, these orders do not require more than around 100 MBq in activity. However, these reactors can produce, in one such shift irradiation, sufficiently active sources of longer half-lives for application as radiotracers in scientific research programmes in, for example, chemistry and biology.

Reactors with medium flux ( $10^{12}\text{--}10^{14} \text{ cm}^{-2}\cdot\text{s}^{-1}$ ) generally operate for longer cycles, and such operational schedules could allow for the production of more marketable radioisotopes. In addition to those listed for the low flux reactors above, it should be possible also to produce the following:  $^{60}\text{Co}$ ,  $^{51}\text{Cr}$ ,  $^{125}\text{I}$ ,  $^{131}\text{I}$ ,  $^{192}\text{Ir}$ ,  $^{177}\text{Lu}$ ,  $^{99}\text{Mo}$ ,  $^{186}\text{Re}$ ,  $^{35}\text{S}$ ,  $^{153}\text{Sm}$ ,  $^{133}\text{Xe}$ ,  $^{90}\text{Y}$  and  $^{169}\text{Yb}$ .

In the case of high flux reactors ( $>10^{14} \text{ cm}^{-2}\cdot\text{s}^{-1}$ ), such as the five major producers of  $^{99}\text{Mo}$ , including the BR2 reactor of Belgium (see Fig. 5.3), the requirements of their experimental users and radioisotope production are complimentary during typical operation cycles of three to four weeks. In addition to the above radioisotopes with higher specific activities, production of the following isotopes should also be possible:  $^{252}\text{Cf}$ ,  $^{75}\text{Se}$ ,  $^{117\text{m}}\text{Sn}$ ,  $^{89}\text{Sr}$  and  $^{188}\text{W}/^{188}\text{Re}$ .

In particular, the production of isotopes requiring the sequential capture of two neutrons (e.g.  $^{186}\text{W}(n, \gamma)^{187}\text{W}(n, \gamma)^{188}\text{W}(\beta^-)^{188}\text{Re}$ ) requires a high flux reactor, since the efficiency of production is given by the square of the neutron flux density.

### 5.2.3. Reactor facility requirements

Radioisotope production usually requires not only a reactor or facility as the indicated source of neutrons, but also additional processing ability in production lines. This typically can involve major investments in shielded gloveboxes up to equipped hot cells for the related radiochemical processing. Strategic planning and feasibility evaluations for such facilities are thus essential prior to embarking on such a project. Furthermore, before making this a major part of the strategic plan for the facility, the viable production of radioisotopes needs to incorporate the evaluation of several economic aspects. These might include the requirement to analyse international market prices, evaluation of the market need situation and establishment of contact with potential users to identify the customer base. In many cases, however, it is appropriate for the reactor operator to partner with industrial companies and to provide only the irradiation service, whereas the processing to form the desired final product is carried out by the partner or customer.

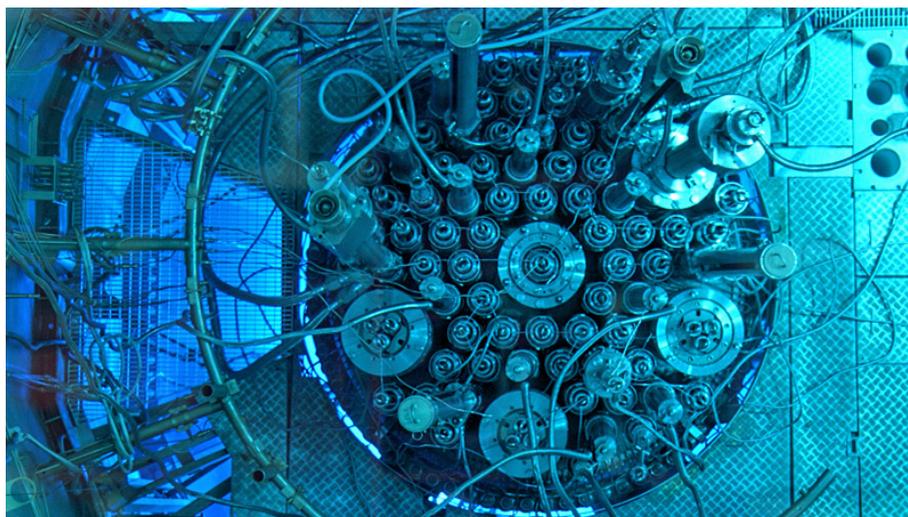


FIG. 5.3. View from above the high flux reactor core (courtesy of the Belgian Nuclear Research Centre).

#### 5.2.3.1. Target preparation and encapsulation

Targets are to be encapsulated suitably before being introduced into the reactor irradiation channel. The majority of irradiation facilities at high flux reactors make use of targets encapsulated in aluminium capsules, whereas in low and medium flux reactors, both polyethylene and quartz vials can be used, depending on fluence and temperature. Polyethylene and quartz are easy to handle because of the low induced radioactivity. Aluminium is chosen because the absorption cross-section is low and the material has good thermal conductivity. The purity of the aluminium must be taken into account upon estimation of the induced radioactivity in aluminium and, consequently, the cooling time before handling the unshielded irradiated aluminium capsule. The primary radiological limitation is the contribution of  $^{24}\text{Na}$ , whether produced by means of the threshold reaction  $^{27}\text{Al}(n, \alpha)^{24}\text{Na}$  or at insufficient purity by the formation of  $^{70}\text{Ga}$ ,  $^{72}\text{Ga}$ ,  $^{65}\text{Zn}$  and  $^{69\text{m}}\text{Zn}$ . An additional advantage of aluminium is the relative ease in making such capsules airtight and watertight by cold rolling or welding. In special irradiation applications, some facilities make use of zirconium, titanium or stainless steel capsules.

Quartz or silica ampoules are easily sealed by a glass blower. Some procedures require that both capsules and ampoules are pre-cleaned in an ultrasonic bath and oven dried. The integrity of the seal weld is often subjected to a quality inspection for leak-tightness, which can be carried out by submersion in water.

Metals or their oxides remain the preferred target materials, often incorporating the form of wire, pellets or plates. Irradiation capsules are often filled with targets in powder, powdered metal, sponge or molten form. The latter case involves materials such as sulphur or tellurium dioxide, which are melted and carefully poured into an aluminium capsule. The capsule is then cooled and sealed prior to irradiation.

In summary, the method of target encapsulation generally depends on:

- Physical form of the target (solid, liquid or gas);
- Characteristics of the target;
- Duration of the irradiation;
- Design of the irradiation assembly in the reactor;
- Type of coolant used in the reactor;
- Post-irradiation handling or end use of the radioisotope.

The reactor facility can develop encapsulation techniques for the applicable ranges of fluxes, fluences and irradiation environments available. After irradiation, a shielded device, often in a fume cupboard, must be available for opening irradiation capsules safely. Procedures such as capsule loading, array, welding, welding seam control,

marking, unloading and storage inventory are generally fully or semiautomated processes contributing to reliability and precision, as well as decreased radiation dose and increased production capacity.

### 5.2.3.2. Irradiation conditions and limitations

As mentioned above, conditions during target irradiation are generally determined by the parameters of neutron flux such as neutron flux density, neutron spectrum, time of irradiation and cooling time. The irradiation periods can cover a wide range, from minutes to weeks (e.g. irradiation of sublimated sulphur for  $^{32}\text{P}$ ) or even up to months of irradiation (e.g. gadolinium (III) oxide for  $^{153}\text{Gd}$  or mercury (II) oxide for  $^{203}\text{Hg}$  for industrial applications). The heat generated in target materials also determines and often limits the irradiation conditions.

The duration of the reactor operation cycle, type of decay, half-life of the final product and expected specific activity are interrelated with the radioisotope production capabilities, particularly where the targets can only be loaded or unloaded when the reactor power has been lowered. In addition, the radiochemical processing schedule, as well as the requirements of the radioisotope end user, must often be taken into consideration during the planning of the reactor operation cycles.

It is often necessary that a specific target irradiation is performed in pure thermal or fast neutron flux. These conditions are created using special shields (e.g. boron or cadmium layers) around irradiated samples. However, the limitation of self-shielding effects in irradiated targets can be achieved using special graphite containers with the targets placed inside the capsules (see Fig. 5.4).

### 5.2.3.3. Irradiation facilities

In most cases, irradiation channels in the reflector are selected for radioisotope production by thermal neutron fluxes. In some cases, however, irradiation positions are located inside the fuel element region to take advantage of the available fast neutron fluxes. Therefore, it might be beneficial to have capabilities for both thermal and fast neutron irradiations. Neutron flux traps and a variety of other irradiation facilities are often used in reactors, where desirable. Pneumatic or hydraulic transfer systems enabling the irradiation of samples in holders (e.g. rabbit capsules) in the core or in beam tubes constitute basic reactor irradiation equipment and support devices.

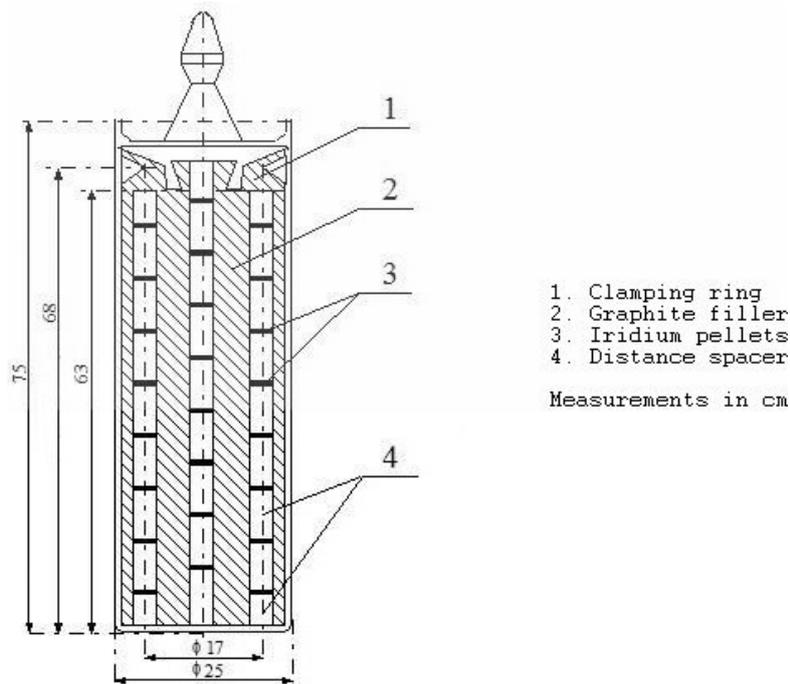


FIG. 5.4. Graphite capsule for the irradiation of iridium pellets, an example of a special design to minimize the self-shielding effect during irradiation (adapted from an image from the Institute of Atomic Energy POLATOM).

Hydraulic transfer systems which allow the transfer of irradiated capsules from the reactor core to a nearby hot cell or processing area during full power reactor operation are useful — in particular for the production of radioisotopes based on short irradiation periods.

The irradiation facility needs to be equipped with special handling tools, a storage place for decay and a decontamination facility. An important aspect of the system is the monitoring equipment, as this often dictates the quality of the product. SPNDs can form a positive contribution to the flux monitoring for short irradiations at in-core irradiation channels.

It should be borne in mind that some irradiation procedures require specially equipped irradiation channels (e.g. for the irradiation of a xenon target). Natural xenon gas is initially inserted into an aluminium capsule and sealed using special equipment. The pressure in the target capsule containing natural xenon can increase by up to 50 kg/cm<sup>2</sup> during irradiation, and water cooling of the target within the irradiation channel is necessary for the full duration of the irradiation. After irradiation, a cooling period of at least 120 hours is required for the  $\beta^-$  decay to permit the formation of <sup>125</sup>I. In addition to this, a further cooling period of 30 to 40 days is necessary so that the <sup>126</sup>I content can decrease to an acceptable level.

Particular attention must be paid to radioisotopes produced as fission products from the nuclear fuel (e.g. <sup>99</sup>Mo/<sup>99m</sup>Tc) because the heat produced during the irradiation of nuclear fuel increases up to levels of several hundred kilowatts and requires reliable forced cooling for removal. In addition, movement of these samples could have a serious impact on the reactivity of the reactor upon rapid or undesired insertion or removal, which could lead to an unscheduled automatic shutdown initiated by reactor safety instrumentation.

#### 5.2.3.4. Hot cells, handling and transportation systems

Depending on radioactivity levels, the preparation of selective radioisotopes, as in sealed sources, can be carried out in shielded conventional hot cells supplied with master–slave manipulators, or even with tongs in special shielded gloveboxes.

The hot cells generally used for handling and preparation of radioisotopes for transportation typically include master–slave manipulators and shield windows, and are equipped with a dedicated ventilation system monitoring the radioactivity in the exhaust gas. The cell should preferably be lined, or coated, with a material that facilitates decontamination. A waste containment box inside the cell needs to be provided to minimize the spread of contamination. Additional equipment can include a conveyor for interconnecting with other hot cells, a connection port with the reactor pool, an overhead shield plug to enable the removal of the hot cell containment box and equipment and shield doors. In general, special tools which can be used with master–slave manipulators are necessary. In some cases, protection of the cell and related equipment against heat and corrosion effects may also be required.

Usually, a new hot cell is not easily installed at existing reactors owing to the difficulty of integration with the reactor pool and because of the required infrastructure, including ventilation and structural foundations.

#### 5.2.3.5. Reactor safety evaluation

Prior to introducing any targets into the reactor for irradiation, the following need to be evaluated:

- Reactivity effects;
- Nuclear heating effects;
- Radioactivity produced in the target;
- Pressure buildup from decomposition;
- Radiation shielding requirements for safe handling and transport.

Significant issues related to safety analysis and licensing or regulatory requirements must be addressed prior to radioisotope production. Possible abnormal occurrences during the production process must be taken into account, and assurance must be given that all resultant conditions are within the bounds of the reactor design basis and the operation limits and conditions. The first production of radioisotopes at a facility typically requires the attention of several professionals, including engineering and radiochemical specialists to ensure that procedures are established correctly and subsequently implemented by well trained personnel.

#### 5.2.3.6. *Quality assurance and measurement systems*

It is essential that well established QA/QC programmes are in place for the reactor and isotope processing facilities before any commercial work in this field can be successfully implemented. For example, a gamma ray counter or spectrometer and selective analytical and measuring systems are usually required to provide reliable measurements of isotopic content, final product purity and radiological clearance levels for customer and transport requirements.

Industrial partners increasingly require complete traceability of the product. Therefore, careful documentation of each irradiation and long term storage of the files are highly recommended.

In addition, the use of radioisotopes requires licensing by a competent authority, and producers need to enquire from their partners about standards for QA/QC systems. For first time users, the operating organization needs to be willing to assist the user in the licensing process. Special requirements are necessary for the use of medical isotopes on humans and animals, and this may require special considerations during the production process.

#### 5.2.3.7. *Radiological protection*

The design of a radioisotope production facility needs to take into account appropriate radiation protection requirements which aim to limit radiation exposures of occupational workers to acceptable limits, both from acute and delayed radiation effects. Adequate shielding (e.g. concrete, lead or iron) around the handling facilities needs to be provided to keep the radiation to acceptable levels. Remote handling devices, such as master–slave manipulators or tongs, can be used for performing operations through the relevant shielding. The training of workers in radiological safety and implementation of proper operating procedures play an important role in maintaining radiologically safe conditions.

#### 5.2.3.8. *Processing facilities*

A number of radionuclides of different half-lives and energies can be produced, along with the radioisotope of interest, during the irradiation of targets. The irradiated target often requires chemical processing to separate the radioisotope of interest, and sometimes additional processing and purification steps to obtain a final product which can be applied effectively and safely for the intended purpose. The radiochemical processing of irradiated targets, where feasible, need to be located in the vicinity of the research reactor.

Radioisotopes can be delivered to users as sealed or open radioactive sources that are either encapsulated in a suitable container or supplied in a form that provides equivalent protection against mechanical disruption and fulfils all safety requirements. Capsules are generally sealed by electric arc welding, while laser welding is often used for sealing miniature sources. The sealing processes must be of high precision to provide high quality welding seams and leaktightness of the capsules. In several instances, the desired technical quality is readily achieved using automated welding machines with capsule rotating tools.

### **5.2.4. Personnel requirements**

In general, the number and expertise levels of the personnel required to operate the radioisotope production and processing facilities are much dependent on the capacity and variety of isotopes to be supplied. Typically, engineers, radiotechnicians, radiological protection staff, handling staff and reactor shift operators are all involved.

### **5.2.5. Funding requirements**

As in the case of the personnel requirements above, the funding requirements for radioisotope production and processing are very much dependent on the extent of the supply chain requirements. Special consideration are to be given to the following activities:

- Design;
- Regulatory approval;
- Commissioning;

- Building design and commissioning;
- Decommissioning;
- Waste and processing;
- Operating staff;
- Fuel consumption;
- Radiological controls;
- Plate purchasing;
- Loss of irradiation facilities for scientific research.

### 5.2.6. Time requirements

Depending on the intended radioisotope applications and available infrastructure, the following expectations for installation and commissioning can be expected:

- Radiotracer applications for science or industry: 6 months.
- Sealed sources: 6–12 months.
- Open sources for small scale medical applications: 6 months–2 years.
- Open sources for commercial medical applications: 2–10 years.

## 5.3. SOME EXAMPLES OF MAJOR RADIOISOTOPE PRODUCTION

### 5.3.1. Molybdenum-99 production

An example of a radioisotope very frequently used to support nuclear medicine diagnostic procedures at present is  $^{99}\text{Mo}$ , which is a source product for the preparation of  $^{99}\text{Mo}/^{99\text{m}}\text{Tc}$  generators. Molybdenum-99 is a fission product obtained from irradiating targets (e.g. in the form of alloyed plates) containing uranium. The uranium can be of various degrees of enrichment, although the international non-proliferation preference at present is to use low enriched uranium (i.e. <20%  $^{235}\text{U}$  by mass). A nuclear processing facility supplies  $^{99}\text{Mo}$  (parent isotope) in a suitable form, which then decays into  $^{99\text{m}}\text{Tc}$  (daughter isotope), with a half-life of around 2.75 days. Owing to its favourable nuclear and chemical properties,  $^{99\text{m}}\text{Tc}$  is the most frequently used radionuclide worldwide and is utilized in nuclear medicine applications for several million diagnostic images annually.

Fission  $^{99}\text{Mo}$  based on chromatographic  $^{99}\text{Mo}/^{99\text{m}}\text{Tc}$  generators has become the most generally used radioisotope in practice owing to its relatively simple processing and handling, reliability of product quality and high radioactive concentration. It can be obtained using three different processes:

- (1) Eluted from fission  $^{99}\text{Mo}$  based on a chromatographic column generator;
- (2) Eluted from irradiated  $^{99}\text{Mo}$  based on a gel generator;
- (3) Separated from irradiated  $^{99}\text{Mo}$  compounds by solvent extraction (see Fig. 5.5).

Selective problems with the handling, transport and delivery logistics of  $^{99}\text{Mo}$  are related to the situation of research reactors and processing facilities, as well as planned reactor operation and shutdown schedules. At the same time, considerable efforts have been made to develop new technologies based on irradiation of low enriched uranium at some reactor centres around the world. One should note that  $^{99}\text{Mo}/^{99\text{m}}\text{Tc}$  generators can be produced for local markets also through neutron activation of enriched  $^{98}\text{Mo}$  targets. New producing reactors encounter large financial investments and strict regulation requirements before establishing fission product  $^{99}\text{Mo}$  production and processing, making market penetration difficult. On the other hand, the radiopharmaceutical industry clearly prefers  $^{99}\text{Mo}/^{99\text{m}}\text{Tc}$  generated as a fission product owing to its higher achievable specific activity.

### 5.3.2. Other new radioisotopes

Lutetium-177 has been touted internationally in the medical community as a new important radionuclide treatment. It emits a beta particle with a moderate energy, which animal experiments suggest would be useful



FIG. 5.5. Technetium-99m solvent extraction unit (courtesy of the Korea Atomic Energy Research Institute).

for treating moderate size tumours while sparing nearby healthy tissue. In addition, it has a gamma photon that allows imaging of its biodistribution in patients. This allows a more accurate estimate of target and critical organ dosimetry.

A new improved process for production of this potential radioisotope has recently been developed for the production of no carrier added  $^{177}\text{Lu}$  of high specific radioactivity using an ytterbium target enriched in  $^{176}\text{Yb}$  nuclides. It has been developed to the production of high purity  $^{177}\text{Lu}$  radionuclides through the nuclear reaction  $^{176}\text{Yb}(n, \beta^-)^{177}\text{Lu}$  as high as tens of GBq. Lutetium-177 is also produced through the nuclear reaction  $^{176}\text{Lu}(n, \gamma)^{177}\text{Lu}$ . The diversity of currently produced radioisotopes at research reactors can be seen in Fig. 5.6.



FIG. 5.6. Radioisotope and radiopharmaceutical products generated at Dalat Nuclear Research Institute (courtesy of Dalat Nuclear Research Institute).

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## 6. GEOCHRONOLOGY

The use of research reactors for geochronology is a more specialized application. Reasonable power levels are required, and the user base is a relatively small but widespread group of geologists from many universities and institutions. In addition, there is a tendency towards loyalty to the reactor facilities that are currently utilizing geochronological equipment owing to the lengthy facility calibrations normally required for such equipment.

### 6.1. ARGON GEOCHRONOLOGY

Argon geochronology is a dating method whereby the age of small quantities of minerals, typically measured in milligrams, can be determined by methods based on the radioactive decay of natural potassium. Radiogenic  $^{40}\text{Ar}$  is generated in the sample as a result of  $^{40}\text{K}$  decay. The determination of the amount of  $^{40}\text{Ar}$  present, as well as the original quantity of potassium, enables the age of a sample to be determined. The amount of potassium is determined by placing the sample in a reactor and using the  $^{39}\text{K}(\text{n}, \text{p})^{39}\text{Ar}(\text{n}, \gamma)^{40}\text{Ar}$  interaction. Using automated gas extraction mass spectrometry systems, the ratio of  $^{40}\text{Ar}/^{39}\text{Ar}$  is measured in the material after irradiation. Depending on their nature, samples as young as 2000 years and as old as the Earth itself, around 4.6 billion years, can be dated.

Because of its specialized nature and the expense of the equipment involved, there are only about six laboratories worldwide performing this type of geochronological work. Reactor facilities thus generally perform only sample irradiations before sending them off-site for analysis.

#### 6.1.1. Flux/power level requirements

The  $^{39}\text{K}(\text{n}, \text{p})^{39}\text{Ar}$  reaction has a threshold of around 1.2 MeV, with most of the interactions occurring with neutrons in the 1.2–7 MeV range, necessitating the requirement for fast neutrons for this methodology.

All of the argon isotopes from  $^{36}\text{Ar}$  to  $^{40}\text{Ar}$  are usually measured. Since each isotope has more than one source, efforts are made to reduce contributions from interfering reactions to negligible levels. In particular, there is an interference reaction ( $^{40}\text{K}(\text{n}, \text{p})^{40}\text{Ar}$ ) which is driven entirely by thermal neutrons. This can be corrected by a factor that is constant with age (e.g. it is very small in the case of rocks around five million years old or older). The inaccuracy of not applying the correction factor is therefore often insignificant. In the case of younger rocks, however, not applying the correction can add significant uncertainty to the age — as much as 50% or more. There are two ways to correct for this uncertainty:

- (1) Measure the correction with each sample position in each irradiation, which can be cumbersome;
- (2) Reduce the influence of thermal neutrons by shielding with cadmium.

The latter method is preferred and reduces the correction to the point where it can be applied precisely, even with rocks only a few thousand years old. Although the use of cadmium shielding can be very important for young rocks, it is not necessary for older samples, but can be used anyway, as it does not interfere with the process.

Since it is best to keep the  $^{40}\text{Ar}/^{39}\text{Ar}$  ratio in the range of 1:1 to 1:100, young rocks need much lower neutron fluence than older rocks. The requirement can range from a fast neutron fluence of around  $2 \times 10^{15} \text{ cm}^{-2}$  for young rocks up to  $10^{18} \text{ cm}^{-2}$  for very old rocks. Typically, this could imply an irradiation time from 30 minutes to 300 hours in a 1 MW reactor.

#### 6.1.2. Reactor facility requirements

Based on the foregoing fluence requirements, it is evident that the neutron flux must be high enough to avoid excessively long irradiation times. Typically, this means a power level of around 1 MW for young rocks and 10 MW for very old rocks. In addition, the fast neutron flux has to be reasonably flat over the entire sample. Reproducibility of the fluence is of high importance for these determinations. This means that the sample must be able to be placed in exactly the same position in the core every time.

Samples for these irradiations are typically individually wrapped in foil and stacked on top of one another inside heat sealed quartz vials. Flux monitors are placed at intervals in each stack. Several vials are often irradiated together. The length of the vials is usually a function of the flux variation in the irradiation position. The stack height needs to be limited so that the flux variation over the length is not more than a few per cent. Clearly, the flatter the axial flux in the irradiation position, the more samples can be irradiated at one time. A sufficient irradiation position could typically be an in-core position of 2–3 cm in diameter and 15–20 cm long.

To ensure that the argon does not diffuse out of the mineral matrix and that errors are not introduced into the dating analysis, an important aspect of argon irradiation is that the temperature of the sample should not exceed 200°C.

### **6.1.3. Experimental equipment requirements**

At reactor facilities which do not have geochronological monitoring equipment already installed, it is normally not a logical consideration to install this expensive equipment for relatively few analytical samples. Instead, samples are almost always analysed off-site by the experimenter, so that, except for sample holders and a simple mechanically operated irradiation rig, no experimental equipment is required at the reactor facility itself.

### **6.1.4. Personnel requirements**

Additional reactor staff are not required. However, since irradiated samples are shipped off-site, often internationally, expertise in radioactive material shipment is required. An obvious necessity is that the customer is equipped with the required laboratory equipment, such as mass spectrometers to perform  $^{40}\text{Ar}/^{39}\text{Ar}$  analysis, and also has regulatory clearance to work with the applicable level of radiological samples.

### **6.1.5. Funding requirements**

The initial investment for a facility to perform dating irradiations involves the in-core irradiation facilities. Most reactors already have such irradiation positions. As discussed above, a cadmium lined facility is not required for the analysis of older rocks, but is essential for the analysis of young rocks. If such a facility is not available, it could be typically installed for US \$1000–3000. The only recurring expense is for irradiation capsules and shipping materials.

### **6.1.6. Time requirements**

Since the research reactor operator generally only provides the irradiation service and, in most cases, is able to use an irradiation channel, only the design of a few additional simple tools and the verification of the homogeneity of the irradiation position are required to offer the irradiation service. The required time for these tasks is estimated to be six months or less.

## **6.2. FISSION TRACK GEOCHRONOLOGY**

Fission track geochronology is a method for dating minerals containing uranium, particularly apatites and zircon. Apatite is a calcium phosphate that is common in granite and metamorphic rocks. Zircon is a zirconium silicate that is also common in similar rocks. The sample age is determined by counting fission tracks in the material from spontaneous fission of  $^{238}\text{U}$ . These tracks are a function of the uranium content and the age since ‘closure’, when the fission track clock started. A research reactor is then used to irradiate the samples and induce fission in the  $^{235}\text{U}$  present in the sample. By comparison of the before and after track count, the uranium content in the sample is determined. The process of making fission tracks visible is outlined in Fig. 6.1. Apatites are separated from the mineral, then immersed in Araldite epoxy adhesives. The sample is then cut into thin slices, polished over its surface and finally etched in a proper chemical solution to make the fission track visible to an optical microscope.

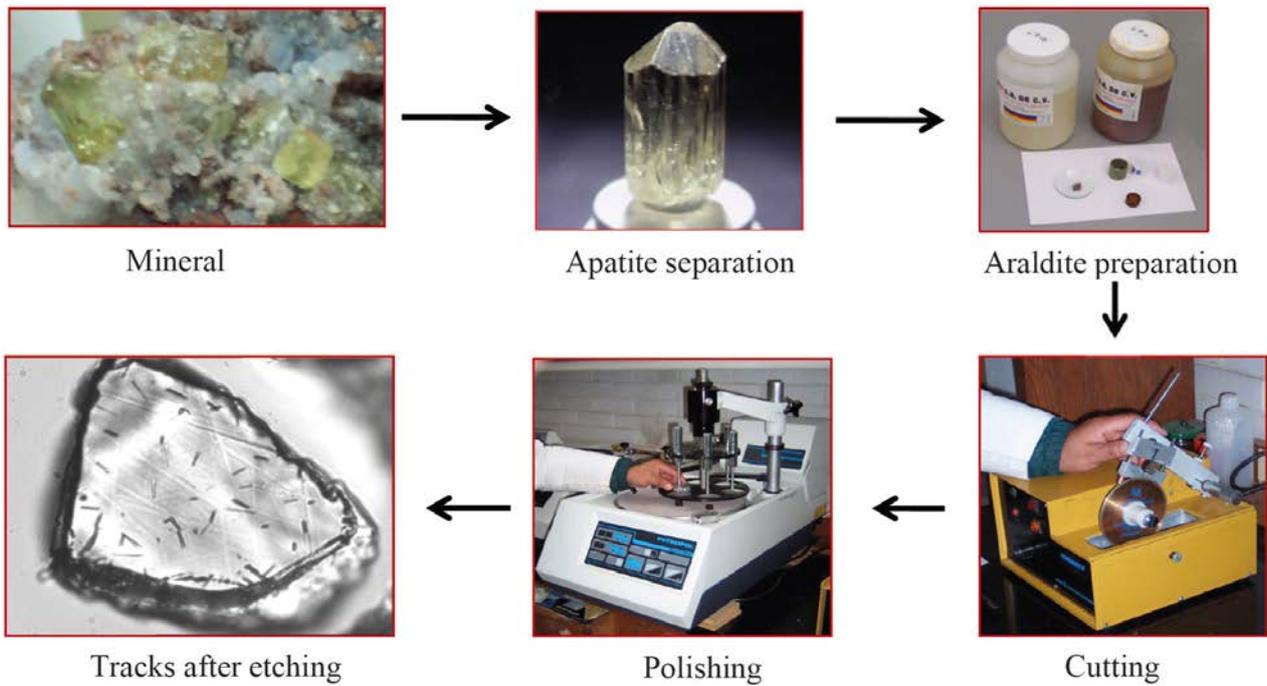


FIG. 6.1. The process of fission track geochronology (adapted from an image from the National Nuclear Research Institute, Mexico).

Since tracks are annealed at temperatures above about 120°C for apatites and 200–300°C for zircons, the method is also useful to determine the thermal history of a sample. Because of the specialized nature of the analysis, most reactor facilities normally only perform the irradiations and then send the samples off-site for analysis.

### 6.2.1. Flux/power level requirements

In addition to the uranium, all the above sample types contain low concentrations of thorium. Both thorium and  $^{238}\text{U}$  undergo fission when irradiated with fast neutrons. Therefore, it is important that the samples are only irradiated with well thermalized neutrons. Hence, it can be assumed that all of the fission events produced during irradiation are from the fission of  $^{235}\text{U}$ . An additional important parameter is that the flux must be stable during the irradiation.

Total fluence requirements are determined by the uranium concentration present in the mineral under examination. Apatites typically have uranium concentrations in the range of 5–100 mg/kg, requiring a thermal neutron fluence of around  $10^{16}\text{ cm}^{-2}$ . Zircons, on the other hand, with uranium concentrations of 50–500 mg/kg, require around  $2\text{--}4 \times 10^{15}\text{ cm}^{-2}$ . As a practical example, this translates to about 30 hours in the thermal column of a 1 MW reactor. However, the irradiations do not have to be continuous. Alternatively, the irradiations can be performed using a heavy water moderated reactor providing a ratio of thermal to fast neutrons as high as 1000 or more. In those reactors exhibiting at least a power of 10 MW, the exposure time can be reduced to only several minutes, which helps to increase the frequency of samples. In addition, a reactor fuelled by a single core offers excellent stability in the neutron flux at the irradiation position over different reactor cycles, since the occasional rearrangement of fuel assemblies in reactors with extended cores is avoided.

### 6.2.2. Reactor facility requirements

Fission track geochronology requires highly thermalized neutrons, such as those produced in a thermal column or a heavy water moderated reactor. The irradiation samples consist of mineral grains mounted, polished and etched, then sandwiched between thin mica detectors. All new fission events within 10  $\mu\text{m}$  of the mineral surface are recorded in the overlying mica, provided the fragments are directed outward. Glass monitors are also packaged with the layers to measure the flux gradient. This results in a typical package about 5–10 cm long and

2 cm in diameter encapsulated in a polyethylene tube. The irradiation channel must be designed such that it can accommodate one or more such packages.

A mounting device is required for ease of handling the radioactive samples at a distance, as well as reproducibly placing the samples in the thermal column.

### **6.2.3. Experimental equipment requirements**

Except for the sample handling and mounting device, no experimental equipment is required, as the samples are usually prepared and analysed off-site by the experimenter.

### **6.2.4. Personnel requirements**

Additional reactor staff are generally not required, but, as in the case of argon geochronology, due to off-site analysis, expertise in radioactive material shipment is often required. To comply with regulations of dangerous transportation and the customer's limit, spectrometric analysis of irradiated samples must be conducted before shipment. Usually, the necessary equipment and expertise is available in a radioprotection department. Once again, a customer with the required laboratory equipment and expertise, together with the applicable regulatory clearance to work with radiological samples, is necessary.

### **6.2.5. Funding requirements**

Some reactors have a suitable irradiation channel providing a sufficiently thermalized neutron spectrum, but it is highly unlikely that a facility will install a thermal column or a special irradiation rig for this application alone, and therefore the cost of doing so is not relevant. Hence, only minimal funds are necessary to construct a sample mounting and handling device.

### **6.2.6. Time requirements**

Similar to the case of argon geochronology, the time required for installation and commissioning an irradiation device dedicated to fission track analysis is six months.

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The following source is recommended as a supplement for those interested in recent literature regarding geochronology techniques used at research reactors.

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## 7. TRANSMUTATION EFFECTS

This category includes those applications in which neutrons or gamma radiation are used to bring about a change in the material properties. The transmutation effects under discussion usually require significant fluences to induce the effect within a reasonable time period. This requires the application of medium and higher powered research reactors. Additionally, to produce sufficient quantities of product to make it worthwhile, fairly large uniform flux irradiation positions are often required.

### 7.1. SILICON TRANSMUTATION DOPING

Neutron transmutation doping (NTD) is defined as the process of creating impurities in an intrinsic or extrinsic semiconductor by neutron irradiation, thereby increasing its value for various applications.

The targets or candidate materials for NTD include gallium arsenide, gallium nitride, gallium phosphide, germanium, indium phosphide, indium selenide, mercury cadmium telluride and silicon, among others. Of these, silicon is currently the only target utilized for commercial NTD applications and is the most widely, internationally used semiconductor material. NTD technology is a preferential method for the production of extremely high quality silicon semiconductors due to its extremely uniform dopant concentration. As a result of the ability to provide excellent product quality, the demand for NTD of silicon has become an internationally significant commercial opportunity.

NTD for silicon is based on the conversion of  $^{30}\text{Si}$  into phosphorus by a neutron absorption reaction, thereby producing the n type silicon semiconductor (see Fig. 7.1). The NTD process takes place when the silicon is irradiated in a thermal neutron flux. The purpose of silicon doping is to create almost free charge carriers such as electrons and holes (i.e. low resistivity) by the introduction of group III or group V elements into the silicon crystal lattice. Only a thermal neutron captured by the  $^{30}\text{Si}$  atom, which has a 3% abundance in pure silicon, leads to the production of a phosphorus donor atom. Due to the high neutron/proton ratio of  $^{31}\text{Si}$ , it releases beta radiation and, by converting a neutron to a proton, the  $^{31}\text{Si}$  atom transmutes to a  $^{31}\text{P}$  atom.

It is known that uniform n type doping with conventional technologies is difficult owing to the volatility of dopants and small distribution coefficients, among others. Since silicon is very transparent to neutrons, a carefully designed irradiation device for uniform and accurate neutron irradiation can provide very high doping uniformity. This extremely uniform dopant distribution is the dominant advantage of NTD compared with conventional doping methods.

The main target of NTD of silicon is doping of the floating zone silicon used for high power devices such as thyristors, insulated gate bipolar transistors (IGBTs), integrated gate commutated thyristors and gate turn off thyristors. These devices require a high breakdown voltage and low on-state resistance. The NTD ability of silicon to provide excellent levels of uniform doping readily meets these system necessities. The NTD method has greatly

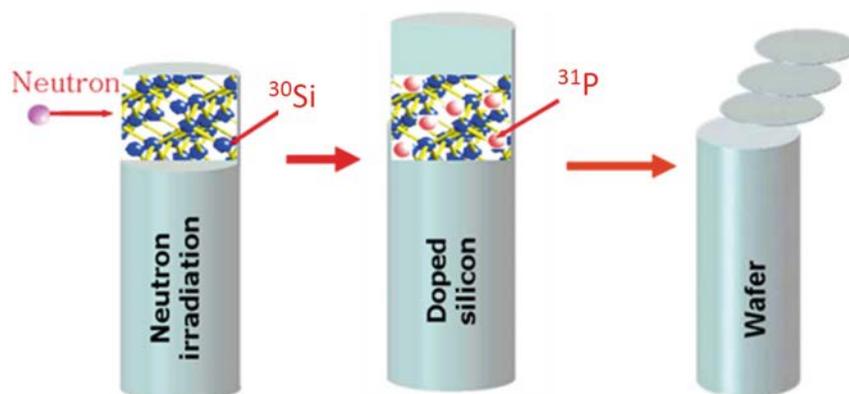


FIG. 7.1. Outline of neutron transmutation doping of silicon ingots (adapted from an image from the Jordan Atomic Energy Commission).

contributed to a substantial gain in the voltage rating of thyristors since initial application in the late 1970s. The semiconductor materials produced by the NTD method are already popular and widely used in various industrial fields, especially in the production of high power semiconductor devices or production of sensors and standard devices. Among the various fields of research reactor utilization, NTD of silicon is regarded as one of the most useful applications in the aspect of the economy of the research reactor, and is a field that can provide a direct commercial income.

Since the real commercial breakthrough of the NTD method in 1973 for the production of thyristors, the worldwide demand for NTD increased rapidly up to the early 1990s. During the 1990s, the demand decreased due to the development of other competitive doping technologies. Up to the mid-2000s, the demand fluctuated at capacities greater than a hundred tonnes per year (i.e. roughly 20% of the floating zone silicon production). Due to the rapid rise of green energy technologies since then, the demand for high power devices has increased even more rapidly. Production of hybrid cars using electric motors is now strongly encouraged to reduce the amount of discharged air pollution substances, such as carbon dioxide. These cars require inverters with power devices like IGBTs for the electric traction motors. It is known that NTD floating zone silicon wafers are remarkably suitable as a base material used in the IGBTs. The rapid increase in demand for small scale electricity generation such as solar cells and wind power will certainly also require more power semiconductor applications

NTD is currently dominated by the utilization of  $\varnothing = 150$  mm, often still called 6 inch, silicon ingots. The  $\varnothing = 125$  mm (5 inch) ingots still have a considerable but decreasing market share, whereas the demand for NTD of  $\varnothing = 100$  mm (4 inch) or less ingots has nearly disappeared. The demand for an irradiation of  $\varnothing = 200$  mm (8 inch) has slowly grown within the last few years. Its share of the market will probably also be extended in the future, since the ability of various reactors to produce equivalent uniformity of doping throughout the increased diameter due to resultant neutron attenuation has been demonstrated.

### 7.1.1. Flux/power level requirements

In facilities where it is possible to control the irradiation time and the neutron flux exposure to the ingot very accurately, the required resistivity target can be specified and accomplished within a very tight tolerance. The deviation distribution for the final resistivity of the irradiated silicon ingot from the target resistivity typically has a Gaussian distribution with a standard deviation within 3%, particularly in cases in which the research reactor has considerable applicable NTD experience.

As NTD uses mainly thermal neutrons, a higher thermal neutron flux allows for shorter irradiation times. However, if the irradiation time is too short, keeping an accurate irradiation time is difficult. Therefore, a very high flux limits its application to a relatively low resistivity only. On the other hand, if the flux is too low, a very long irradiation time is needed, which limits the practical application for NTD. The resistivity of NTD silicon can vary from  $30 \Omega \cdot \text{cm}$  to more than  $1000 \Omega \cdot \text{cm}$ . It is worth noting that very high target resistivities of several hundreds of  $\Omega \cdot \text{cm}$  should preferably be achieved using a heavy water moderated reactor because it offers a spectrum with a very high ratio of thermal to fast neutrons. This ratio is desirable because fast neutrons generate electrically charged large defect clusters which are difficult to be annealed thermally, whereas thermal neutrons only generate thermally unusable point defects.

In NTD methods, the generated dopant ( $^{31}\text{P}$ ) concentration is proportional to the irradiation's neutron fluence, which, in turn, is proportional to the thermal to neutron flux and the irradiation time. This implies that a higher thermal neutron flux will require a shorter irradiation time to achieve an equivalent fluence. The final resistivity of the irradiated silicon is inversely proportional to the total concentration of the produced dopants and initially existing ones. For example, a neutron flux of  $10^{13} \text{ cm}^{-2} \cdot \text{s}^{-1}$  requires around 17 hours of irradiation time to achieve a  $50 \Omega \cdot \text{cm}$  modification to the ingot. An irradiation time of at least one hour is normally appropriate for an accurate irradiation fluence considering the time required to insert and withdraw an ingot. Therefore, about  $800 \Omega \cdot \text{cm}$  is the upper limit of resistivity change when the flux is  $10^{13} \text{ cm}^{-2} \cdot \text{s}^{-1}$ . When the flux is five times higher, the upper limit is  $160 \Omega \cdot \text{cm}$ . Irradiation requirements for a very high target resistivity of around several hundred  $\Omega \cdot \text{cm}$ , even up to  $1000 \Omega \cdot \text{cm}$ , have recently been more often requested by clients. The irradiation of these ingots would be appropriate for a low flux reactor with a thermal neutron flux of several  $10^{12} \text{ cm}^{-2} \cdot \text{s}^{-1}$ , since the irradiation time is more appropriate to maintain the irradiation quality.

In addition to the axial and the radial uniformity of the doping profile mentioned earlier, the accuracy of the resistivity after NTD regarding target resistivity is the major parameter with respect to the commercial exploitation

of a silicon irradiation service. Initially, most commercial contracts included an accuracy penalty associated with a target resistivity of typically  $\pm 7\%$ , but the standards set by the semiconductor industry have become more restrictive recently, and an accuracy of around  $\pm 4\%$  will likely be required in the near future.

#### 7.1.1.1. Constraints on flux

The advantage of NTD compared to other doping methods is the uniformity of resistivity throughout the silicon ingot. Since the silicon ingot for NTD is a solid cylinder, its irradiation uniformity is expressed by radial and axial (or longitudinal) variation of the final resistivity after irradiation. Thus, the uniform irradiation, not only radially but also axially, is the prime target of an acceptable irradiation device's design and operation.

The planar distribution of the resistivity of an NTD silicon wafer is represented by the radial resistivity gradient (RRG). The neutron flux at the inner part of an ingot is slightly lower than at the periphery due to neutron attenuation by silicon. This non-uniformity becomes more severe for an ingot with a larger diameter. The RRG of NTD with no initial resistivity distribution is expected to be less than 1.5% and 2% for 5 inch (12.7 cm) and 6 inch (15.24 cm) silicon single wafers, respectively. The requirement for RRG is usually less than approximately 4–5% for 5 inch ingots.

The longitudinal variation of the resistivity through the whole length of an irradiation batch composed of several cylindrical ingots is represented by the axial resistivity variation. This variation is caused by the non-uniformity of the longitudinal neutron flux inside the reactor. To achieve longitudinal uniformity, three basic irradiation concepts are used:

- (1) Reciprocating motion: An ingot moves inside a cavity from one side to the other side.
- (2) Inversion: An ingot is irradiated at the linear neutron flux distribution for half of the total irradiation time, then the ingot is inverted and irradiated for the remainder of the time.
- (3) Flux screen: An ingot is irradiated with a proper flux screen providing a flat neutron flux.

Even though the requirement for resistivity uniformity depends on the customers and contracts, the requirement for the axial resistivity variation is usually around 5–8%. It is recommended that the axial variation of the neutron flux of an irradiation facility should be within around 3%.

Since fast neutrons cause defect clusters which cannot be annealed completely, the fast neutron flux must be as low as possible. Gamma rays are the major source of heat generation in the ingot. The gamma ray flux should thus be as low as possible, and the ingot must be sufficiently cooled during the irradiation.

The handling and transportation of NTD silicon irradiated in a reactor can usually be exempted from radioactive material requirements based on international criteria. The most likely sources of residual radioactivity are, for example,  $^{32}\text{P}$  (half-life of 14.3 d), as well as radiological products generated by neutron interaction with any initial resident impurities in an ingot. Since the irradiated ingots will be processed after quite a long cooling time, only longer lived radioisotopes must be identified during post-irradiation analysis. Therefore, the residual radioactivity is most likely to be proportional to the neutron fluence (i.e. inversely proportional to the resistivity).

#### 7.1.2. Reactor facility requirements

The diameter of the irradiation cavity needs to be large enough to accommodate the ingot, the coolant path, the canister containing the ingot and the flux monitor. Since the radial resistivity non-uniformity due to a neutron flux gradient in the radial direction of a reactor is always compensated for by rotating the ingot during irradiation, equipment or mechanisms to maintain an equal rotation of the ingot are required.

In addition, the neutron absorption and scattering characteristics of silicon and water significantly differ from each other. In many cases, it is necessary to compensate for this interference by the use of silicon dummies or aluminium spacers closely covering the silicon ingot to be doped. Finally, the insertion of the silicon ingot into a water filled core position can influence the neutron flux in a research reactor and subsequently other experimental facilities. This interference is to be examined carefully.

### 7.1.3. Experimental equipment requirements

Since a silicon crystal is very brittle, even glass like, careful design and operation of transfer and manipulation tools are required. Induced radioactivity of the irradiated silicon ingots must decrease below the radiation level of exemption prior to handling and transportation. Appropriate storage, cleaning and packaging space is thus needed to allow the irradiated silicon to cool down and be cleaned and examined using suitable monitoring systems.

A commercial NTD irradiation service requires systematic QA/QC to be implemented. For example, a typical QA/QC programme might require a neutron fluence monitor such as an activation foil to be irradiated at the same time as the ingot irradiation. This would require the acquisition of equipment for measuring the activity of such a fluence monitor.

### 7.1.4. Personnel requirements

Personnel requirements for a commercial NTD process at a research reactor depend largely on the scale of the NTD production and the level of ability of personnel available. Generally, NTD related activities can be grouped into, or involve:

- Administration;
- Irradiation physics;
- Facility design;
- Ingot handling before, during and after irradiation;
- Quality control;
- Maintenance;
- Radiation monitoring.

In several cases, due to the longer irradiation periods and the requirement to remove the ingots from the flux positions at specific times, the reactor must be operated continuously by personnel able to perform such operations on a shift basis. Depending on the relative importance of NTD for the particular reactor, the minimum of a remote termination capability of the irradiation process is necessary.

### 7.1.5. Funding requirements

The funds required greatly depend on the local situation, such as the capability to fabricate the irradiation facility and additional equipment and operation and maintenance costs. If depreciation costs are ignored, then the cost for the facility could be roughly estimated as follows:

- (a) Irradiation rig: US \$100 000–300 000 per rig, including design cost.
- (b) Equipment for ingot loading and handling: US \$50 000–100 000.
- (c) QA/QC programme:
  - (i) SPND and module: US \$30 000;
  - (ii) Equipment for fluence monitoring and radioactivity surveying: US \$40 000.
- (d) Total: US \$220 000–470 000.

The establishment of a new NTD facility requires additional costs for professional personnel and fuel due to extended hours of operation. Once the NTD facility is well established, additional expenses other than those of maintenance, additional personnel and neutron (reactor) costs are unlikely.

### 7.1.6. Time requirements

The installation and commissioning of a new silicon doping facility from the very beginning includes:

- Choice of an appropriate irradiation position;
- Neutronic and thermohydraulic calculations;

- Design and manufacturing of the irradiation rig, including special handling tools;
- Training staff;
- Calibration measurements to guarantee the required flux parameters.

The time needed is estimated to be a minimum of three years. The installation and commissioning of a (semi) automatic facility may require five years.

#### **7.1.7. Customer relations**

The industrial application, and thus demand, of NTD materials is growing internationally and is greatly based on the competitive efficiency of the research reactor facility. This includes applications of a reliable QA/QC service ensuring the following aspects are maintained: the production of dopant axial and longitudinal uniformity; turnaround time, the period from delivery to return to the customer; reliability of supply; and of course, the cost of the service.

Due to the precision required when achieving the customer's resistivity target — often better than 4% — and thus correctness of the fluence application, it is often essential for the facility to interact with the customer prior to the commercial contract. Achievement of irradiation targets as reported by the customer following postannealing treatment can provide important information to the facility, enabling a form of product calibration such as resistivity change versus fluence for the targets supplied. In several cases, the ability of a facility to establish annealing facilities and subsequent end resistivity measuring ability is expensive and often limited without the cooperation of selected customers to share the related technology. Since NTD is only one step in a long industrial supply chain, a minimum established QA certification, such as International Organization for Standardization ISO 9001:2008, must be acquired by the reactor to become an eligible supplier for potential customers.

## **7.2. GAMMA IRRADIATION**

The discussions in the preceding sections have indicated that a significant neutron flux is generally required to enable the useful irradiation of most materials. On the other hand, almost any reactor can make use of its activated fuel (e.g. during shutdown cycles and for gamma irradiation).

### **7.2.1. Reactor facility requirements**

A gamma irradiation facility can readily be developed with a relatively small investment at a research reactor (e.g. for irradiating plants and seeds). If fuel assemblies are utilized, the dose rate achievable is dependent on reactor power and cooling time. Fuel element storage racks can be used to hold the assemblies during the gamma irradiation, and very little extra is needed with respect to facilities, equipment, personnel and funding.

Dose rates can vary from around 2 Gy/h for fuel elements from a low power reactor up to about 200 kGy/h for fuel elements from a high power reactor (i.e. those with fluxes greater than  $10^{14} \text{ cm}^{-2} \cdot \text{s}^{-1}$ ).

Operation of such gamma facilities is generally straightforward, even with the reactor shut down. The operating staff can usually perform such gamma ray irradiations.

### **7.2.2. Experimental equipment requirements**

The required additional experimental equipment includes:

- Gamma dose rate and total dose monitoring equipment;
- Temperature measurement and temperature control equipment, in most cases;
- Special conditioning equipment for items to be irradiated and tested;
- Handling equipment.

### 7.2.3. Personnel requirements

Additional staff are generally not necessary for the operation of such a facility.

### 7.2.4. Funding requirements

For example, if the control of parameters such as temperature or pressure is not required, a gamma irradiation facility can be built at a cost of approximately US \$10 000.

### 7.2.5. Time requirements

The time for construction, manufacturing and commissioning is not to exceed six months. Once again, however, these estimations are greatly dependent on the individual reactor.

## 7.3. GEMSTONE COLORATION

Gemstones may be irradiated either with neutrons or gamma quanta of high energy of the order of several tens of MeV produced by electron beams to improve their properties (e.g. changing to a more desirable colour to increase their appeal and monetary value). One of the more significant commercial neutron irradiation activities currently being performed at research reactors is the coloration of topaz. The following information is pertinent to topaz irradiation. However, before proceeding to gemstone coloration, the legality of this activity must be verified with national authorities, as several States have banned the practice.

Similar to NTD of silicon, there is potential for the generation of a significant income through gemstone irradiation. Unfortunately, due to commercial sensitivity, there is an understandable reluctance among those performing the irradiation to disclose the specific details of their processes — in particular, for both NTD and gemstone applications. In addition, there are some difficulties associated with gemstone coloration activities that need to be heeded.

Commerce in gemstones and the field of reactor management, operation and utilization are culturally very different. The market for irradiated gemstones is difficult to assess, as it is dependent on the size and colour of stones currently in fashion. This often makes the behaviour of the gem trader also appear to be somewhat whimsical. In addition, the operating schedule for the research reactor and the time required for the irradiation and processing the stones is often incompatible with the needs of the trader.

The decay times involved in the radiological cooling of irradiated gemstones is often quite long, resulting in a trader's investment risk in the virgin material, particularly when compared with the gem trade's cultural time frame. The reactor facility has an investment risk in both the irradiation devices and the development of the measuring equipment. The facility is also dependent on the delivery of good quality virgin material, so that it is not left with a large quantity of material that is useless because of long lived activation products.

Nevertheless, although several research reactors worldwide irradiate topaz commercially and perform research on irradiation of gemstones, only three facilities have applied for the licence required in the United States of America.

#### 7.3.1. Flux/power level requirements

The colour of topaz is induced by the interaction of fast neutrons. If thermal neutrons are allowed to irradiate the topaz, then significant undesirable radioactivity is induced in the gemstone.

The required fluence is dependent on the specific batch of topaz stones and the depth of the desired blue coloration. For example, the fast neutron fluence required is typically of the order of  $10^{17}$ – $10^{18}$  cm<sup>-2</sup>. This equates to about 50–100 hours of irradiation required to achieve this fluence in a 2 MW research reactor.

### **7.3.2. Reactor facility requirements**

The stones are typically batch irradiated in containers, and a batch usually consists of around 2 kg of stones. Since only fast neutron irradiations are desired, the containers or the irradiation facility are often shielded with boron or cadmium. As the temperature during irradiation must be controlled, some method of cooling the stones is also necessary.

If the temperature of the gemstones approaches 300°C, the damage to the topaz will anneal and discolouration will occur. In addition, if the temperature is too high, the stones are prone to flaking during post-irradiation handling. A typical temperature during irradiation is 100–150°C. The conditions of irradiation and the characteristics of a topaz batch could typically produce a heat load of around 0.5 W/g in topaz. The batch of stones must thus be cooled during irradiation, but if this is done using water, undesirable thermalization of the neutrons can take place.

A dedicated irradiation container and facility is required. The container or the facility into which it is placed might be cadmium covered, in which cooling is provided by a small amount of water. Ideally, it might consist of a boron shielded container or facility in which the stones are cooled by the flow of nitrogen gas. This latter device is more expensive to operate.

The irradiation could take place in an aluminium tube placed in a grid position and fixed at the upper reactor bridge structure. It could also take place in a beam tube, which obviously makes it easier to provide pure fast neutron irradiation since the thermal neutrons can be absorbed by shielding materials, apart from the irradiation position.

### **7.3.3. Experimental equipment requirements**

At the end of the irradiation period, a storage facility must be available for the stones until their radioactivity has decayed below the specified release limit. The storage time period is greatly dependent on the original characteristics of the stones. In some cases, it has been experienced that it was possible to release 70% of the stones after two months, and only after eight months had a further 10% of the stones been released.

Because of market forces, it is desirable to release the stones as soon as possible. This requires frequent determination of the radioactivity of the stones and predictions of a release date. Automatic handling and activity measuring robots can be used to perform these periodic measurements of the stones.

In some States, the radioactivity limits for releasing the stones have become nuclide specific (e.g. the upper limit is 70 Bq in the United States of America). This requires the use of an MCA as well as a system for determining the beta emissions from the stones prior to their release for transport.

### **7.3.4. Personnel requirements**

Topaz irradiation is a time consuming process with high worker involvement. The initial preparations will require the services of a radiochemist and an individual trained in heat transfer calculations and techniques. Even when automated equipment is used for the post-irradiation analysis, several technicians are required for both the operational and maintenance activities.

### **7.3.5. Funding requirements**

The cost of an automated system with commercial viability is high. Cost recovery from customers may be possible over time, but is very much dependent on the batch frequency and turnaround time to delivery.

A facility manager wanting to enter this field can start with a small production test quantity, using a simple irradiation rig and existing counting systems to become familiar with the techniques. Modification to the reactor facilities and subsequent expansion should not take place without a commitment from a customer.

### **7.3.6. Time requirements**

Design, construction, manufacturing and commissioning of an irradiation rig dedicated to the coloration of gemstones includes the identification of a suitable irradiation position and proof of the desired change of colour,

as well as proof of the acceptability of the neutron spectrum with respect to the necessary low activation of the gemstones. A lead of at least 2–3 years is estimated.

#### 7.4. ACTINIDE TRANSMUTATION

It has been recognized for many years that it is theoretically possible to transmute some long lived actinides in spent nuclear fuel into shorter lived products, thereby reducing the potential waste disposal hazard. To this end, some actinide ‘burners’ have been designed, but none has yet been built for this specific purpose. It is possible that some reactors may be utilized in the future for test irradiation of fuel plates or elements.

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## 8. NEUTRON IMAGING

The sensitivity of light elements to the absorption of neutrons compared with X ray imaging provides neutron imaging with an advantage in both 2-D and 3-D visualizations. Until the 1990s, the dominant detection systems for neutron imaging were film based (e.g. X ray film and converters with track etch foils) and known simply as neutron radiography. Since then, detection methods have been improved drastically, digital processing systems have been introduced, and imaging beam lines have been built with the capability to scan in energy through the range in which structural materials show Bragg diffraction edges.

### 8.1. ADVANCED METHODS IN NEUTRON IMAGING

As a result of these innovations, and owing to different attenuation contrasts compared with the more common X ray methods, new and challenging application fields have been established, in particular:

- Determination of hydrogen in electrochemistry analyses of fuel cells;
- Non-invasive study of cultural heritage objects and biological samples;
- Applications in geology and soil physics;
- Applications with nuclear fuel and its cladding;
- Investigation of several aspects of materials research.

New links to industry have been established where neutron imaging complements other techniques or, in some cases, where neutron imaging is the only possible approach for a suitable solution of the problem. A single neutron image is a 2-D shadow image of the object under investigation obtained from the transmitted neutron beam. All object layers in the beam direction are superimposed and cannot be readily distinguished. Neutron tomography overcomes this limitation, and provides a full 3-D sample structure as ‘seen’ by the neutrons. For this purpose, the sample is rotated around one axis, and many single projections from different viewing angles are produced. The volume information is obtained with the help of mathematical algorithms in the form of a reconstruction step after the real data acquisition. Figure 8.1 shows an example of neutron imaging where its advantage — the high transmission of metals and high contrast for hydrogenous materials — is used in the best possible manner. It has become possible to introduce neutron tomography for 3-D imaging as a standard tool at several neutron beam facilities.

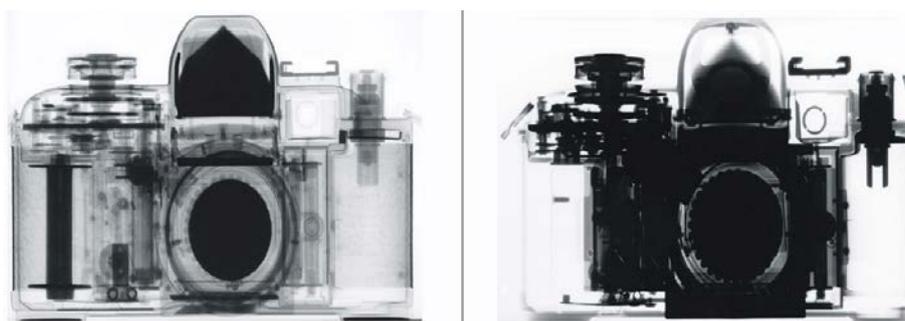


FIG. 8.1. Comparative images of a camera as depicted by neutron imaging (left) and X ray radiography (right) (courtesy of the Paul Scherrer Institute).

Time resolution is another advance in neutron imaging at high flux facilities, whereby frames of an acceptable quality can be obtained within a few milliseconds. The stroboscopic approach, in which the image frame acquisition is synchronized to a repetitive process, involves the combination of many underexposed images to produce a valid image in high quality for a specific short snapshot. One important application of this technique is the observation of processes in combustion engines, pumps and other technical devices running at high rotation speeds. For example,

it is possible to visualize lubricants and fuel under various operational conditions in engines. In such an image, most of the aluminium parts are transparent, while the sealing materials and the lubricant are clearly visible. Furthermore, these devices can be inspected under real working conditions at approximately 1000 rev/min in the stroboscopic mode. A similar effect is shown in the left image of Fig. 8.1, alongside a comparative image of the camera formed by X ray radiography, in which light elements are mostly transparent, but heavier elements are projected as an opaque mass.

Through digital neutron imaging, quantification of the sample content is possible if the transmission process is understood in terms of cross-section data, the neutron spectrum and detection efficiency. Parallel to the direct measurements, Monte Carlo simulations can be performed to understand the results. Using the refraction properties of neutrons at material boundaries, edge enhancement of structures becomes possible through a process known as phase contrast imaging. This is particularly important when the absorption contrast of the involved materials is weak. Most neutron imaging is performed with neutrons from a wide spectral range of thermal or cold neutrons. The resulting images are averaged over this spectrum, and features such as Bragg diffraction edges cannot be resolved. Recently, through energy selective imaging, which scans through narrow spectral ranges, crystal structures from the consolidation process of welds have been visualized directly, improving quantification in radiography and tomography and stress analysis in structural materials.

Imaging of magnetic domains is achieved by the addition of a neutron beam polarizer before the object under investigation and a polarization analyser behind it. The change in polarization through interaction with magnetic dipoles in the magnetic domains can be observed directly.

## 8.2. REQUIREMENTS FOR PERFORMING NEUTRON IMAGING

### 8.2.1. Flux/power level requirements

The range of potential applications for imaging with neutrons is largely limited by source reactor flux because advanced neutron transport systems are generally not advantageous, and therefore rarely used. Choices of neutron moderator and image resolution have a strong impact on beam flux. Annex III summarizes many neutron imaging installations currently operating or under construction around the world.

### 8.2.2. Neutron source

Most of the strong neutron sources suitable for neutron imaging are reactor based. Although almost every research reactor possesses a beam port capable of supporting a neutron imaging facility, only a limited number of these are currently equipped with functional neutron imaging facilities. Most of these deliver thermal neutrons from the water moderator surrounding the reactor core. The beam is often accompanied by a field of gamma radiation also emitted from the core, which initiates an undesirable background signal on the detector.<sup>1</sup> There are also beam lines available with cold neutrons from a cold source and for fast neutrons from a converter system. Adapted detection systems are required to use fast neutrons efficiently.

### 8.2.3. Experimental equipment requirements

A neutron imaging device generally consists of a neutron source, a collimator, a sample, a detection system and its specific sample environment (see Fig. 8.2). The whole installation must be encased in thick shielding capable of reducing fast, epithermal and thermal neutron background radiation, as well as source and secondary gamma radiation. All of these components contribute in a specific manner to the final result of the investigation. A perfect imaging result is only possible under optimization of their individual options.

The quality of the image is determined by the beam conditions that are, in turn, modified through collimation. To produce optimal beam conditions, the collimator consists of the following components, not all of which are necessarily applied simultaneously:

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<sup>1</sup> Spallation sources and other accelerator driven neutron sources are also able to provide a neutron imaging capability if the beam properties and the detector performance are matched together in the right manner.

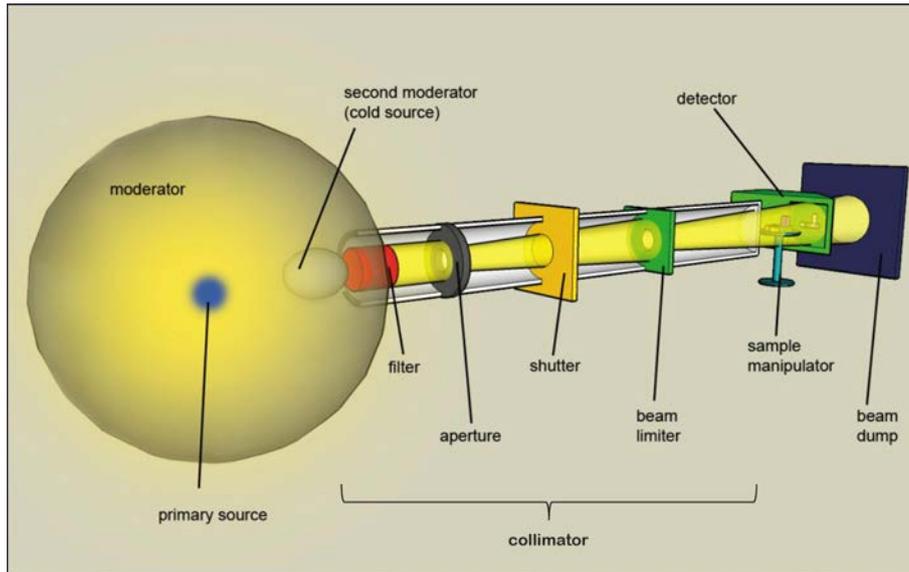


FIG. 8.2. A simplified layout of a neutron imaging system (adapted from an image from the Paul Scherrer Institute).

- Inlet apertures;
- Filters for fast and epithermal neutrons and gamma radiation and polarizers;
- Energy selectors such as velocity selectors and double crystal monochromators;
- Beam limiters;
- Flight tubes, evacuated or helium filled;
- Shutters.

The beam intensity is strongly linked to collimation. Treating the primary neutron source as a point source, the neutron intensity  $\phi$  is related to the aperture diameter  $D$  and the source to detector distance  $L$  by:

$$\phi \propto 1/(L/D)^2 \quad (8.1)$$

The ideal case is a parallel beam for a high  $L/D$  ratio, with the field of view adapted to the object size and having a well defined neutron spectrum but no gamma contamination.

As beam intensity is critical to most of the advanced neutron imaging techniques, a compromise must be found between the collimation and flux, which determine the resolution and exposure time, respectively.

The experimental set-up in modern imaging investigation is also highly important for successful imaging. Very often, complex processes are studied inside the neutron imaging facility while a permanent observation takes place. The structure around the sample and the running process needs to be as transparent as possible for the neutron beam. If the object under investigation is larger than the beam diameter, an inspection becomes possible with a remote controlled movement across the beam using a sample manipulator. The obtained partial images of the extended object can be combined or unified off-line with postprocessing tools.

### 8.2.3.1. Detector system

Current neutron imaging systems deliver digital image data in common data formats. Therefore, any advanced software for postprocessing can be used to correct image artefacts and to derive quantitative information from the raw database. Figure 8.3 gives a qualitative overview of the application areas of different modern neutron imaging systems with respect to their properties for spatial and temporal resolution, given by their sensitivity, dynamic range and readout behaviour.

Camera based neutron imaging systems, in which a neutron sensitive scintillation screen is the primary detector, are generally considered to be superior to other systems owing to their higher flexibility and their fixed

position in the beam. Figure 8.4 shows one such detection system, where a lighttight box covers the scintillator, the mirror and the optical system. It boasts a variable field of view and spatial resolution, which are managed by changing the camera distance or the lens, respectively, and can be used for tomography and other investigations with a high demand for quantification and referencing. Systems with a spatial resolution of 10  $\mu\text{m}$  exist [8.1], but improvements continue.

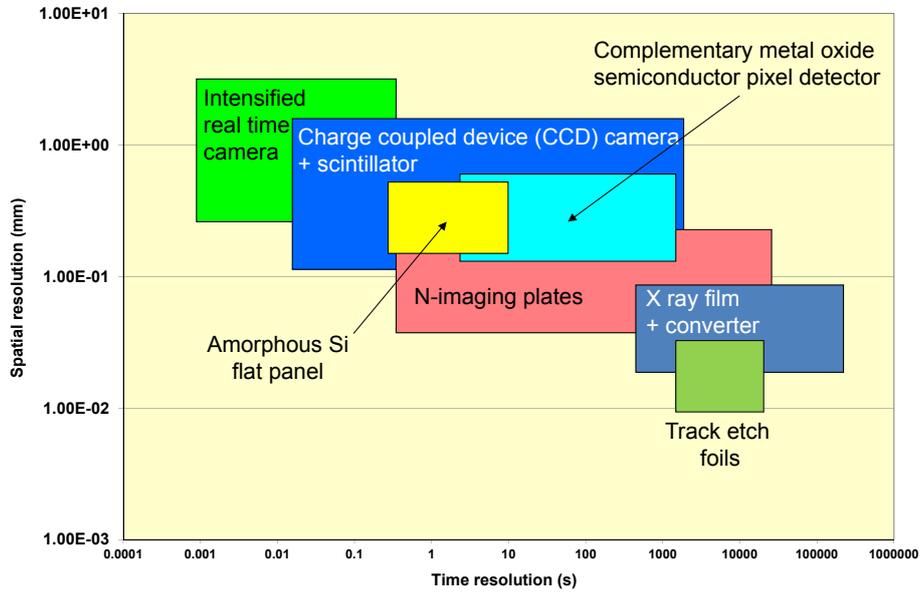


FIG. 8.3. Application areas of different imaging systems (adapted from an image courtesy of the Paul Scherrer Institute).

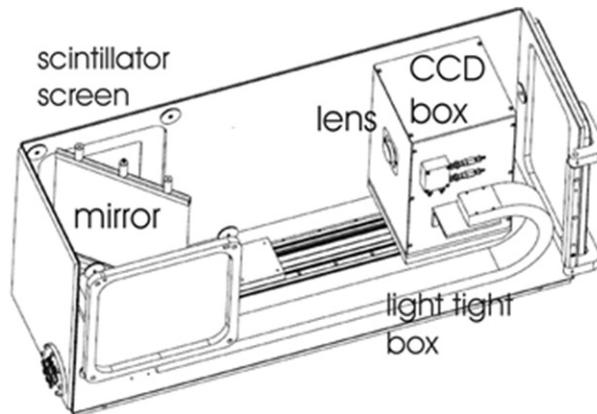


FIG. 8.4. Layout of a charge coupled device camera based neutron imaging detector (courtesy of the Paul Scherrer Institute).

#### 8.2.4. Personnel requirements

The design, fabrication and installation of such facilities require a professional nuclear engineer working with appropriate workshop mechanics and technicians. A procedure must be developed by a professional staff member for each type of neutron imaging application performed. After the procedure has been tested, a technician, or higher qualified person initially, needs to be available to perform the experimental and imaging applications and to process the data according to specific user requirements. The most efficient utilization of neutron imaging processes is acquired if the operating organization provides a complete service.

TABLE 8.1. COST ESTIMATES OF MAJOR COMPONENTS OF NEUTRON IMAGING INSTALLATIONS

Component	Cost estimate (US \$)	Comment
Primary shielding	~500 000	A complete interlocked shielding cave is required for safety and quality.
Primary collimators	10 000–50 000	A system with multiple source apertures provides a range of $L/D^a$ ratios at increased cost.
Energy selective filters	25 000–250 000	Simple double crystal monochromators cost ~US \$25 000. High performance velocity selectors are ~US \$250 000.
Flight tubes, shutters and apertures	10 000–50 000	
Sample translation and rotation stages	25 000–250 000	Simple sample position devices cost ~US \$5 000. Applications such as tomography require precision translation and rotation at a cost of ~US \$250 000.
Digital detection system	5 000–100 000	A simple digital system is ~US \$5 000–7 000. <sup>b</sup> More advanced imaging systems are generally <US \$100 000.

<sup>a</sup>  $L$  — source to detector distance;  $D$  — aperture diameter.

<sup>b</sup> It should be noted that Institut Laue-Langevin and Technische Universität München have built relatively inexpensive devices that can readily be copied or ordered.

### 8.2.5. Funding requirements

Information concerning cost estimates for some selected techniques is provided in Table 8.1. By far the most expensive component in a modern imaging facility is the shielding, but other components, such as digital detection systems, velocity selectors and precision sample translation and rotation stages, can also be expensive. A basic facility may cost as little as US \$150 000, but for an advanced neutron imaging facility at a high flux reactor, an initial investment in hardware of around US \$1 million is required.

### 8.2.6. Time requirements

Equipment procurement, modelling and design, and commissioning of a neutron imaging facility can be expected to require two to four years.

## 8.3. FUTURE OF NEUTRON IMAGING

The development of new detection systems, the installation of new and improved imaging systems and the demand for new applications provide a promising foundation for the future of this methodology. However, its development significantly depends on the availability of neutron sources of sufficient strength and access to suitable beam ports at these installations. Neutron imaging is accepted as a powerful tool in non-destructive testing with strong potential for growth as an effective research tool in the future. If it is well designed, the imaging facility can be used flexibly and efficiently for various applications (e.g. for scientific and industrial customers).

Apart from further improvements to the newer applications discussed in Section 8.1, the demand for studies with the highest possible spatial resolution is growing. The present limitation of the neutron detection process is around 10  $\mu\text{m}$ , and it will be difficult to overcome this limit using current methods. There are, however, sufficient compelling applications demanding imaging in this resolution range to drive further innovation in improved resolution neutron imaging. New neutron optical devices such as reflective focusing devices or refractory lenses and detection systems are under development with the potential to approach a resolution of 1  $\mu\text{m}$ .

### REFERENCE TO SECTION 8

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## 9. MATERIAL STRUCTURE AND DYNAMICS STUDIES

### 9.1. USE OF NEUTRON BEAMS FOR SCIENCE

The development and engineering of new complex materials with improved properties and functionalities are key requirements for providing solutions to major problems affecting society in the twenty-first century. These problems are posed by new sociological and technological demands in areas such as health, energy, national security and the environment. The capacity to accomplish the appropriate solutions is crucially dependent on the understanding of materials, which, in turn, requires the mastering of different experimental and computational techniques to unravel and optimize their properties.

In order to explain the physical properties of a material or of a class of compounds, it is essential to understand the interactions between its elementary components. On an atomic scale, these interactions determine:

- The ordering relations between atoms or molecules and between electronic magnetic moments.
- The dynamic characteristics of each atom or molecule, known as individual dynamics, and the phase correlations between the motions of neighbouring atoms, or collective dynamics. The same analogy exists for magnetic moments.

Over the past century, numerous techniques have been developed for investigations on an atomic scale that enable scientists to describe these fundamental interactions with increasing precision.

Neutron beam techniques have made key contributions to our scientific knowledge since the 1940s, when research began on material structure at the Oak Ridge Research Reactor by Clifford Shull and co-workers, and soon after at the Chalk River Nuclear Laboratories' NRX research reactor by Bertram Brockhouse and co-workers in atomic and molecular dynamics. Shull and Brockhouse were subsequently honoured in 1994 for their pioneering achievements in neutron beam methods with the Nobel Prize in Physics. This was in recognition not only of the significance of their discoveries and of the discoveries made possible by their work, but also of the enormous impact that neutron scattering has had on our understanding of atomic and molecular structure and dynamics. This is illustrated in Fig. 9.1, which notes the number and distribution of neutron beam users working in European States fifty years after the establishment of the basic methods [9.1].

Neutron beam techniques are now well established, and have become one of the first techniques of choice for detailed characterization of atomic and magnetic structures and dynamics for many important new classes of materials and their processing. These have significantly improved the understanding in areas such as condensed matter physics and chemistry, nanotechnology, polymer science, life science, sustainable energy research, sensors and smart materials, biotechnology, spintronics, engineering and archaeology.

Neutrons are extracted through beam tubes from the moderator and reflector region surrounding the reactor core. The emerging neutrons have energies in different ranges depending on whether they are emitted directly from the reflector at near room temperature or from re-thermalizing media within it, which can result in neutrons of lower temperatures (e.g. 10–30 K) or higher (e.g. 1000–3000 K). In this way, researchers may use thermal, cold or hot neutron sources to optimize the experimental conditions according to the structural and dynamic characteristics of the material under study.

An incident neutron beam falling onto a given sample may interact with it in different ways, with probabilities determined by the corresponding cross-sections. As a result of an interaction, a neutron may be absorbed, scattered or transmitted by the sample material. Specialized instruments record the outgoing neutron distribution either using a well defined incident energy or wavelength, or a range of energies in the experiments. Neutron scattering instruments are generally referred to as spectrometers when some combination of the scattered neutron angular momentum, energy and polarization is analysed, or as diffractometers when only the scattered neutron angular momentum is recorded.

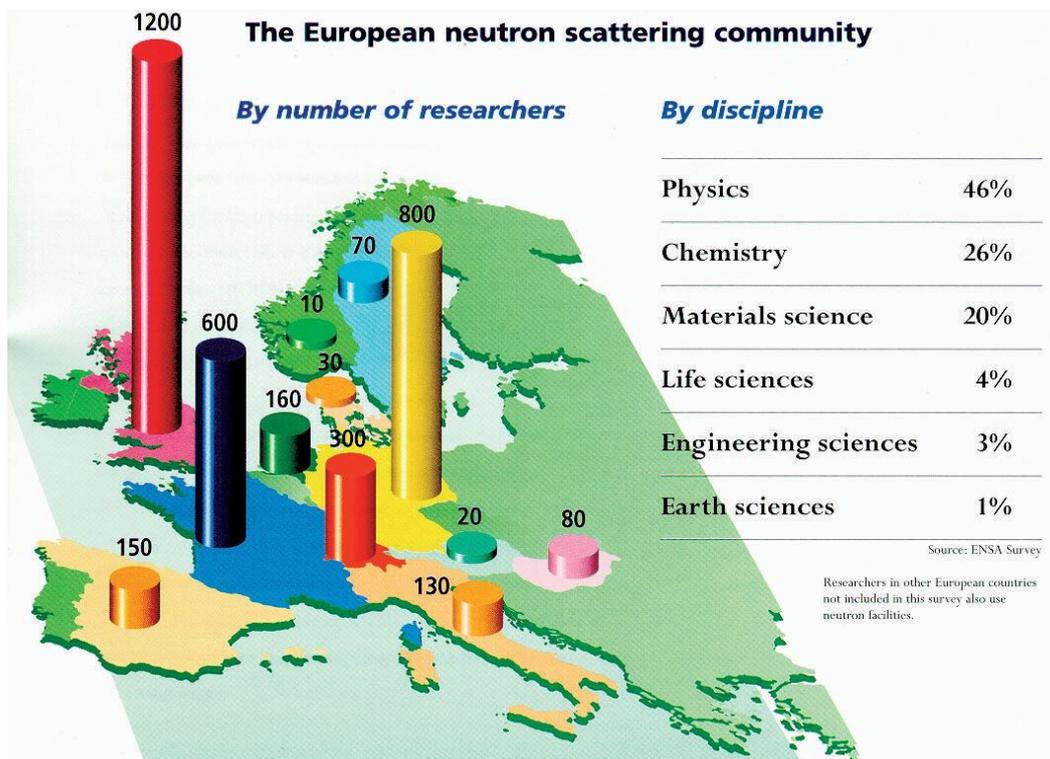


FIG. 9.1. Distribution of neutron users across European States and scientific disciplines (courtesy of the European Spallation Source Council).

## 9.2. NEUTRON SCATTERING APPLIED TO SCIENTIFIC RESEARCH

Two interactions of neutrons with matter make neutron scattering a unique probe for materials research, namely, short range strong nuclear interactions and electromagnetic interactions due to the neutronic magnetic moment. In particular, as neutrons have no net electric charge, they can penetrate the bulk of materials, making metallic objects largely transparent to their transmission. Furthermore, thermal neutrons play a crucial role, among many other engineering functions, in locating light atoms such as hydrogen and oxygen in intricate structures, resolving the arrangements of magnetic moments in complex magnetic systems and assessing residual stress distribution in mechanical components. Typical example media include hydrogen storage materials, electrolytes and battery materials, and magnetic films. Isotopic substitution is exploited to elucidate the precise location of hydrogen atoms, for example, in polymers and biological molecules.

Many types of neutron scattering spectrometer are required in materials science since the accessible momentum and energy transfers depend on neutron energy and since resolution and detector coverage must be tailored to the science. Different neutron beam instruments not only complement each other, but are also used in conjunction with other modern research techniques using light in both optical and X ray energy ranges, as well as electron beams. In the following subsections, a brief introduction to the more common types of neutron scattering instrument is provided. Readers seeking advice on installing neutron beam instruments at their research reactors, in the first instance, may wish to review the web sites of the neutron beam facilities listed at the end of this section, followed by contacting the management of one or more of those facilities. In addition, it is often beneficial to involve senior scientists of the local synchrotron radiation research community, to obtain an appreciation of the scientific areas that a neutron beam facility could support.

Concerning the range of applications, the various types of neutron beam instrument can be grouped into three scientific themes:

- (1) Small scale structure studies of metals and alloys, inorganic compounds and ceramics, which use diffraction and mostly rely on thermal neutron beams, but sometimes require hot neutron beams;

- (2) Large scale structure studies of polymers, surfactants, micelles, macromolecules such as biological molecules, and multilayered solids, which mostly rely on cold neutron beams;
- (3) Atomic and molecular dynamics, which concern motion in all of the systems listed in the above two themes and may require thermal, cold or hot neutrons, depending on the energetics of the dynamical characteristics of the system of interest.

### 9.2.1. Small scale structures with diffraction methods

Modern materials sciences, including condensed matter physics and solid state chemistry, aim to explain the function of materials by their microscopic origin. Thermal neutrons have a wavelength of the order of the interatomic distance in matter, and therefore can be diffracted into different directions by any kind of material. Mathematically, this is formulated in the Bragg equation:

$$n\lambda = 2d(hkl)\sin\theta \quad (9.1)$$

In this equation, the diffraction angle  $\theta$ , which is one half of the scattering angle, of neutrons of wavelength  $\lambda$  depends on the distance  $d$  between the planes of atoms with Miller indices  $(hkl)$  contributing to the diffraction peak. Diffraction produces strong peaks in the scattering neutron distribution according to the Bragg equation, making it a valuable method for detailed atomic structure determination. There are several subsets of neutron diffraction methods as described in the following subsections. A comprehensive neutron beam facility is likely to include at least one member from each subset.

#### 9.2.1.1. Powder diffraction

When a polycrystalline material is placed in a beam, neutrons are scattered at specific angles corresponding to the spacing between atomic planes. By measuring these angles and intensities, the atomic structure of the material can be deduced. If an amorphous or liquid sample is used, instead of a crystalline powder, there will only be broad peaks at specific angles corresponding to average interatomic distances. Combined with a high flux neutron source, high intensity powder diffractometers are capable of performing very rapid, real time, in situ measurements of chemical and metallurgical processes (e.g. those involved in synthesizing new materials). The advantage of neutron scattering to investigate atomic and magnetic arrangements simultaneously is of crucial importance in the development of new magnetic materials of interest in computer and energy technologies.

#### 9.2.1.2. Single crystal diffraction

Single crystal diffraction is traditionally performed by rotating a single crystal with a volume of around  $1 \text{ mm}^3$  through all possible angles relative to a monochromatic neutron beam and measuring the intensity of scattering at selected orientations. This is known as the four circle method, which can provide precise information on the atomic structure and thermal vibration, particularly when hot neutrons are used. Although this is a sophisticated method for structure determination in complex materials, particularly in respect of the location of light elements such as hydrogen and oxygen, data collection times are long (i.e. several days) even at high flux reactors.

A new, faster method known as the quasi-Laue method, which uses a broad wavelength band and large area detector to increase drastically the speed of data collection, has recently emerged. With this method, data collection times are reduced to hours in high flux reactors, albeit with some loss of resolution.

#### 9.2.1.3. Residual stress

Internal and residual stresses have a considerable effect on material properties. Fatigue or application of strong forces causes changes in the interatomic distances that will locally affect the interatomic binding. Very small changes of the Bragg angle ( $\Delta\theta$ ) due to variations of the interplanar spacing ( $\Delta d$ ) are a measure of the internal stress thus created. Neutron diffraction provides a powerful non-destructive tool for stress analysis deep within a crystalline material. Apertures along the incoming and scattered beam define a small measuring volume within the object such that the crystal lattice used as an atomic strain gauge allows measurement of the strain distributions with

a 'millimetre' spatial resolution. The need for residual stress analysis in actual mechanical components, which is of great importance for industrial users, has led to the design of powerful neutron strain scanning instruments with enough space at the sample position and load capacity to precisely position very heavy parts up to approximately one tonne.

#### *9.2.1.4. Texture*

The crystallographic texture represents the orientation distribution of the crystallites in a polycrystalline sample. The existence of a non-isotropic orientation distribution for a specific material is an indication that some particular form of anisotropic process was involved in its production. It is then convenient to refer these orientation distribution functions to an appropriate sample coordinate system. The function may, of course, depend not only on a certain coordinate dimension but also on certain positions inside the sample. The measured crystallographic texture may also convey the state of the material, as it is expected to change as a function of thermomechanical treatment or a nucleation process. Neutron diffraction enables the measurement of complete pole figures in transmission mode and the analysis of large volumes up to several cubic centimetres. This technique is useful for large grain materials or heterogeneous materials, and in this case no surface preparation would be required for the sample. It also allows for characterization of the texture of minority phases.

#### *9.2.1.5. Diffuse neutron scattering*

Whereas diffraction provides information on the average crystallographic arrangement of atoms and molecules in solids, diffuse scattering provides information about departures from the average (i.e. atomic distributions that persist over regions of one to several nearest atomic neighbour distances) typically around 0.2–3 nm. Sometimes, particularly in more complex materials, the information obtained can be crucial in explaining physical properties. Diffuse neutron scattering is often performed with cold neutrons, and when polarized neutrons are used, this method has the unique ability to determine local magnetic order.

#### *9.2.1.6. Liquid and amorphous materials diffraction*

Liquids and amorphous materials such as glasses may possess highly regular molecular arrangements in terms of coordination and interatomic distances. However, the molecular building blocks are randomly arranged either statically (as in glasses) or dynamically (as in liquids). Neutron diffraction provides a powerful method for structural determination in these materials. The full power of the technique is only achieved when hot, short wavelength neutrons are used.

### **9.2.2. Large scale structures**

In a scattering experiment, neutrons are scattered by structures having a certain size and contrast to the surrounding matrix. Under the same conceptual basis of Bragg's law, larger bodies scatter to smaller angles and vice versa. A widespread interest has developed over the utilization of longer wavelength and smaller energy neutrons for diverse applications. These cold neutrons ( $\lambda \geq 0.4$  nm) are employed in studies on large scale structures such as polymers, biological molecules, aggregates in metallic alloys, magnetic flux lines in superconductors and magnetic domains, as well as in the investigation of low frequency modes in molecular clusters, quantum tunnelling effects and fundamental physics. A comprehensive neutron beam facility is likely to include at least a pinhole small angle neutron scattering (SANS) instrument and a reflectometer fed by a cold neutron source.

#### *9.2.2.1. Small angle neutron scattering*

By far the most popular neutron beam method for large scale structure determination is SANS using the pinhole method. This is applicable for materials in which structural correlations exist over a length scale from around 1 nm to around 0.5  $\mu\text{m}$ . It is a highly versatile technique that has been applied to investigations on a wide range of materials, including emulsions, polymers, colloids, superconductors, nanoparticles, porous media, geological samples, alloys, ceramics and biological molecules. In most of these studies, the unique capacity to

modify the contrast between the scattering object and the matrix, or even specific portions of the object, like certain units in a macromolecule, has been fully exploited by isotopic substitution. For example, in biological samples, hydrogen atoms in a molecule are commonly substituted by deuterium or contrast variations are produced by changing the H<sub>2</sub>O/D<sub>2</sub>O ratio in the solution. Magnetic nanoscale objects such as magnetic particles, clusters and vortices can also be studied using polarized neutron beams.

Good pinhole SANS instruments are very large (around 100 m<sup>2</sup>) and expensive in addition to requiring cold neutrons to be truly competitive. Several new concepts are under development to improve performance and reduce cost without increasing size. The most promising of these use beam focusing devices such as refractory lenses, magnesium fluoride crystals, sextuple magnets or ellipsoidal mirrors. Potentially, these devices can extend the range of pinhole SANS beyond correlation lengths of 1 μm.

#### *9.2.2.2. Ultra small angle neutron scattering*

Ultra small angle neutron scattering (U-SANS) is an alternative approach for characterization of large scale structures that is particularly effective for the largest correlation lengths, typically around 100 nm to around 10 μm, such as those found in industrial materials like paints and structural steels. This approach is sometimes referred to as the Bonse–Hart method, which was first developed for X ray small angle scattering. It is a relatively inexpensive and compact method that can be optimized with a thermal neutron beam. The main disadvantage of the method is the loss of 2-D information, which is critical for characterization of anisotropic distributions such as those found in materials under shear forces and in electromagnetic correlations in magnets and superconductors.

#### *9.2.2.3. Neutron reflectometry*

Many new synthetic materials with novel physical properties and applications have been developed with the production of thin films and multilayer systems using advanced technologies. A neutron reflectometer is dedicated to the study of interfaces by neutron reflection. The reflected intensity at the grazing angle of a non-polarized white neutron beam is measured as a function of wavelength. The variation of this reflection coefficient, or reflectivity, with the wave vector is linked to the concentration profile perpendicular to the interface. If this profile is represented by a succession of different layers, the thickness, composition and roughness of each layer may be determined within wide ranges. It is particularly useful for studying surfaces, buried interfaces, complex fluids such as surfactants, magnetic films, multilayered structures and processes that occur at surfaces and interfaces. A polarizer and a flipper can be installed in the reflectometer to perform polarized neutron measurements. With this method, surfaces and interfaces within a depth of around 100 nm can be studied. The use of polarized neutrons also allows a unique method for the reconstruction of magnetic properties and depth profiles near surfaces. Neutron reflectometry requires cold neutrons to be truly competitive.

#### *9.2.2.4. Quasi-Laue diffraction for biology*

The quasi-Laue method, which was mentioned in reference to single crystal studies of small scale structures, is beginning to find a role in the study of crystallography of macromolecules such as proteins and enzymes. In these applications, the quasi-Laue diffractometer is fed by a cold neutron beam, and selective isotopic substitution, such as deuterium for hydrogen, is employed to uniquely label functional end groups in the large molecular assembly. This method is highly complementary to those used at the more common X ray protein crystallography diffractometers found in modern synchrotron radiation research facilities.

### **9.2.3. Atomic and molecular dynamics**

The study of subatomic dynamics is of equal importance for the understanding of microscopic functionality of many materials as the determination of the positions of atoms. Inelastic scattering of neutrons, the measurement of the energy change of neutrons during scattering, illuminates several aspects of this field. When a neutron hits a molecule or atom, it can exchange kinetic energy with that particle. Quantum mechanics predicts that this energy transfer can only happen in units of the internal vibration frequencies of the bound atom or, in general, collective excitations that might be sustained by the scattering system. By nature, thermal neutrons have about the same

speed or kinetic energy as the vibrations in the solid state. Consequently, the neutron's speed alters drastically when scattered inelastically, and is therefore an excellent probe to measure amplitude and direction of elementary excitations. Powerful neutron inelastic techniques enable determinations of the strength, the characteristics of the interatomic forces and the magnetic coupling in a material.

Quasi-elastic scattering is sometimes seen as a broadening around elastic scattering peaks such as diffraction peaks. It signals the presence of diffusive processes in the material under study, such as ions or spin waves moving through the lattice. This knowledge is particularly useful for understanding the nature of diffusion of hydrogen in materials such as in battery technology, the atomic origin of thermoelectricity or the magnetic coupling responsible for the superconductivity in ceramic cuprates.

Inelastic and quasi-elastic scattering experiments tend to be more complicated than elastic scattering experiments. Several types of spectrometer have been developed, including the use of polarized neutron beams and polarization analysis, to explore different parts of the energy and momentum transfer phase space of materials. A comprehensive neutron beam facility is likely to include at least one triple axis spectrometer and one chopper spectrometer.

#### *9.2.3.1. Triple axis spectrometry*

Triple axis spectrometry was the first and remains the most common form of inelastic scattering spectrometer at research reactors. It is essentially an extension of the basic two axis diffractometer, which was developed for powder diffraction. In a triple axis diffractometer, a third axis is added between the sample and detector to measure the energy of the scattered neutrons by diffraction from a crystal analyser. This type of spectrometer has played a major role in understanding collective atomic and magnet dynamics in condensed matter physics and solid state chemistry. Its great strength can only be realized when large single crystals of around  $1\text{ cm}^3$  are available. This requirement also represents the main limit to the application of the method. Triple axis spectrometers are generally served by thermal or cold neutron beams, but those for high energy excitations use hot neutron beams. The choice depends on the range of energy transfers that are of interest to the user community. Energy transfers of around 1–100 meV are accessible when both cold and thermal triple axis spectrometers are installed, generally with energy resolutions in the range of 0.1–3 meV.

Data collection is generally quite slow, as is the case with traditional single crystal diffraction, because the inelastic scattering signals are often weak and because of the need to carefully reorient the crystal for each successive data point. In some instances, considerable increases in data collection speed can be achieved by adding a multianalyser system.

#### *9.2.3.2. Time-of-flight spectrometry*

Another common form of inelastic spectrometer uses the TOF method to measure changes of energy through the change in velocity of scattered neutrons. The most versatile form of TOF spectrometer uses disc choppers to provide pulses of a broad spectrum of neutrons. The spectrometers themselves can be relatively simple, but simplification of the information gained can be quite complicated. One great advantage of the method is that surveys of the excitation spectra can be quickly obtained from polycrystalline materials. However, the great speed requires a wide angle detector bank, which is a considerable additional cost. TOF spectrometers tend to have broad appeal for research into chemical interactions and for a first survey of excitation spectra. Similarly to triple axis spectrometers, TOF spectrometers can be served by thermal, cold or hot neutron beams. Once again, the choice depends on the range of energy transfers that are of interest to the user community. Accessible energy transfers are similar to those of triple axis spectrometers, and in some instances, energy resolution can be tuned to around 50  $\mu\text{eV}$ .

#### *9.2.3.3. Backscattering spectrometry*

For really high resolution spectroscopy up to around 1  $\mu\text{eV}$  for studies of low energy atomic and molecular dynamics such as diffusive phenomena, triple axis and TOF spectrometers cannot compete. The backscattering method is now emerging as the most common form of high resolution inelastic and quasi-elastic scattering spectrometry. The method uses a chopped monochromatic beam of cold neutrons with a wavelength of around 0.628 nm and a wide angle array of perfect silicon crystals to analyse the energy of the scattered beam. Similarly

to TOF spectrometers, backscattering spectrometers measure energy transfers by the change in velocity of the scattered beam. These spectrometers are generally quite expensive but relatively simple to use, and are therefore quite well supported in major neutron beam facilities around the world.

#### 9.2.3.4. *Neutron spin echo*

A spin echo instrument measures the incoming and outgoing velocity of a neutron by making use of the Larmor precession of the neutron spin. A small energy transfer by the sample can be measured from the change in the neutron velocity. This concept differs significantly from the usual inelastic scattering instruments. The spin echo instrument is included in the category of inelastic scattering instruments with particular application to soft matter systems such as colloids, microemulsions and surfactants. This is by far the most sensitive of all inelastic scattering spectrometer, with an energy resolution as low as 1 neV. It requires cold neutrons and specialized expertise to build and operate.

#### 9.2.4. **Other neutron beam methods**

There have been many other neutron beam developments for specialized applications, some of which have made significant contributions to scientific research. Of these, perhaps the most common include instrumentation for fundamental physics, neutron optics and neutron radiography. The latter, the simplest neutron beam technique, is a direct imaging technique, described in Section 8.

Applications of neutron beam methods in fundamental physics address such issues as neutron lifetime, electric dipole moment and quantum gravity — all of which are closely linked to an understanding of the physical principles of nature as described by quantum mechanics. Generally, such instruments are fed by ultracold neutrons, which are neutrons with exceptionally low energy and velocity, which can be sourced by selective deceleration of neutrons produced in a cold neutron source. Such applications and instrumentation are beyond the scope of this publication, but further information can be found on the web site of Institut Laue-Langevin (see Bibliography to Section 9).

Neutron optics concerns the development of devices dedicated to the handling of neutron beams. Commonly available optical mechanisms for neutron beams include reflective, refractive and diffractive processes — all of which are used in the various forms of neutron spectrometry. In addition, there is a range of spin dependent processes that can be utilized for control of neutron polarization (see Section 9.3.4). Neutron resonances are also sometimes used for selective filtering of neutron beams. Further information on such devices can be found in *International Tables for Crystallography, Volume C: Mathematical, Physical and Chemical Tables* [9.2].

### 9.3. NEUTRON BEAM SOURCES

Most material structure and dynamics studies are performed using research reactors in the medium and high flux ranges. In fact, many high flux research reactors have been constructed primarily for this purpose, although an alternative production method is one based on spallation neutron sources. Spallation neutron sources are based on accelerated proton beams that strike a heavy metal target, thus evaporating neutrons from the excited target nuclei. The fast neutrons thus produced must be slowed down to thermal energies by use of appropriate moderator materials in optimized configurations. In addition to the mechanism for neutron production, the neutron sources can also be classified as stationary or pulsed sources, depending on whether the fast neutrons are produced continuously or not. Most spallation facilities are pulsed neutron sources such as the Spallation Neutron Source at the Oak Ridge National Laboratory, in the United States of America. A very useful continuous beam spallation source is the Swiss Spallation Neutron Source at the Paul Scherrer Institute, in Villigen. Some comparative characteristics between reactor and spallation sources are provided in Table 9.1.

TABLE 9.1. COMPARISON OF REACTOR AND SPALLATION SOURCES

Short pulse spallation source	Reactor
Energy deposited per useful neutron is $\sim 20$ MeV	Energy deposited per useful neutron is $\sim 180$ MeV
Neutron spectrum is ‘slowing down’ spectrum — preserves short pulses	Neutron spectrum is Maxwellian
Constant, small $\delta\lambda/\lambda$ at large neutron energies, excellent resolution, especially at large quantities and high energy	Resolution can be more easily tailored to experimental requirements, except for hot neutrons, for which monochromator crystals and choppers are less effective
Copious hot neutrons, very good for measurements at large quantities and high energy	Large flux of cold neutrons, very good for measuring large objects and slow dynamics
Low background between pulses, good signal to noise ratio	Pulse rate for TOF can be optimized independently for different spectrometers
Single pulse experiments are possible	Neutron polarization is easier

**Source:** Adapted from Pynn, R., “Lecture 2: Neutron scattering instruments and facilities”, Los Alamos National Laboratory.

In both fission and spallation processes, neutrons are produced with high kinetic energies of several MeV, and are referred to as fast neutrons. Different types of moderator are employed to decelerate these fast neutrons down to appropriate energies in the range of around  $10 \mu\text{eV}$  to around  $600 \text{ meV}$  for neutron beam applications. While the key parameter for most applications is the flux at the sample location in the beam, one can give a broad assessment of a reactor’s potential for materials studies by discussing the peak thermal flux in the reflector vessel. Peak thermal neutron fluxes in the reflector vessel exceeding  $10^{15} \text{ cm}^{-2}\cdot\text{s}^{-1}$  are currently available at only a few high flux research reactors. However, in most research reactors currently operating with neutron beam instruments, the thermal neutron flux is  $10^{14}\text{--}10^{15} \text{ cm}^{-2}\cdot\text{s}^{-1}$ . While some neutron scattering experiments are possible in reactors with a thermal neutron flux less than  $10^{14} \text{ cm}^{-2}\cdot\text{s}^{-1}$ , the scope for original materials research is quite limited. Such low flux reactors can sometimes develop new techniques (e.g. advanced neutron optical devices and detectors) or unconventional conceptual experiments that can then be taken to high and medium flux research reactors for further development. In addition, low flux research reactors can be used for demonstration experiments for students and hands-on training of scientists for research careers in neutron scattering. The layout of the neutron beam hall at the 14 MW Orphée reactor is an example of a dedicated medium flux research reactor, is shown in Fig. 9.2. Note that Orphée is equipped with cold, thermal and hot neutron sources.

### 9.3.1. Reactor sources for neutron beam experiments

Extraction of neutron beams requires the insertion of helium gas filled beam tubes into the reflector area of the reactor vessel. For effective beam delivery, it is essential to position the beam tube ends as near as possible to the peak in the thermal neutron flux distribution, generally less than  $0.5 \text{ m}$  from the core edge. Care must be taken to orient the beam tubes to minimize contamination by fast and epithermal neutron and source gamma rays. This is normally achieved by positioning the tubes tangentially to the core. Commonly, neutron guide tubes are inserted into the beam tubes to transport neutrons to distance spectrometers. The starting point of the neutron guides are to be close enough to the source end to extract a large portion of the neutrons entering the tubes, but far enough away to avoid degradation of the reflecting surfaces due to radiation damage. Generally, this distance is around  $2 \text{ m}$  from the source end of the beam tube. Care must also be taken in the selection of metals for neutron beam tubes because the radiation doses in the reflector may result in expansion or embrittlement. A replacement mechanism often is included in the design of beam tubes to guard against failures in service.

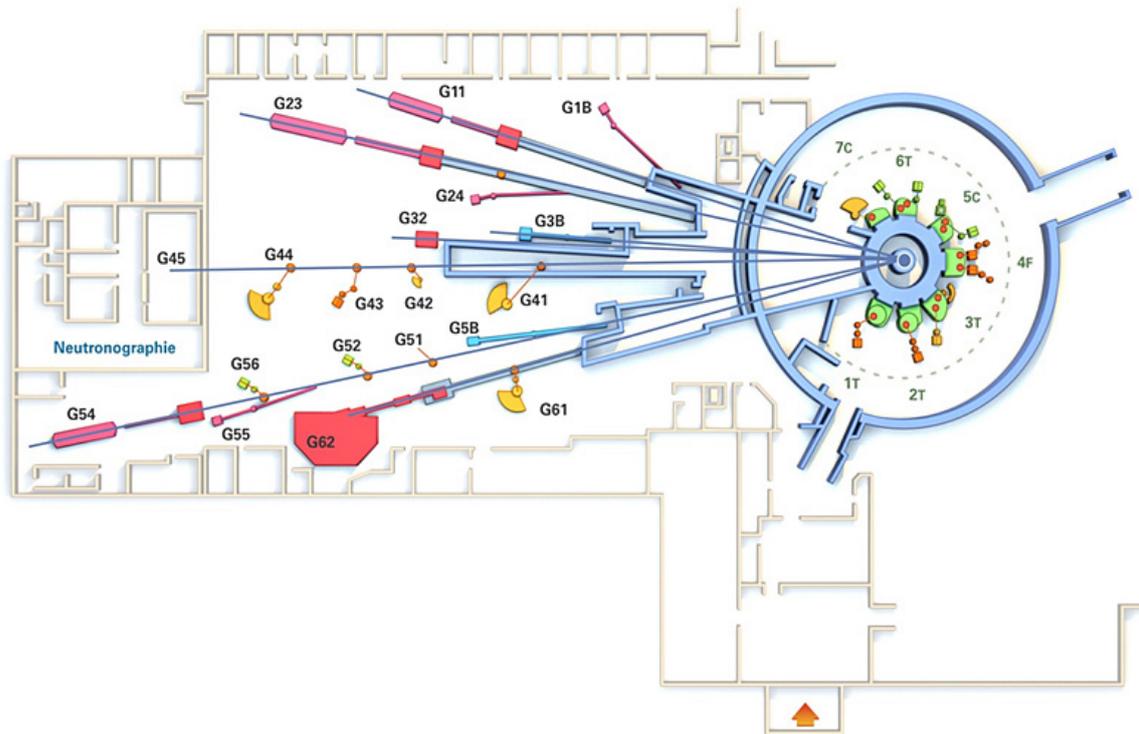


FIG. 9.2. Layout of neutron scattering instruments around the Orphée research reactor (courtesy of the French Alternative Energies and Atomic Energy Commission).

### 9.3.2. Neutron moderators for neutron beams

The most effective neutron moderators feature high concentrations of light elements because energy transfer from neutrons is maximized when the mass ratio between the target element or molecule and the neutron is lowest. Consequently, the more common moderators are heavy water, beryllium and graphite. Note that thermalization by light water tends to be too strong for efficient extraction of thermal neutron beams. The energy spectrum of a neutron beam is normally characterized by the temperature of the moderator, so that thermal neutrons are sourced in the reflector at typically around 310 K, cold neutrons are sourced in cryogenic moderators at typically around 20 K, and hot neutrons are sourced in graphite moderators at temperatures around 2300 K.

A cold neutron source positioned beside the isotope irradiation and NTD silicon irradiation cavities in the core assembly of the Open Pool Australian Lightwater (OPAL) reactor of the Australian Nuclear Science and Technology Organisation (ANSTO) is illustrated in Fig. 9.3. A cold neutron source is essentially a medium that re-thermalizes the thermal neutron spectrum established in the moderator in which it is immersed. The re-thermalization process originates as a thermodynamic equilibrium between the thermal neutron field and a moderating medium characterized by a low effective temperature. Such an effective temperature and the corresponding characteristic energy of the Maxwellian distribution of neutron velocities are directly related to the physical temperature of the cold source, the molecular dynamics properties of the constituent material and the cross-sections of the elements involved. The most common reactor based cold source moderators are liquid hydrogen and liquid deuterium. The former is relatively inexpensive to build and operate because it is more compact and requires less cryogenic cooling capacity, typically less than 1 kW cooling power, to maintain. Liquid deuterium moderators are generally considered to provide a superior cold neutron spectrum, but are much larger and therefore more expensive to build and maintain, typically requiring around 5 kW cooling power.

When a cold neutron source is installed with cold neutron guides, potential utilization is greatly enhanced for the study of large scale structures by SANS or reflectometry, for example, as well as diffuse scattering, fundamental physics, inelastic scattering and quasi-elastic scattering. The extent of the guide facility for the new cold neutron beam at the High-flux Advanced Neutron Application Reactor (HANARO) in the Republic of Korea can be seen

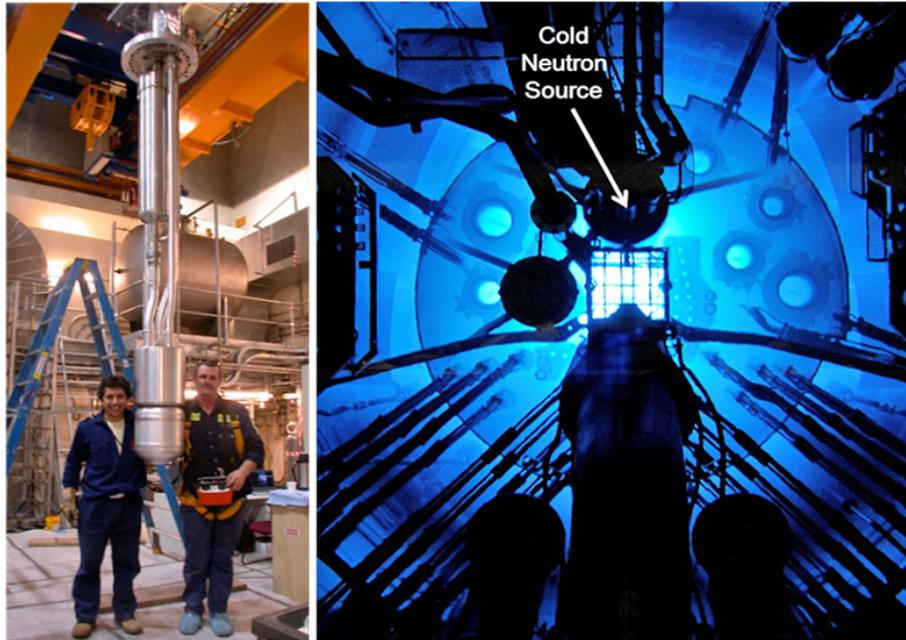


FIG. 9.3. The 20 L cold neutron source before (left) and after (right) being positioned in the OPAL research reactor (courtesy of ANSTO).



FIG. 9.4. The newly installed cold neutron guide hall at the HANARO (courtesy of the Korea Atomic Energy Research Institute).

in Fig. 9.4. However, the costs for construction of a cold neutron source, as well as the maintenance and operating costs, can be very high and are in the range of US \$1–5 million. These costs and the potential number of users must be considered when a cold neutron source installation is planned for a medium flux reactor.

Hot neutron moderators at research reactors are less common than cold neutron moderators. Those in current operation are made of graphitic compounds, have a volume in the range of 10–20 L and rely on nuclear heating to maintain high temperatures. The main applications for hot neutron beams are high resolution crystallography, structural studies of liquids and amorphous materials such as glasses, magnetic spin density studies and high energy inelastic scattering.



FIG. 9.5. Thermal neutron beam guide tubes at the 20 MW OPAL research reactor (courtesy of ANSTO).

### 9.3.3. Neutron beam transport

The optical properties of neutrons enable them to be guided by simple reflection from perfect surfaces. This is only possible for small incident angles due to the relatively small difference in the refraction index between vacuum and matter. Modern neutron guides have supermirror coatings to improve transport performance. Supermirror coatings consist of several hundreds of thin alternating layers of nickel and titanium sputtered onto near perfect glass substrates, thus adding a broad band of Bragg like diffraction to the angle of total reflection.

Guides are used to conduct neutrons from the beam tube to a region far from the background radiation near the reactor. In many cases, the neutrons are conducted through a guide into a separate building adjacent to the reactor building to provide a major improvement in the signal to noise ratio in neutron transportation. While guides are routinely utilized at high flux research reactors, they are also useful for medium flux reactors. An example of a layout of neutron beam guide tubes is shown in Fig. 9.5. The guide tubes are coated with nickel/titanium multilayers, to enhance reflectivity, and encased in steel vacuum housing. The whole system is enclosed in a concrete bunker with wall thickness of more than one metre. Note that the presented guide dimensions are 300 mm high, 50 mm wide and over 50 m long.

In addition to the principles of insertion of neutron guides into the in-pile neutron beam tubes outlined earlier, extra protection with out-of-pile neutron guides must be in place to guard against excess radiation from source neutrons, gamma rays and gamma radiation produced in the guide systems themselves. This issue is far from trivial and necessitates such mechanisms as curvature of the guide system beyond the point of line of sight back to the moderator and construction of thick concrete guide bunkers over distances of more than 20 m from the reactor surface. Once guides emerge from the guide bunkers, they are normally carefully shielded to reduce the radiation levels of gamma rays leaking from the guides.

### 9.3.4. Neutron polarization

Polarized neutrons are used to differentiate between nuclear and magnetic scattering processes. They have always been a popular aspect of research on condensed matter physics at research reactors, and are now becoming more widely used, particularly in the areas of nanotechnology and materials science for information technology.

It is possible to achieve very high neutron polarizations (around 99%) and to rotate the polarization direction of a neutron beam with a range of electromagnetic devices. Such devices are integral to the implementation of methods such as neutron spin echo, described earlier in this section. The choice of polarization method depends on the energy of the neutrons and the scattering phenomena of interest.

Traditionally, Heusler crystals ( $\text{Cu}_2\text{MnAl}$ ) have been used to polarize hot neutrons, while polarizing magnetized supermirrors or ferrous transmission filters have been used for thermal and cold neutrons. Both types of polarizer have the advantage of being passive systems (i.e. once they are built and installed, there is no limit on their usable lifetime with only minimum maintenance).

In recent years, gaseous  $^3\text{He}$  filters have been shown to perform well across a wide wavelength range. The advantage of these filters is that they permit the transmission of wide angle neutron beams, which Heusler crystals and supermirrors cannot. The disadvantage of  $^3\text{He}$  filters is that the technology is more complicated, and the lifetime of the filters is short, typically a few days, implying that the  $^3\text{He}$  cells must be recharged regularly to maintain their high performance. The decision on which technology to invest depends largely on the scope and volume of polarized neutron applications envisaged for the facility. Small scale operations tend to favour the passive methods, whereas larger operations may benefit from investment in  $^3\text{He}$  based technology.

## 9.4. RECOMMENDATIONS FOR UTILIZATION OF A MEDIUM POWER RESEARCH REACTOR

### 9.4.1. Flux/power level requirements

While the staff at a medium flux research reactor need always to consider improving or upgrading the reactor and experimental equipment, thereby increasing the intensity of neutrons available at the sample, the role of these reactors in material structure studies are not to be underestimated. If a thermal neutron flux of the order of  $10^{14} \text{ cm}^{-2}\cdot\text{s}^{-1}$  can be accessed by neutron beam tubes, a range of neutron scattering instruments may be successful. Below this flux level, any neutron beam programme is likely to have limited impact in innovative scientific research.

### 9.4.2. Reactor facility requirements

The main impediment to successful material structure experiments at a medium flux research reactor is the amount of time necessary to collect data. An important role of these research reactors, however, is supplying and testing new concepts for eventual implementation and data collection at a high flux reactor. In fact, many of the ideas and concepts for new research or improving instruments installed at a high flux reactor were initiated at lower flux research reactors. This is because the small research reactor's mission is preferably to providing well established and reliable tools to tackle specific problems, rather than using the extremely expensive capital and human resource infrastructure of a very intense neutron source to explore new ideas.

In addition, graduate training in neutron scattering can be more easily performed in the environment of medium flux research reactors. Scientists trained at these facilities are then a valuable source of skilled personnel at high flux reactors.

A neutron beam facility at a multipurpose reactor requires a dedicated area for the neutron beam instrumentation, preferably removed some distance from the source using heavily shielded neutron guides. In addition, specialized laboratories for processing chemical, biological and materials science samples are required, as well as workshops for mechanical, cryogenics and electronics support.

### 9.4.3. Experimental equipment requirements

Some basic information on the broad parameters of neutron scattering instruments is given in Table 9.2. The reader considering establishment of any of the methods discussed in this section would do well to visit the web sites listed in the Bibliography to Section 9 to obtain up to date information on the performance standards, the latest technology and the scientific outputs of various diffractometers and spectrometers around the world.

Unequivocally, no modern neutron scattering facility can compete without substantial investment in special sample environments for the samples under investigation. Such equipment is to be readily transferrable among a range of spectrometers, and to enable temperature control in the range of 2–2800 K, pressure control between vacuum (mPa) and 10 GPa and magnetic fields up to 10 T. In some cases, more than one environmental condition needs to be regulated by a single device, as in control of temperature and pressure for residual stress measurement, or control of temperature and magnetic field for diffraction and inelastic scattering experiments. As a preliminary

TABLE 9.2. GENERIC TABLE OF NEUTRON BEAM INSTRUMENT PARAMETERS

Instrument type	Applications	Hardware cost (US \$ million)	Source spectrum	Flux at sample ( $\text{cm}^{-2}\cdot\text{s}^{-1}$ )
Powder diffractometer	Magnetic and crystal structures, in situ or real time experiments; also, liquids and amorphous solids with hot neutrons	3–5	Thermal or hot	$>10^5$
Four circle diffractometer	Precise determination of atomic and magnetic structure of single crystals Hot neutrons are more precise with small cells	1–2.5	Thermal or hot	$>10^5$
Quasi-Laue diffractometer	Rapid determination of atomic and magnetic structure of single crystals (also macromolecules with cold neutrons)	1.5–3	Thermal or cold	$>10^6$
Residual stress diffractometer	Determination of residual stress in engineering components	2–4	Thermal	$>10^5$
Pinhole SANS <sup>a</sup>	Large scale structure determination, such as polymers, biological molecules, magnetic domains	4–7	Cold	$>10^6$
U-SANS <sup>b</sup>	Large scale structure determination extending beyond pinhole range	1.5–3	Thermal or cold	$>10^5$
Reflectometer	Structural studies of surfaces and interfaces	2–4	Cold	$>10^6$
Triple axis spectrometer	Atomic, molecular and magnetic excitations in single crystals	3–5	Thermal, cold or hot	$>10^7$
TOF <sup>c</sup> spectrometer	Atomic, molecular and magnetic excitations in polycrystals	4–7	Thermal, cold or hot	$>10^7$
Backscattering spectrometer	High resolution studies of atomic and molecular excitations	4–7	Cold	$>10^6$
Neutron spin echo	High resolution studies of diffusion and excitations in soft matter systems	3–5	Cold	$>10^6$

<sup>a</sup> SANS — small angle neutron scattering.

<sup>b</sup> U-SANS — ultra small angle neutron scattering.

<sup>c</sup> TOF — time-of-flight.

estimate, approximately 10% of the cost of the neutron spectrometers is to be spent on special sample environment equipment.

#### 9.4.4. Personnel requirements

Construction of a front line spectrometer at a high flux reactor requires extensive consultation with stakeholders and international experts. The design and construction process entails development of some unique custom built components. Generally, the period from design through construction to commissioning will take on average 15 person-years and 5 years to complete.

For full user service over an operational period of 150–250 days per year, five to six staff members per neutron beam instrument are required. Note that this figure factors in overheads such as management, administration, a user office and a pool of technical staff for the sample environment, user support and facility maintenance. Generally, two to four dedicated scientific and technical staff per instrument are required.

#### 9.4.5. Funding requirements

Construction of a front line spectrometer at a high flux reactor generally costs US \$5–10 million per instrument. These figures are full cost estimates including cost of labour (approximately 25%) and project related activities such as consultation, project equipment and travel (approximately 10%).

The cost of a neutron guide system is generally in the range of US \$20 000–30 000 per metre of guide for manufacture, installation and alignment.

### REFERENCES TO SECTION 9

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- [9.2] INTERNATIONAL UNION OF CRYSTALLOGRAPHY, International Tables for Crystallography, Volume C: Mathematical, Physical and Chemical Tables (PRINCE, E., Ed.), Kluwer Academic Publishers, Dordrecht (2006).

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BLAKELEY, M.P., LANGAN, P., NIIMURA, N., PODJARNY, A., Neutron crystallography: Opportunities, challenges, and limitations, Curr. Opin. Struc. Biol. **18** 5 (2008) 593–600.

CAMPO, J. (Ed.), Neutrons Reveal All: A Complimentation of Divulgative Articles, European Spallation Source, Bilbao (2008).

REIMERS, W., PYZALLA, A.R., SCHREYER, A., CLEMENS, H. (Eds), Neutron and Synchrotron Radiation in Engineering Materials Science, Wiley-VCH Verlag GmbH & Co, Weinheim (2008).

Web sites of reactor based neutron beam research facilities include:

- ANSTO: <http://www.ansto.gov.au>
- HANARO: <http://hanaro4u.kaeri.re.kr/main.html>
- Helmholtz-Zentrum Berlin: <http://www.helmholtz-berlin.de>
- Institut Laue-Langevin: <http://www.ill.eu>
- J-PARC: <http://j-parc.jp/en/access.html>
- Leon Brillouin Laboratory: <http://www-llb.cea.fr/en>
- NIST Centre for Neutron Research: <http://www.ncnr.nist.gov>
- Oak Ridge National Laboratory: <http://neutrons.ornl.gov/facilities/HFIR>
- Technische Universität München: <http://www.frm2.tum.de/en/index.html>

## 10. POSITRON SOURCES

High intense positron beams are of major interest in various fields of physics. The positron annihilation technique is applied in solid state physics, materials science and surface physics, in which positrons are used as a non-destructive microprobe for defect studies owing to their high sensitivity to open volume defects. In atomic and nuclear physics, scattering experiments with positrons and positronium, as well as the investigation of positronium and its excited states, require intense positron beams.

Doppler broadening measurements of the positron annihilation line with a scanning positron beam enable defect mapping in samples. With coincident Doppler broadening spectroscopy, information about the chemical surrounding of the positron annihilation site is also obtained. Depth dependent information on the defect type and concentration is obtained by positron lifetime measurements using a pulsed beam. A submicrometre sized positron beam can be created for defect depth profiling on a lateral scale smaller than one micrometre. Two dimensional angular correlation of annihilation radiation (2D-ACAR) allows high resolution measurements of electron momentum distribution and thus the electronic structure of matter.

In the laboratory, positron beams are based on  $\beta^+$  sources (e.g.  $^{22}\text{Na}$ ) combined with tungsten moderators with beam intensities usually not exceeding a few million moderated positrons per second. High intensity positron beams of  $>10^7$  moderated positrons per second can be produced at research reactors.

Positrons can be created by  $\beta^+$  active sources or by pair production. There are different methods to establish a positron source at a research reactor:

- (a) Production of  $\beta^+$  sources by neutron activation is brought about when a suitable target is placed at, or near the reactor core to activate the source material (e.g. copper) by thermal neutrons. The positrons of the  $\beta^+$  active source can then be extracted (e.g. by electric fields) or the positron emitting source is moved out of the activation zone of the reactor to the experimental set-up.
- (b) The absorption of high energy gamma rays by a high Z target, such as platinum or tungsten, leads to the production of electron–positron pairs. These gamma rays may be emitted directly from the reactor core as a consequence of fission or following the capture of thermal neutrons by a material with a high capture cross-section. In this latter case, cadmium is a suitable material since the emitted gamma rays are sufficiently energetic for creating electron–positron pairs in materials such as platinum and tungsten. In addition, positrons are also created by hard gamma radiation that arises either from neutron capture gamma rays from surrounding materials or directly from fission and fission products.

These positron sources show a broad positron spectrum that reaches up to 1–3 MeV. Therefore, positron moderation is required to produce a positron beam within a sharp energy range. Platinum and tungsten are suitable materials since they have a negative positron work function of 1–3 eV. Because the positron diffusion length inside the moderator is typically about 50–100 nm, almost all positrons slowed down in a border zone of 50–100 nm at the surface have a high probability of being emitted from the surface. The emitted slow positrons are extracted by electrostatic fields and guided through a static magnetic field. Inside the magnetic field, the transversal momentum of the positron leads to helical positron trajectories.

The main challenges are coping with the hostile environment to maintain an ultrahigh vacuum and electrical fields located close to the reactor core. In addition, radiation induced defects by high energy neutrons in the moderator material lead to a degradation of the moderator. The design of the positron source converter and moderator, as well as the extraction of slow positrons to form a high brilliant beam, is also a delicate matter. Figure 10.1 shows the layout of the positron source POSH at Delft. On the left, four discs of cylindrical tubes generate the positrons either by neutron activation of copper or by pair production. The positrons are moderated by tungsten foils inside the cylinders. A few calculated trajectories are shown of moderated positrons that are accelerated and focused by electrical lenses and transported by an axial magnetic field generated by a coil.

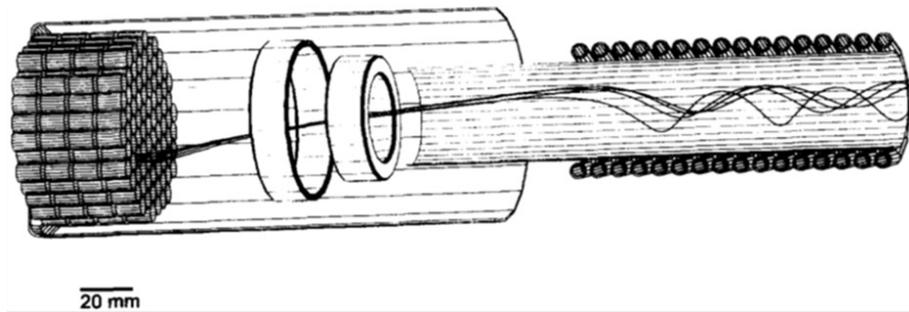


FIG. 10.1. The positron source POSH of the Delft reactor (courtesy of Delft University of Technology).

The Neutron Induced Positron Source Munich (NEPOMUC), an in-pile positron source, is mounted inside the tip of the inclined beam tube SR11 inside the heavy water moderator tank of the reactor FRM II at Technische Universität München (see Fig. 10.2). After thermal neutron capture in cadmium, positrons are generated by pair production following absorption of high energy prompt gamma rays in platinum. After moderation in platinum foils, the positrons are extracted by electrical lenses and then magnetically guided to the platform outside the biological shield of the reactor

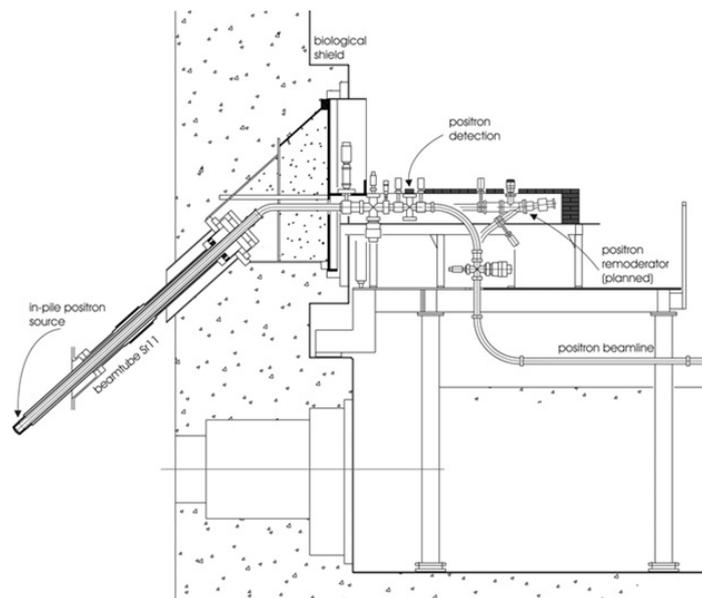


FIG. 10.2. The NEPOMUC at the research reactor FRM II (cross-sectional view) (courtesy of Technische Universität München).

## 10.1. POSITRON SOURCE REQUIREMENTS

### 10.1.1. Flux/power level requirements

The positron intensity obtained depends on many aspects, such as the basic method used to create the positrons, source design, converter and moderator material. A rough estimate of positron intensity indicates that approximately  $10^7$  moderated positrons per second can be created at the minimum reactor power level of 100 kW, with an available thermal neutron flux of around  $10^{12} \text{ cm}^{-2} \cdot \text{s}^{-1}$ .

### 10.1.2. Reactor facility requirements

The target normally occupies a position at, or near, the reactor core, with the positron beam emanating from a beam tube. Ideally, the installation will be retractable when not in use to minimize target costs and interference with other reactor experiments. Shielding needs to be designed for enhanced flexibility and mobility.

The total floor space required by the experiment largely depends on the application of the positron beam. At least 20 m<sup>2</sup> of floor space is required at the beam port. A 2D-ACAR experiment requires a floor space with a minimum length of 30 m. An effective floor space of 25 m<sup>2</sup> is desirable for each additional experimental set-up.

### 10.1.3. Experimental equipment requirements

The minimum additional equipment requirements are:

- Ultrahigh vacuum equipment;
- Positron beam extraction devices, including electric and magnetic field equipment with high voltage supplies;
- Positron and gamma detectors;
- Source and target handling equipment;
- Radiation detection and shielding equipment;
- Facilities required for irradiated target disposal.

The desirable additional equipment includes positron beam remoderation equipment and general laboratory facilities.

Facilities for target production are:

- A clean room facility;
- Experimental measurement facilities such as Doppler broadening, coincident Doppler broadening, positron lifetime and 2D-ACAR;
- Experimental equipment for complementary techniques, such as a scanning electron microscope.

### 10.1.4. Personnel requirements

A scientist and a technician are required for the initial feasibility study and design of the positron source. Personnel from the reactor operations group are required for the safety review and installation of the beam tube with in-pile components.

A scientist, as a positron facility supervisor, and at least one operator are required for the positron source and operating the positron beam facility. A technician needs to be available for the beam line and the experimental facilities. In addition, at least one scientist is to be responsible for each spectrometer; additional scientists, such as postdoctorate and doctorate students, are desirable.

A facility of this type would normally be utilized by a large group of knowledgeable resident professional scientists and scientific guest users conducting research in several areas. Utilization of the positron facility is greatly enhanced if the operating organization or host group provides expertise and support to potential and actual users.

### 10.1.5. Funding requirements

Investment funds for the beam tube and in-pile components are estimated at US \$500 000–700 000, excluding the experimental measurement facilities listed in Section 10.1.3.

The annual budget to operate a reactor based positron source is approximately 10–20% of the investment cost. A positron source at a research reactor is an attractive user dedicated facility. Funding needs to be available to invite external scientists — contract research agreements are normally a component of the utilization of the positron facility.

### 10.1.6. Time requirements

Installation requires 5–10 years.

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## 11. NEUTRON CAPTURE THERAPY

When  $^{10}\text{B}$  absorbs a neutron, it emits an alpha particle, which is highly ionizing and has a range in tissue about equal to the diameter of a cell. The reaction is  $n + ^{10}\text{B} \rightarrow ^4\text{He} + ^6\text{Li}$ . The applicable methodology in boron neutron capture therapy (BNCT) is thus to inject a tumour with a borated compound that irradiates it with thermal neutrons. If the conditions are right, the tumour dose is much higher than that of the rest of the surrounding tissue, resulting in preferential eradication of the tumour cells.

Thermal neutrons are desired at the tumour location because the  $^{10}\text{B}$  interaction probability is much higher with slower neutrons. Surface or shallow tumours can therefore be irradiated with thermal neutrons, while those at a depth of a few centimetres can be irradiated with epithermal neutrons, which are then thermalized as they pass through and interact with overlying tissue. Thermal neutrons are also useful for BNCT research involving cell cultures or small animal irradiations. Although most neutron capture therapy currently practised makes use of boron compounds, other compounds (e.g. those of gadolinium) can also be used.

The majority of neutron capture therapy research has focused on malignant melanomas and brain tumours, particularly glioblastoma multiforme. Current research efforts are directed towards providing the dose to the tumour in a short time period of minutes, instead of hours, and reducing the dose to normal tissue through higher flux, better neutron energy selectors, shielding and collimation, and better drugs.

BNCT research varies in several aspects from one treatment centre to another, for example:

- (a) In Japan, thermal neutron beams have typically been used in conjunction with surgery.
- (b) In Europe and the United States of America, epithermal beams are more commonly used.
- (c) In the United States of America, the dose is usually administered in one fraction or session.
- (d) In Europe, the (multi)fractionated dose technique is used.

The reactor facility, while obviously important, is only one part of a very large and necessary infrastructure for the performance of neutron based therapy, as discussed later (see Fig. 11.1).

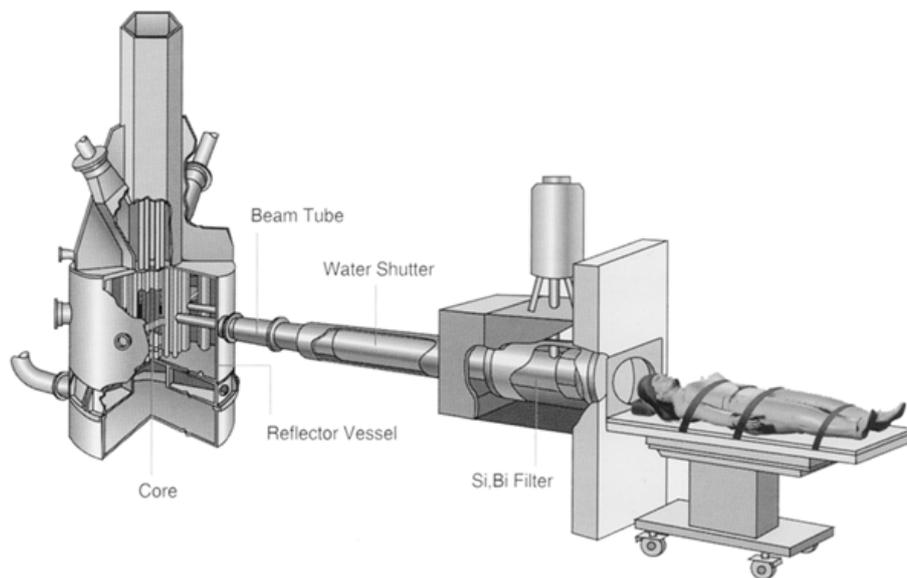


FIG. 11.1. Example of a neutron capture therapy arrangement utilizing a research reactor (taken from Ref. [11.1]).

## 11.1. BORON NEUTRON CAPTURE THERAPY REQUIREMENTS

### 11.1.1. Flux/power level requirements

In the early years of BNCT, most facilities used thermal neutron beams, but since the mid-1960s, the use of epithermal beams has dominated. Both systems are still used and both depend on reactor characteristics and the needs of the clinicians (i.e. which type of tumour is to be treated). The current discussion focuses on epithermal beams, since that is the direction which research appears to be taking. The accepted definition in the BNCT community for epithermal neutrons is those in the range of 0.5 eV to 10 keV. Current experience shows that the desirable minimum epithermal neutron beam intensity is  $10^9 \text{ cm}^{-2}\cdot\text{s}^{-1}$ . Beams of around half this intensity are usable, but result in rather long irradiation times.

Tumour boron concentration affects the requirements of beam intensity. If the concentration can be raised from the current values, the beam intensity requirements and thus the treatment times are reduced proportionally.

It should be recognized that the quality of the beam is often more important than the intensity. Beam quality is determined by four parameters under free beam conditions. These are listed in order of importance:

- (1) The fast neutron component needs to be reduced as much as possible while keeping the dose from epithermal neutrons as high as possible,  $\geq 2 \times 10^{-13} \text{ Gy/cm}^2$  per epithermal neutron.
- (2) The gamma ray component needs to be as low as possible to reduce unnecessary dose to normal tissue. As before, a target value of  $\geq 2 \times 10^{-13} \text{ Gy/cm}^2$  per epithermal neutron is desired.
- (3) The ratio of thermal flux to epithermal flux needs to be minimized to reduce damage to the overlying tissue (e.g. the scalp). A target value for this ratio is 0.05.
- (4) The ratio between the total neutron current and the total neutron flux provides a measure of the fraction of neutrons that are moving in the forwards direction. A high value of  $>0.7$  is desired.
- (5) Reasonably sized apertures (typically circular) of 12–15 cm are desirable.

### 11.1.2. Reactor facility requirements

A filtered collimated neutron beam with the characteristics discussed above is required to perform BNCT for deep seated tumours such as glioblastoma multiforme. There are several ways to obtain such a beam. A popular method moderates the fast neutrons channelled from the core down to the appropriate energy. This works well in facilities with a large thermal column that can be relocated and replaced with appropriate moderators and filters. The addition of a fission converter consisting of a row of fuel elements can significantly increase the epithermal neutron fluence rate at a distance from the core. Removal of some of the shielding around the core is an alternative to provide direct access to the core or for the installation of a converter in the case where the facility does not incorporate a thermal cavity or other large void space near the core.

The pure filtering method must be used in a reactor that only has a rather narrow and long beam tube available. Filtering transmits neutrons of the desired energy while blocking those of other energies. In general though, because filtering is more wasteful of neutrons, a much higher source flux and therefore reactor power is necessary.

In addition, it has been demonstrated that even a quite low power fast reactor, as low as 5 kW, can produce epithermal neutrons at the desired intensity levels.

At a multipurpose reactor, shutters are also required to stop the beam without shutting down the reactor. The beam must emerge into a well shielded treatment room that allows for observation of the patient during treatment, and the reactor control and safety systems must be expanded to include an interlocked system for the BNCT facility.

New reactors frequently incorporate facilities to enable BNCT capability in their design. However, most existing reactors were designed before the concept of BNCT, and generally have not done so. The ease at which an existing reactor can be modified depends on the facility design.

### 11.1.3. Experimental equipment and additional facility requirements

#### 11.1.3.1. At the reactor

It is necessary to create medical facilities such as a patient preparation and holding area at the reactor. These facilities are to provide a hospital atmosphere for the comfort of patients.

#### 11.1.3.2. At a nearby hospital

BNCT is a medical procedure that is administered under the supervision of a physician (see Fig. 11.2) — in particular a radiotherapist and medical staff associated with a nearby hospital. Often, the hospital is a teaching hospital associated with a medical school.



FIG. 11.2. A patient being carefully aligned and made comfortable prior to boron neutron capture therapy radiation (taken from Ref. [11.1]).

The hospital staff provide diagnostic equipment and services such as magnetic resonance imaging (MRI), computed axial tomography (CAT) scans, computer modelling, and patient selection and transportation. Clinical application of BNCT also requires monitoring boron in the blood and tissue of patients, both for treatment planning and during the actual application. The most commonly chosen methods to provide quick and reliable boron analysis are inductively coupled plasma optical emission spectrometry, inductively coupled plasma mass spectrometry or PGNAAs (the latter is naturally part of the reactor facility).

#### 11.1.3.3. Other requirements

BNCT requires the use of a dedicated treatment planning code based on mathematical modelling of the tumour and surrounding tissue, as well as modelling and incorporating into the planning calculations a well defined, fully characterized, incident radiation beam. These calculations determine the optimal treatment mode (i.e. the direction of the beam, number of irradiations, time of irradiation and the individual dose components). Normally, such codes are a variation of reactor physics codes based on a Monte Carlo algorithm. The treatment planning code requires the approval of a governmental health agency in addition to a nuclear regulatory body. In certain cases, medical liability insurance may be a requirement for the research reactor facility.

In addition to the clinical applications discussed above, research reactor facilities providing a thermal or epithermal neutron beam can also be used for preclinical and basic research. Most efforts take place in the fields

of dosimetry, treatment planning and radiation biology, including in vitro and in vivo studies. A fully functional BNCT facility is not required for many projects.

#### **11.1.4. Personnel requirements**

A large infrastructure with experts in many areas is required for the development and operation of the facility and for the medical treatment. Cooperation and interaction is required between these experts during the design, installation and operation of the BNCT facility.

##### *11.1.4.1. Physics organization*

Normally, it is the physicists or the nuclear engineers at the reactor facility that are required to provide the personnel for a feasibility study, the development of specifications, design, installation, testing and operation of the facility at the reactor. The host organization normally provides a senior staff member who is well acquainted with the reactor and facility, as well as most aspects of the treatment process, to serve as a joint supervisor for treatment, along with a physician.

##### *11.1.4.2. Medical organization*

The medical organization provides the support personnel for all the medical aspects of the design, testing and treatment planning, and the treatment itself. In addition to the personnel required to provide normal hospital functions such as MRI and CAT scans, the medical organization provides any expert personnel required. Typically, this might include:

- Several specialists (e.g. neuro-oncology, radiation oncology, nuclear medicine and radiology);
- Research nurse;
- Medical physicist for dosimetry;
- Medical technicians;
- Technician for computer modelling.

A clinical protocol must be prepared by the principal clinical investigator or treating physician. The protocol must generally be approved, and the treatment authorized by the responsible national health authorities. Within the protocol, apart from a description of the treatment itself, selection criteria for patients and informed consent must be included.

The medical organization is normally led by a senior physician who is well acquainted with BNCT and the BNCT facility, and who must work closely with the lead physicist.

#### **11.1.5. Funding requirements**

The cost for the installation of a BNCT facility at a reactor depends greatly on the modifications necessary to install the facility, as discussed earlier. Based on actual reactor modification costs at several research reactors, the following expenses are:

- Install a fission converter facility using reactor fuel into an existing space: US \$3.2 million.
- Create a large cavity in the reactor shield: US \$1 million.
- Design and fabricate shielding, filters, collimators and shutters: US \$650 000.
- Install the shielding, filters, collimators and shutters: US \$650 000.
- Install a facility in an existing thermal column: US \$775 000.

### 11.1.6. Time requirements

Based on worldwide experience, the time necessary for development of a BNCT facility, before any patient treatment can begin, is around five years.

### 11.1.7. Customer relations

Before any facility considers becoming involved with neutron capture therapy, two main factors are to be evaluated:

- (1) Since the efficiency of BNCT is still unproven, many people believe that there are sufficient facilities currently available for the necessary studies and that additional facilities are not necessary at this point.
- (2) Even if BNCT eventually becomes a viable treatment option, a market or needs analysis needs to be performed to determine the number of potential candidates that would be available for treatment. An example of a simplified approach to the study is given below.

The Central Brain Tumor Registry of the United States (CBTRUS) estimates that 3.2 out of 100 000 people develop glioblastoma multiforme in the United States of America each year [11.2]. The largest single age group diagnosed with this cancer is age 65–74, followed by age 75 and up, with approximately two thirds of diagnosed patients over the age of 65. Taking this incident rate, a State with a population of 30 million would expect 960 cases per year. Because the expected lifetime in some States is lower than in the United States of America, the incident rate should be lower. In addition, for a State with a widely dispersed rural population, some diagnoses will be missed. Based on this, the number of patients in such a State could be well below the 960 calculated.

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## 12. TESTING

One of the major applications of research reactors, whether low, medium or high power, is their ability to be utilized to perform testing and calibration experiments. This can apply to various types of instrumentation, as well as to materials intended for use in either nuclear fuels or reactor structural components. Some aspects of such applications are discussed in this section.

### 12.1. INSTRUMENT TESTING AND CALIBRATION

Almost any type and size of reactor can conduct some form of instrument testing and calibration, even if it is just for low level radiation protection instruments. Typically, the work involves neutron and gamma detection instruments that need to be tested to ensure they have the appropriate characteristics and need to be calibrated to ensure accurate readings. Examples of such detectors are fission chambers, SPNDs, nuclear heating calorimeters and radiological protection instruments.

#### 12.1.1. Flux/power level required

Calibration of neutron flux measurement instruments can typically be performed at any flux up to  $10^{14} \text{ cm}^{-2}\cdot\text{s}^{-1}$ , although relatively powerful installations are preferred. For example, the ISIS research reactor in Saclay, in France, a neutron mock-up of the OSIRIS reactor, is commonly used from zero power to its nominal rating of 700 kW for such experiments. Moreover, in the case of fission power chambers (see Fig. 12.1), the flux level requirement also depends on the operating mode to be tested, whether a pulse, Maxwell or current mode.

In the particular case of radiation protection instruments, the actual neutron flux and gamma radiation level requirements are quite low. The minimum requirements for this type of work depend on the type of testing and calibration being performed, but the necessary dose rate levels are often within the range of 1–3  $\mu\text{Sv/h}$  to 1–3 mSv/h.



FIG. 12.1. Examples of fission chambers with different diameters for the measurement of neutron flux (courtesy of the French Alternative Energies and Atomic Energy Commission).

### **12.1.2. Reactor facility requirements**

The test and calibration facility can be located inside the core, at the periphery or external to reactor shielding, with neutrons delivered, for example, outside the shielding through neutron guides. Nevertheless, citing safety reasons, this latter case is more complex. The risk of reactivity perturbations may indeed prohibit any movement of the device during the entire reactor operating period, limiting the measurement's operability.

The first requirement is unrestricted access to the different experimental locations inside the reactor. For example, open pool type reactors allow relatively trouble free access to different locations outside or inside the core while allowing simple handling of the instrument due to good visibility through water. Within the core, the neutron or gamma radiation fields must be well characterized to facilitate movement and adjustment of the instruments. Such a facility is normally external to the reactor shielding. The second requirement is an ability to operate the reactor in order to follow the experimental needs and not vice versa. This mandatory operational flexibility is often not possible when the reactor must accommodate several different experiments at the same time, for example, material or fuel irradiation as well as medical radioisotope production. Finally, a radiation field monitor is necessary to indicate the actual dose received.

### **12.1.3. Personnel requirements**

Two different categories of staff are required to support testing and calibration applications. The first is responsible for operating the reactor, while the other is responsible for experiments. Concerning the latter, nuclear scientists with good instrumental knowledge are required to perform experiments in precise and safe conditions. Nevertheless, preliminary neutron calculations, and mechanical or thermohydraulic studies could also be necessary for experiment design to support the safety study or for the posttreatment of experimental results.

In addition, a member of the radiological protection staff is required to set up, analyse and calibrate the radiation field recalibrations at routine intervals. Routine work involving positioning and adjusting the instruments can be performed by reactor operational staff.

### **12.1.4. Funding requirements**

The cost of these experiments can vary significantly according to whether new investments are required for qualified monitoring and control. Examples of specific devices to regulate experimental conditions might include pressure and temperature controllers. Additional costs then depend on experiment duration and the support staff required. As a preliminary approximation, capable radiation monitors cost around US \$1000–2000, while experimental facilities can normally be constructed by reactor staff without much additional funding.

### **12.1.5. Other considerations**

If calibrations are being performed, then there is usually the requirement to have the facility certified or accredited by an appropriate national or international organization. This certification entails a significant amount of additional effort for quality assurance, record keeping and similar requirements. In addition, facility accreditation (e.g. the International Organization for Standardization) may cost US \$10 000–20 000 initially, and approximately US \$5000 every few years thereafter for reaccreditation.

## **12.2. NEUTRON IRRADIATION FOR TESTING**

Interaction of fast neutrons with materials leads to atom displacements and microstructure evolution. These evolutions are studied using ageing tests performed in experimental reactors. The results are used primarily to establish a relationship between neutron fluence and the corresponding physical or mechanical properties of materials. Examples of materials testing include:

- Metals such as power reactor pressure vessel steel to determine the service lifetime because of property changes (e.g. embrittlement, stress corrosion and ageing);

- Electronic components;
- High temperature reactor materials such as graphite and silicon carbide;
- Materials for use with a spallation neutron source such as window materials;
- Fusion reactor materials such as blanket testing, first wall materials, plasma facing materials, windows for microwave heating systems and breeder blanket materials.

### 12.2.1. Flux/power level requirements

As nuclear material ageing is mainly caused by fast neutrons, such experiments are preferentially performed inside the reactor core. The flux required for these experiments is typically greater than  $10^{12} \text{ cm}^{-2} \cdot \text{s}^{-1}$ . The fast fluence required for observable effects ranges from  $10^{17}$  to slightly more than  $10^{21} \text{ cm}^{-2}$ . The lower limit is typically applicable to some electronic components and organic materials (e.g. rubber gaskets), while the upper limit is required more for the suitable evaluation of metal properties under irradiation.

### 12.3. LOOPS FOR TESTING NUCLEAR FUELS

For fuel irradiation experiments, as opposed to structural material irradiations, the thermal neutron flux is more important than the fast flux, since thermal neutrons are much more efficient for inducing fission.

Many medium and high power research reactors operate closed loops such as those shown in Figs 12.2 and 12.3 for testing prototype fuel elements for high power research and power reactors. These loops are designed to isolate the test specimen from the reactor and to provide a controlled environment for the specimen. These tests are usually performed in government owned reactors at sites that are part of national laboratory facilities.

Two different types of irradiation test are generally performed. One is used to age fuel samples — for example, to study burnup increases — and can be performed using steady state power conditions. The other is used to test fuel and cladding behaviour under power transient conditions, which requires a specific device to direct variations of the neutron flux received by the fuel sample during the test.

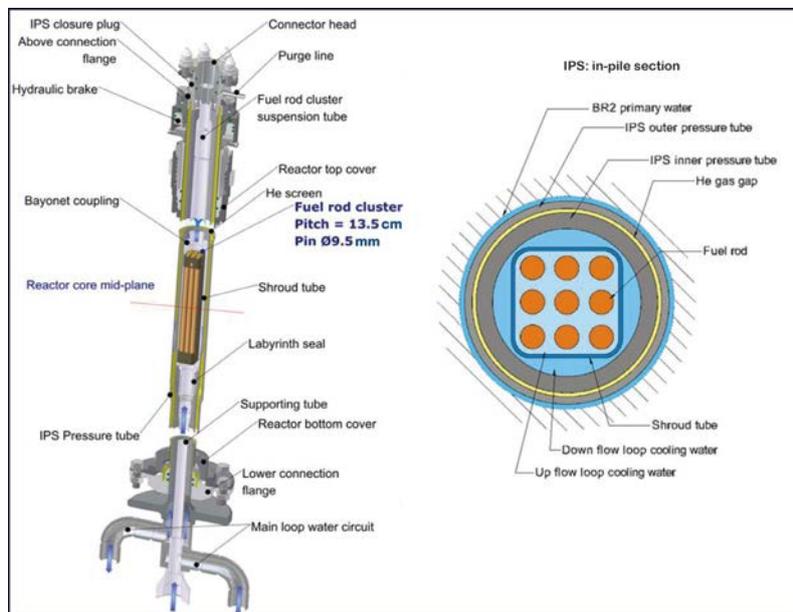


FIG. 12.2. CALLISTO pressurized water reactor steady state irradiation loop at the BR2 (adapted from an image from the Belgian Nuclear Research Centre).



FIG. 12.3. Hot laboratory for material post-irradiation examination at the Japan Materials Testing Reactor (courtesy of the Japan Atomic Energy Agency).

### 12.3.1. Flux/power level requirements

The sample holder is designed to receive a neutron spectrum that is representative of the one seen by the fuel during its industrial use (i.e. the loop reproduces industrial conditions). Materials, screens and distance adjustments can be used for that purpose. The minimum thermal flux required at the location of the specimen is typically in the range of  $(1-50) \times 10^{13} \text{ cm}^{-2} \cdot \text{s}^{-1}$ . Power transient test loops are often located outside the core housing.

### 12.3.2. Reactor facility requirements

The reactor facility requirements typically include:

- Monitoring and control equipment for temperature, pressure and water flow;
- A cladding failure detection system for activity detection in the loop coolant and fission products analysis (e.g. gamma spectrometry);
- Monitoring equipment for power produced by sample fuels, neutron flux or fluence.

In addition to these minimum requirements, the loop could also include specific devices to perform power transients or loss of coolant accident tests. The monitoring and control portions of the loop external to the reactor could comprise equipment such as pumps, heat exchangers, pressurizers, heaters and coolers, together with their control systems and shielding. These components may require a large area, necessitating special space provisions in the reactor facility. In addition, post-irradiation examination facilities with hot cells, associated equipment for examination, and provisions for transferring irradiated fuel between the reactor and hot cell are required if the loop is to be utilized meaningfully.

### 12.3.3. Experimental equipment requirements

In situ characterization techniques have been developed to monitor fuel sample behaviour during irradiation tests. For example, fuel rod released gas pressure and composition were measured during the REMORA 3 irradiation experiment led by the French Alternative Energies and Atomic Energy Commission in 2011. Nevertheless, these measurements must still be supplemented by other complementary techniques, which must be performed before irradiation for non-destructive techniques or after irradiation for non-destructive and destructive techniques.



FIG. 12.4. Example of a neutron radiographic picture of a fuel rod performed in the OSIRIS reactor (courtesy of the French Alternative Energies and Atomic Energy Commission).

#### 12.3.3.1. Non-destructive test facilities

Neutron radiography produces images of the fuel rod showing details of the fuel column inside the cladding, such as dimensions, spaces between each fuel pellet, fracture paths of each fuel pellet and eventual water ingress inside the cladding. One result of this procedure can be seen in Fig. 12.4. Gamma spectrometry enables the evaluation of radionuclide distributions along the fuel column. On the other hand, this technique allows the evaluation of the total nuclear power delivered by each pellet of the column during irradiation. Eddy current characterizations can identify eventual flaws inside the fuel cladding. Dimensional measuring equipment is also a key component of these facilities.

#### 12.3.3.2. Destructive testing equipment

Gas and fuel radiochemical analyses (e.g. inductively coupled plasma techniques) and metallographic studies of the cladding evolution after the experiment (e.g. electron probe microanalyses) also require specialized equipment.

#### 12.3.4. Personnel requirements

Engineers and scientists are required for the design, safety review and installation of the loop. In addition to the personnel normally required for reactor operation, dedicated staff are required for operation of the loop facility.

#### 12.3.5. Funding requirements

The range and subsequent cost of testing equipment mentioned above is directly related to the detail of the parameters required by the relevant post-irradiation examination programme. In addition, data processing, storage and retrieval hardware and software are required to establish a representative database.

An initial investment of US \$1–3 million is typically required for a new loop facility dedicated for fuel testing. In addition, facility maintenance throughout the life of the loop must be guaranteed, and additional fees for support facilities such as radioactive waste treatment and disposal centres must be taken into account. Note that all the different instruments used to characterize nuclear fuels, under water or inside hot cells, can also be very expensive to install, adapt and maintain.

#### 12.3.6. Time requirements

Based on the above and the selective complexity of the in-core testing rigs and control equipment and instrumentation, the development time for these applications can involve several years of design, manufacture, testing, commissioning and evaluation before a satisfactory level of reliability of data collection is achieved.

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## 13. OTHER APPLICATIONS

Although this publication has detailed many applications of research reactors, more (those that are highly specific) can certainly be mentioned. For example, research reactors have provided vast uses in the provision of nuclear data through utilization of their inherent capabilities for cross-section measurements, integral experiments, benchmarking and code validation analyses. Other fields in which nuclear data are needed relate to:

- Testing of materials required for innovative facilities;
- Evaluation of radioisotope production and their medical applications;
- Simulation using computer software of radiation doses to patients and advanced cancer therapies;
- Studies on transmutation of nuclear waste for safer disposal;
- Improvement of analytical techniques adopted for cultural diagnostics and material composition analysis.

In particular, assessments of safety margins and improvement of economic efficiency in the development and licensing of future nuclear power plants and innovative fuel cycles have been instrumental in the advancement of nuclear power plant operation, maintenance and system development. Research reactors continue to occupy a visibly important place in these areas of study and application, along with dedicated accelerator based neutron sources. Some cross-section measurements for very short lived and on-line produced radioactive targets nuclei are possible only at research reactor facilities, given the high neutron fluxes available at some reactors.

Another related application of research reactors is shielding experiments, mainly for neutron attenuation problems. The experiments are carried out to validate shielding calculations with actual measurements, and are particularly useful for advanced shielding designs for new research or power reactors. These experiments are typically conducted using actual shielding material of varying thickness, composition and geometry. A measurement of both incident neutrons and gamma dose is carried out on either side of a shielding material by placing different foils to capture reaction rates for different energy spectra. This is particularly effective with movable core reactors, which have a designated facility for these experiments. The incident neutron flux can also be increased by displacing water by air containers between the core and experimental facility.

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## 14. USER AND CUSTOMER RELATIONS

The operation of a research reactor facility is normally only relevant if stakeholders (those who have a direct interest in the operation of the facility) or customers (usually people that are willing to pay for the research reactor services) justify the reason for the existence of the reactor. In addition to the discussions in the relevant sections of this publication, a few generic principles regarding customer relations are provided below.

Whereas stakeholders are generally classified as those who have control over the day to day operation of the research reactor due to their managerial or financial positions, paying customers can become the livelihood for those reactors where operational and maintenance expenditures are not sufficiently covered by the financial disposition of the direct stakeholders.

A priority of reactor managers in the implementation of a strategic plan for their facility is thus to identify very clearly the potential customers that will utilize or benefit from the facility. A more concise explanation is given in the IAEA publications on milestone determination for a new reactor and strategic planning for research reactors in general [14.1, 14.2].

In the case where a significant investment in equipment is required, it is essential that a technoeconomic feasibility study be completed by the facility to evaluate the balance of capital and operational expenditure versus income generation by provision of the applicable service.

Customers may originate from industries such as mining or agriculture, trade companies, government agencies, medical centres, universities and research institutions — each with specific requirements with respect to their needs. These may include turnaround time (time elapsed between providing the sample and reporting), typical number of samples offered, ease of analysis (element or interference levels) and willingness to pay. As examples, industry usually has a requirement for short turnaround times; governmental agencies may generate a continuous flow of samples, but they may argue that analyses need to be carried out for free because the nuclear centre exists due to governmental support.

Customers from scientific organizations may have insufficient resources to pay for the services. The objectives of such research may be of such high public relevance or lead to extensive reactor utilization that they overcome the need for generating income.

The analysis of market demand and associated market prices if applicable, whether local or international, is crucial, and the subsequent establishment of a potential user and an identifiable customer base is required as an input to the strategic planning exercise. It is indispensable that good customer relations be established and maintained during the life of the services being offered to ensure sustainability of income from such sources on a 'satisfied customer' basis.

An immediate implication of such requirements is that the research reactor facility must implement a suitable integrated management system. This could typically incorporate requirements for quality assurance, safety, security, health and environment, as well as other aspects such as system maintenance, finance and any topics relevant for good reactor management. In most cases, a suitable level of conformance to the selected stakeholder or customer requirements will necessitate certification to acceptable standards (e.g. ISO 9001:2008 and ISO 14000).

For example, the production of radioisotopes for medical and industrial applications, as well as doped silicon for semiconductors, are very attractive sources of income, provided that the market can be secured by a good quality and reliable service at a competitive cost. In most cases, this service will only be able to be established on a regular basis once the facility has demonstrated such a track record.

The needs of the customer and their ability to handle the resultant experimental equipment or products or services provided must be clearly understood by both the research reactor facility and the customer. The level of assistance can then be readily determined and negotiated (e.g. extra technical personnel, shipping and transport assistance, and radiological and regulatory equipment and requirements).

In addition to the typical selection of scientific and technological researchers and industrial application interests, other major customers (non-paying) are the general public, scholars and students. The approach to awareness and training was discussed in Section 2 of this publication, and detailed descriptions of customer requirements were included in Sections 3, 7 and 11. These include typical generic considerations that could also be applicable to the customer relation situations for all applications discussed throughout this publication.

## REFERENCES TO SECTION 14

- [14.1] INTERNATIONAL ATOMIC ENERGY AGENCY, Strategic Planning for Research Reactors, IAEA-TECDOC-1212, IAEA, Vienna (2001).
- [14.2] INTERNATIONAL ATOMIC ENERGY AGENCY, Specific Considerations and Milestones for a Research Reactor Project, IAEA Nuclear Energy Series No. NP-T-5.1, IAEA, Vienna (2012).

## 15. STRATEGIC CONSIDERATIONS

The earlier version of this publication presented descriptions of typical forms of utilization of research reactors. The necessary criteria to enable an application to be performed were outlined for each application, and, in many cases, the minimum as well as the desirable requirements were given. This revised publication has maintained the original purpose, but has specifically taken into account the changes in product and service requirements demanded by relevant stakeholders. In particular, the significant improvements in equipment and technology available for such applications have been taken into consideration, incorporating supportive cost values and time required for development as tentative guidelines.

This publication is of particular benefit to those reactor operators seeking to increase the utilization of their facilities and to assist with the strategic planning required prior to the installation of new equipment or modification of an existing facility or even for the construction of a new research reactor [15.1, 15.2]. This is particularly relevant where the owners and operators of these facilities must demonstrate either the financial or the strategic value of their facilities to the relevant stakeholders.

A further purpose of this publication is to assist owners and operators of research reactors in recognizing the requirement for long term sustainability, including the strategic marketing of their facilities. An important initial stage in preparing such a plan is to evaluate the current and potential capabilities of the facility. Such an evaluation provides some factual and advisory information on several peaceful applications of research reactors. Typical application capabilities are specifically addressed throughout this publication. This initial evaluation must be correlated with the ever important identification of stakeholder and customer needs, as outlined in the referenced strategic planning document [15.1]. Using the present publication, each facility owner and operator should now be able to initiate an assessment as to whether or not a new or modified application is feasible at their reactor and to what extent the relevant capability in that application might require further development and accompanying financial support. In particular, the ever increasing need to produce services and products according to satisfactory levels of quality and regulatory requirements, among others, accentuates the need to implement a suitable integrated management system that can be certified according to the needs of respective stakeholders and customers [15.3].

The applications that are presented vary from those that are possible at any power level of research reactor, such as training, to those that require higher power and more specialized reactors with expensive experimental facilities, such as transmutation doping, neutron imaging and scattering, and neutron capture therapy. The simplified research reactor capability matrix has been updated accordingly and now also includes guidelines regarding development time and personnel and investment costing requirements (see Annex I). This once again assists in the determination of the various applications that may be appropriate for a particular power level reactor.

In summary, the basic principles identified in the exercise of strategic planning and ultimate evaluation of the utilization potential of the facility involve the clarification of the facility abilities (including personnel expertise) matched with stakeholder needs based on an ability to rectify any shortcomings to meet such needs, where feasible, according to the following considerations:

- (a) Maintain a suitable long term vision, typically over a 40 year lifetime, for the utilization of the facility.
- (b) Identify the stakeholder and user needs for efficient utilization of the facility.
- (c) Balance carefully, in advance, the limitations imposed on the establishment of the facility and its experimental set-up and ensure flexibility to overcome challenges relating to:
  - (i) Financial obligations and restrictions that may arise;
  - (ii) Regulatory requirements that become more stringent as time develops.
- (d) New versus existing abilities and equipment.
- (e) Commercial availability of new or replacement equipment.
- (f) Cost requirements and various forms of stakeholder or customer funding available.
- (g) Worker skills and abilities and development and training needs.
- (h) Time required to establish the equipment for experimental or commercial applications, taking into account aspects such as design, manufacture, testing, commissioning and implementation, particularly in view of regulatory requirements.

## REFERENCES TO SECTION 15

- [15.1] INTERNATIONAL ATOMIC ENERGY AGENCY, Strategic Planning for Research Reactors, IAEA-TECDOC-1212, IAEA, Vienna (2001).
- [15.2] INTERNATIONAL ATOMIC ENERGY AGENCY, Specific Considerations and Milestones for a Research Reactor Project, IAEA Nuclear Energy Series No. NP-T-5.1, IAEA, Vienna (2012).
- [15.3] INTERNATIONAL ATOMIC ENERGY AGENCY, Implementation of a Management System for Operating Organizations of Research Reactors, Safety Reports Series No. 75, IAEA, Vienna (2013).

Annex I

RESEARCH REACTOR UTILIZATION MATRIX

TABLE I-1. A RESEARCH REACTOR UTILIZATION MATRIX

Power level	Education and training	NAA	PGNAA <sup>a</sup>	Isotope production	Geochronology			Transmutation effects			Neutron imaging <sup>a</sup>	Neutron scattering <sup>a</sup>	Positron source <sup>a</sup>	BNCT <sup>a</sup>	I&C	Materials <sup>c</sup>	Testing	Fuels <sup>c</sup>
					At-40/At-39	Fission track <sup>b</sup>	Silicon doping <sup>c</sup>	Gamma irradiation	Transmutation effects	Gemstone colouring <sup>c</sup>								
<1 kW	F	S												S				
100 kW	F	F	S	S	S	S	S	S					F	S				
1 MW	F	F	S	S	S	S	S	S	S	S	S	S	F	F	S			
10 MW	F	F	F	F	F	F	F	F	F	F	F	F	F	F	F	F	S	
>10 MW	F	F	F	F	F	F	F	F	F	F	F	F	F	F	F	F	F	
Time required (years) <sup>d</sup>	0.5-1	2-3	2-3	0.5-5	0.5	0.5	3-5	0.5	2	2-4	4-10	5-10	5	0.5	3-5	3-10		
Investment costs (US \$,000) <sup>e</sup>	5-80	150-300	200-600	50-5000	10	10	200-1000	10	100-500	≥500	>2000 <sup>g</sup>	500-700	2000-5000	1-20	>2000	>5000		
Staff required <sup>f</sup>	1-3	2	2	2-20	1	1	2-6	1	1-2	2-3	2-4 <sup>g</sup>	2-3	2-3 <sup>h</sup>	1	2-10	5-20		

**Note:** NAA — neutron activation analysis; PGNAA — prompt gamma neutron activation analysis; BNCT — boron neutron capture therapy; I&C — instrumentation and control; S — some capability (e.g. R&D or demonstration capability); F — full capability (e.g. capable in commercial production).

<sup>a</sup> Requires a beam tube.

<sup>b</sup> Requires fully thermalized neutrons.

<sup>c</sup> Requires a loop or special irradiation facility.

<sup>d</sup> Time required for completion and implementation.

<sup>e</sup> Investment costs for completion and implementation.

<sup>f</sup> Staff required for operation (in addition to reactor operation team).

<sup>g</sup> Estimates provided per one modern neutron scattering instrument.

<sup>h</sup> Estimates provided assuming that additional staff are provided from the hospital.

## Annex II

### TYPICAL EXAMPLES OF RADIOISOTOPES PRODUCED AT RESEARCH REACTORS

#### II-1. MEDICAL APPLICATIONS

- (a) Diagnostic applications:
  - (i) Technetium-99m from molybdenum-99 for oncology and cardiology;
  - (ii) Xenon-133 for lung function.
- (b) Therapy applications:
  - (i) Iridium-192 for brachytherapy;
  - (ii) Iodine-125 for brachytherapy and prostate cancer;
  - (iii) Iodine-131 for thyroid cancer;
  - (iv) Erbium-169 for synoviorthesis, the injection of radiocolloids into rheumatoid digital joints;
  - (v) Palladium-103 for brachytherapy or cobalt-60 for teletherapy;
  - (vi) Lutetium-177 for solid tumours;
  - (vii) Rhenium-188 from tungsten-188 for cardiology.
- (c) Palliation applications:
  - (i) Rhenium-186, samarium-153, strontium-89 and yttrium-90 for use in palliation of metastatic bone pain.

#### II-2. INDUSTRIAL APPLICATIONS

- Iridium-192 is utilized during radiography of welds;
- Cobalt-60 is used for sterilization (gamma irradiators);
- Argon-41, bromine-82 and mercury-203 are used in chemistry applications;
- Caesium-137 and cobalt-60 are used for process control measurements and gauging;
- Cobalt-60 is also used in materials sciences (e.g. for radiation modifications of materials).

#### II-3. OTHER APPLICATIONS

- Various radioisotopes for the calibration of instruments and detectors.

### Annex III

#### NEUTRON IMAGING INSTALLATIONS WORLDWIDE

A summary of known installations, without any guarantee of their current state of readiness, is given in Table III-1. All systems are unique in their construction and differ with respect to their performance. The list also includes some known installations that are under development and are foreseen to be equipped with advanced digital detection systems.

TABLE III-1. SUMMARY OF NEUTRON IMAGING FACILITIES IN THE WORLD

State	Location	Institution	Facility	Neutron source	Neutron beam flux (cm <sup>-2</sup> ·s <sup>-1</sup> )	L/D ratio <sup>a</sup>	Field of view
Australia	Lucas Heights	ANSTO	Dingo	OPAL 20 MW	~6 × 10 <sup>7b</sup>	500–1000	25 cm diameter
Austria	Vienna	Atominstitut	Imaging beam line	TRIGA Mark II 250 kW	1.0 × 10 <sup>5</sup>	125	9 cm diameter
Brazil	São Paulo	IPEN	Imaging beam line	IEA-R1 5 MW	1.0 × 10 <sup>6</sup>	110	25 cm diameter
China	Beijing	CIAE	Thermal neutron imaging	CARR 60 MW	~1.0 × 10 <sup>9b</sup>	200–2100	17 cm × 32 cm
China	Beijing	CIAE	Cold neutron imaging	CARR 60 MW	~8 × 10 <sup>7b</sup>	160–1600	30 cm × 30 cm
Germany	Garching	TUM	ANTARES	FRM II 20 MW	9.4 × 10 <sup>7</sup>	400	32 cm diameter
Germany	Garching	TUM	NECTAR	FRM II 20 MW	3.0 × 10 <sup>7</sup>	150	20 cm diameter
Germany	Berlin	HZB	CONRAD	BER-II 10 MW	6.0 × 10 <sup>6</sup>	500	10 cm × 10 cm
Hungary	Budapest	KFKI	Imaging beam line	BRR 10 MW	6.0 × 10 <sup>5</sup>	100	25 cm diameter
Indonesia	Serpong	BATAN	RN-1	G.A. Siwabessy 30 MW	>10 <sup>6</sup>	83	30 cm diameter
Japan	Kumatori	Kyoto University	Imaging beam line	JMTR 5 MW	1.2 × 10 <sup>6</sup>	100	16 cm diameter
Japan	Tokai	JAEA	Imaging beam line	JRR-3M 20 MW	2.6 × 10 <sup>8</sup>	125	25 cm × 30 cm
Republic of Korea	Daejeon	KAERI	Imaging beam line	HANARO 30 MW	1.0 × 10 <sup>7</sup>	190	25 cm × 30 cm
Malaysia	Kajang	Malaysian Nuclear Agency	NuR2	Puspati TRIGA 1 MW	1.04 × 10 <sup>5</sup>	37	8 cm × 15 cm

TABLE III-1. SUMMARY OF NEUTRON IMAGING FACILITIES IN THE WORLD (cont.)

State	Location	Institution	Facility	Neutron source	Neutron beam flux (cm <sup>-2</sup> .s <sup>-1</sup> )	L/D ratio <sup>a</sup>	Field of view
South Africa	Pelindaba	NECSA	SANRAD	SAFARI-1; 20 MW	$1.6 \times 10^6$	150	36 cm diameter
Switzerland	Villigen	Paul Scherrer Institute	NEUTRA	SINQ spallation source	$5.0 \times 10^6$	550	40 cm diameter
Switzerland	Villigen	Paul Scherrer Institute	ICON	SINQ spallation source	$1.0 \times 10^7$	350	15 cm diameter
USA	University Park	Penn State University	Imaging beam line	PSBR TRIGA 2 MW	$2.0 \times 10^6$	100	23 cm diameter
USA	Gaithersburg	NIST	CNR	NBSR 20 MW	$2.0 \times 10^7$	500	25 cm diameter
USA	Davis	McClellan Nuclear Research Centre	Imaging beam line	MNRC TRIGA 2 MW	$2.0 \times 10^7$	100	23 cm diameter

<sup>a</sup> L — source to detector distance; D — aperture diameter.

<sup>b</sup> Calculated flux for instrument not yet in operation.

## ABBREVIATIONS

2D-ACAR	two dimensional angular correlation (annihilation radiation)
ADC	analogue to digital converter
ANSTO	Australian Nuclear Science and Technology Organisation
BATAN	National Nuclear Energy Agency
BNCT	boron neutron capture therapy
CAT	computed axial tomography
CBTRUS	Central Brain Tumor Registry of the United States
CCD	charge coupled device
CIAE	China Institute of Atomic Energy
DNC	delayed neutron counting
DSP	digital signal processor
ENAA	epithermal neutron activation analysis
FWHM	full width at half maximum
GM	Geiger-Müller
HANARO	High-flux Advanced Neutron Application Reactor
HZB	Helmholtz-Zentrum Berlin
I&C	instrumentation and control
IGBT	insulated gate bipolar transistor
INAA	instrumental neutron activation analysis
IPEN	Nuclear and Energy Research Institute
ISO	International Organization for Standardization
JAEA	Japan Atomic Energy Agency
KAERI	Korea Atomic Energy Research Institute
KFKI	Central Research Institute for Physics
MCA	multichannel analyser
MRI	magnetic resonance imaging
NAA	neutron activation analysis
NECSA	South African Nuclear Energy Corporation
NIST	National Institute of Standards and Technology
NTD	neutron transmutation doping
OPAL reactor	Open Pool Australian Lightwater reactor
PC	personal computer
PGNAA	prompt gamma neutron activation analysis
QA/QC	quality assurance and quality control
RNAA	radiochemical neutron activation analysis
RRG	radial resistivity gradient (NTD applications)
SANS	small angle neutron scattering
SPND	self-powered neutron detector
TOF	time-of-flight
TUM	Technische Universität München
U-SANS	Ultra small angle neutron scattering

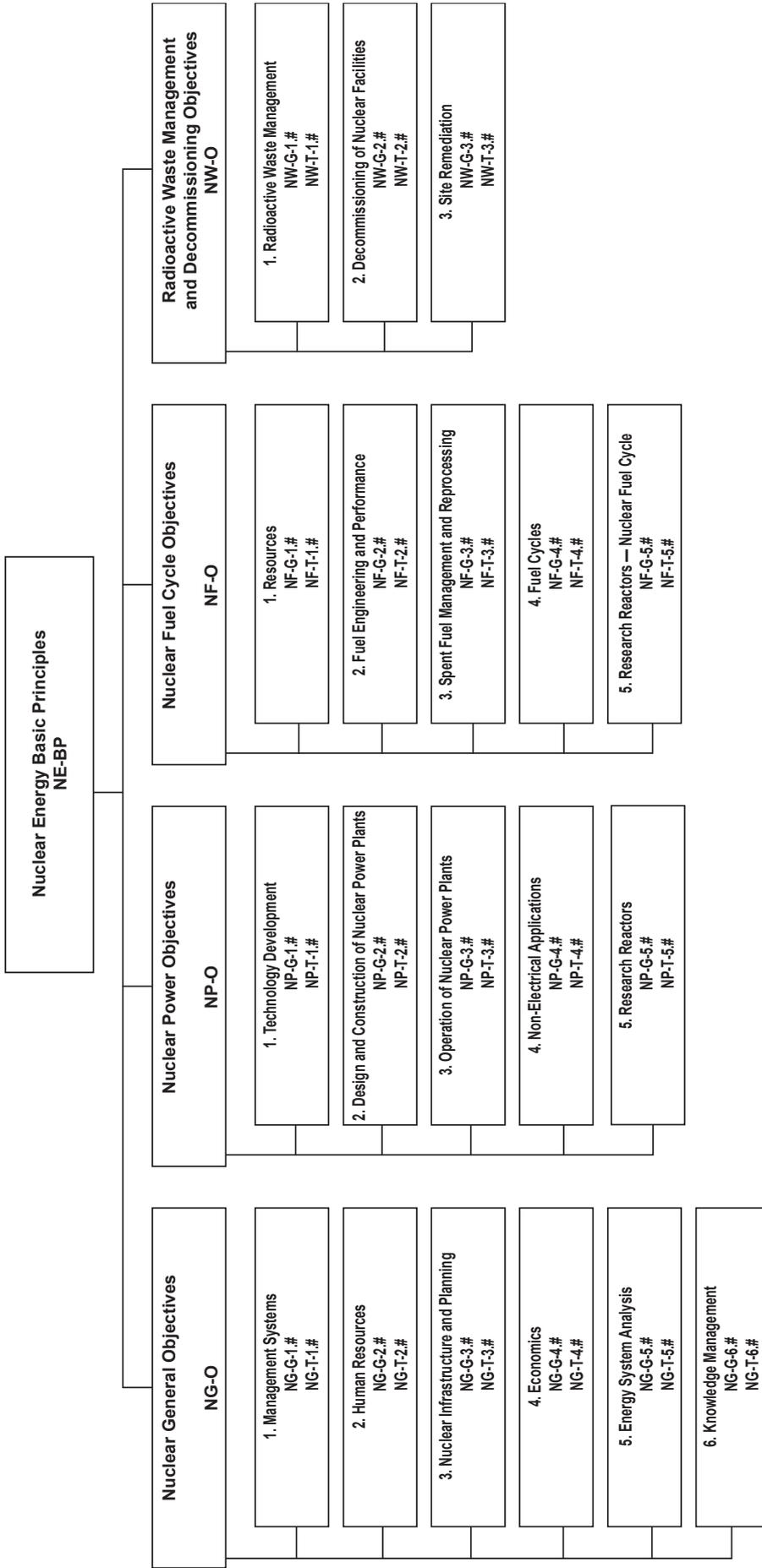
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