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INVITED PAPER

RADIOCHEMICAL DETERMINATION OF NUCLEAR DATA[1]

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A vast knowledge of nuclear data is available and is grouped under three headings, namely, nuclear structure, nuclear decay and nuclear reaction data. Data measurements are done using a large number of techniques, including the radiochemical method, which has been extensively worked out at Juelich. This method is used for preparation of high-quality samples for irradiation, isolation of the desired radioactive product from the strong matrix activity, and preparation of thin suitable sources for accurate measurement of the radioactivity. The radiochemical method is especially suitable for fundamental studies on light complex particle emission reactions and formation of low-lying isomeric states. The neutron induced reaction cross section data are also of practical application in fusion reactor technology, particularly for calculations on tritium breeding, gas production in structural materials and activation of reactor components. The charged particle induced reaction cross section data, on the other hand, are of significance in developing new production routes of novel positron emitters and therapeutic radionuclides at a cyclotron. A brief overview of all those studies is given.

Keywords: Nuclear data, Complex particle emission reactions, Isomeric cross sections, Data for fusion reactor technology, Medical radionuclide production, Novel positron emitters, Therapeutic radionuclides

1. Introduction

The term "nuclear data" is very broad. It includes all data which describe either the characteristic properties of nuclei or their interactions. In general, all those data can be grouped under three headings, namely, nuclear structure, nuclear decay and nuclear reaction data. A large number of techniques are used to determine the data, each of them having its own advantages and limitations. This article deals with the radiochemical technique which, though limited in some ways, is applicable to studies of all three types of data mentioned above. It consists of irradiating a material with neutrons or charged particles, separating the activated (radioactive) product, and measuring its decay characteristics or absolute radioactivity by standard counting techniques. Thus in contrast to purely physical methods, which aim at on-line measurements of the emitted radiation spectra, the activation (or radiochemical, when combined with a chemical separation) technique deals with the residual radioactive nucleus, i.e. off-line detection and measurement of the activated reaction product. The technique has very high sensitivity, especially in case of short-lived radioactive products. In case of stable or very long-lived radioactive products, mass spectrometry (MS) and accelerator mass spectrometry (AMS) are more useful. The radiochemically obtained data are generally application oriented; but some information can be deduced on the reaction mechanism as well. We consider briefly some of the relevant aspects below.

2. Nuclear Structure and Nuclear Decay Data

In general, nuclear decay data are well known [1, 2]. Since many of the decaying nuclei have decay energies of up to about 3 MeV, or somewhat higher, the nuclear levels populated in the decay product are mostly well characterised upto excitation energies of 2 to 3 MeV. More detailed

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information on the nuclear structure, i.e. all the discrete levels upto the continuum of the nucleus, and even beyond, is obtained via spectral studies on nuclear processes. A good example is the in-beam γ-ray spectroscopy following an (n,γ) reaction which can provide information on the level structure of the product nucleus upto about 8 MeV. Those studies are, however, beyond the scope of this article. The discussion here is limited to decay properties of radioactive nuclei and the levels of the daughter fed by the various transitions.

Despite the extensive information available, there are still some areas where more nuclear data work is necessary and where radiochemistry can play an important role. One such area is the study of radionuclides rather far from the stability line. Another area is the search and characterisation of super heavy elements. In both cases fast radiochemical separations are mandatory to isolate the desired product from the much stronger matrix activity, and to characterise it via measurement of radioactivity. In the former case mass separators and in the latter multi-parameter physics experiments are the major competitors to the radiochemical technique.

The radioactive decay data find applications in many areas, e.g. calculation of total activity, heat generation, transmutation products, etc. However, in recent years, with the enhancing application of radioactivity in medicine, especially for in-vivo diagnostic and therapeutic studies, the demands on accurate decay data have considerably increased. A higher accuracy in the data means a higher accuracy in the internal radiation dose calculation. Two areas appear to need special attention – one involves the energies and intensities of low-energy electrons (e.g. conversion or Auger electrons) and the other branching ratios in the decay of positron emitters. Since decay schemes of many of the medically interesting radionuclides were generally determined using mixtures of radionuclides, often utilising rather poor resolution counters, it appears worthwhile to reinvestigate some of the special radionuclides in more detail using radiochemical techniques. Today, many of the medical radionuclides can be produced with very high purities; the use of those ultrapure sources should thus provide accurate information on the decay data as well.

An example of a recent decay data measurement is provided by ^{64}Cu. This radionuclide is becoming increasingly important in positron emission tomography (PET) in connection with radioimmunotherapy. It was produced via the ^{64}Ni(p,n)^{64}Cu reaction on highly enriched ^{64}Ni [3] as well as via the ^{66}Zn(d,α) ^{64}Cu reaction on highly enriched ^{66}Zn [4]. The chemical separation was based on ion-exchange chromatography [3,4] and the final product was obtained as a very thin source with a radionuclidic purity of > 99.9 %. The measurements included ß counting, $\gamma\gamma$-coincidence counting, conventional high-resolution γ-ray spectrometry and, above all, high-resolution X-ray spectrometry using a Si(Li) detector. The latter was absolutely necessary to determine the low-energy (7.47 and 8.26 keV) K_α and $K_ß$ X-rays of the daughter Ni which describe the electron capture (EC) component in the decay of ^{64}Cu. The results are depicted in Fig. 1 [5]. It should be mentioned that the $ß^-$ branching was already well characterised, also through a mass-spectrometric analysis of the daughter ^{64}Zn [6]. There was some discrepancy in the $ß^+$ branching and consequently in the intensity of EC decay; they are now fairly well established. The intensity of the weak 1346 keV γ-ray was also rather doubtful; it has been now measured precisely. Thus the availability of ^{64}Cu of high radionuclidic purity made it possible to determine the decay scheme of that radionuclide with higher accuracy.

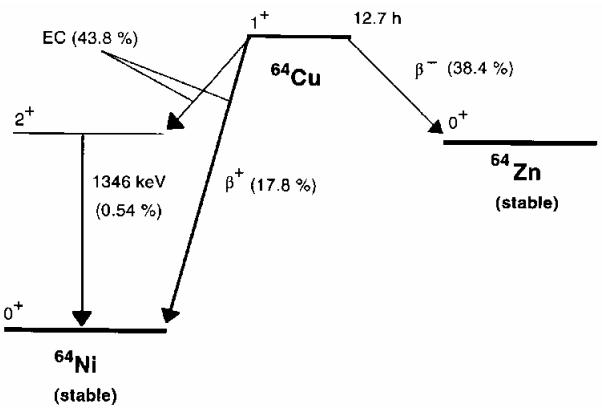

Figure 1. Decay scheme of ^{64}Cu [5].

Similar to ^{64}Cu, the positron emission intensities in the decay of the radionuclides ^{76}Br, ^{120}I and ^{124}I have also been accurately determined [5,7,8]. Again it was possible only through the availability of each of those radionuclides in a highly pure form.

3. Nuclear Reaction Data

In contrast to nuclear structure and radioactive decay data, whose scope is generally limited up to excitation energies of about 10 MeV, the nuclear reaction data cover a very broad span of energies, extending from a few meV upto the region of several GeV. The lower side of the energy scale is typical of neutrons and encompasses cold, thermal and epithermal regions. The major applications of nuclear data in those regions are related to structural analysis of solids, quantitative determination of elements via activation analysis, and fission reactor technology. The neutron capture cross sections and fission yields are also useful for production of radionuclides, especially for medical applications. The energy region from about 10 keV to a few MeV can be reached both by neutrons and charged particles, and it is particularly interesting for astrophysics and fusion research, especially with respect to the interactions of light charged particles. Neutrons upto 20 MeV energy have been extensively utilised in the development work related to fast reactors and fusion technology. With increasing energies, monoenergetic neutrons become rarer, so that work above 30 MeV is done mostly using charged particles.

The body of available nuclear reaction data is huge but well organised. Experimental data published in any part of the world are compiled within a few months in the EXFOR file, coordinated by the IAEA. The data evaluation, making extensive use of the most modern nuclear model codes, is performed in many parts of the world and, after validation and quality assurance, the data are placed at the disposal of users in extensive evaluated nuclear data files (e.g. ENDF-B VII). In addition some special purpose files like Fission Products, Activation Data, Neutron Dosimetry, Fusion Data, Medical Applications, etc. are also prepared. The data files contain all reliable data, measured by all techniques (including the radiochemical method), and substantiated by nuclear model calculations.

The radiochemical technique of cross section measurement is advantageously used in the following cases:

- Preparation of thin samples for irradiation, especially with charged particles. Some of the common methods include electrolytic deposition, vacuum evaporation, alloy formation etc.
- Study of low-yield reactions, i.e. when the cross section of the reaction under investigation is low and the matrix activity (i.e. the radioactivity of the undesired products) is high.
- Study of soft-radiation emitters. This is the case when the product decays via ß$^-$ emission or electron capture (EC) without any accompanying γ-ray. The radionuclides decaying by EC are characterised by X-ray counting, which can be performed only when a thin source has been prepared.
- Characterisation of low-lying isomeric states. The low energy transitions can be detected advantageously after chemical separation and using a high-resolution low-energy detector.

Extensive use was made of the radiochemical technique in the determination of fission yields and many activation cross sections. With the increasing use of high-resolution solid state detectors in γ-ray spectrometry, the importance of radiochemical measurements has somewhat diminished. Nonetheless, there are still many interesting areas where this technique is almost ideally suited or where it has advantages over the other methods. An extensive programme of work utilising this technique has been underway at Juelich for more than 30 years. A brief description of the areas pursued is given below.

3.1. Complex particle emission reactions

In interactions between nuclei and projectiles mostly nucleons and electromagnetic radiation are emitted. The emission of light complex particles (d, t, ^{3}He, α, ^{7}Li, ^{7}Be, ^{10}Be, etc.) is rather rare. Due to both experimental and calculational difficulties not many studies have been done [9]). This is an area of research demanding extensive use of radiochemical separations. Tritium and ^{3}He emission was studied radiochemically mainly in neutron induced reactions using tritium counting and mass spectrometry. The α-particle emission data were obtained both via mass spectrometry and measurement of the reaction products. For ^{7}Be and ^{10}Be, radiochemical separations in combination with γ-ray spectrometry and accelerator mass spectrometry (AMS), respectively, have been applied. Most of the radiochemical and mass spectrometric studies on emission of d, t, ^{3}He, α and ^{7}Be have been carried out at Juelich [9]. Fig. 2 demonstrates, as an example, the importance of radiochemical separation while investigating the ^{7}Be emission. In

a study of the $^{51}V(p,^{7}Be)^{45}Ca$ reaction a vanadium foil was irradiated with 50 MeV protons and the γ-ray spectrum was registered. Over the γ-ray energy range of 450 to 530 keV, except for the ß$^{+}$ annihilation peak at 511 keV, no γ-ray was observed (curve (A) in Fig. 2). The product ^{45}Ca is a ß$^{-}$ emitter and the γ-ray of the emitted particle ^{7}Be at 477 keV is fully masked by the matrix activity. After an ion-exchange separation the ^{7}Be-containing fraction was again submitted to γ-ray spectrometry (curve (B) in Fig. 2). The background was considerably reduced and the ^{7}Be peak could be easily analysed.

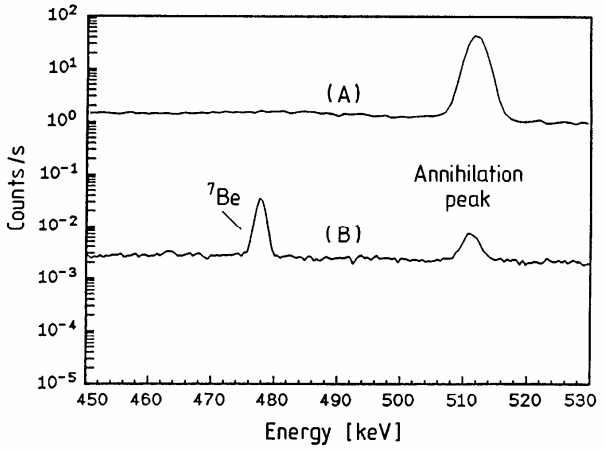

Figure 2 A section of the γ-ray spectrum of (A) vanadium foil irradiated with 50 MeV protons, (B) radio-beryllium chemically separated from the matrix activity.

The experimental work has led to some interesting results. For example, a triton is easily emitted from the light mass nuclei. In medium and heavy mass regions, on the other hand, a bound triton is emitted with a much lower probability than three nucleons; in case of ^{3}He emission, however, this is not so. As far as nuclear model calculations are concerned, there is as yet no satisfactory method of estimating the complex particle emission cross section, the exceptions being triton and α-particle emissions. In the former case the statistical process has been partly successful [10] and, in the latter, a combination of statistical, pre-compound and direct processes has successfully reproduced the experimental data [11, 12]. In other cases, e.g. d and ^{3}He emission, contributions of direct interactions appear to be dominant [13, 14]. For all the investigated reactions development of some systematic trends proved to be very advantageous in describing some characteristics of those reactions. The results for the neutron induced reactions are reproduced in Fig. 3 [cf. 15]. The data for (n,t) reactions were obtained by tritium counting, for (n,^{3}He) reactions by mass spectrometry and for (n,^{7}Be) reactions by γ-ray spectrometric measurement of ^{7}Be. Only in the case of (n,α) reactions residual radioactive nuclei were studied. Since the (n,nα) processes were neglected, the measured cross sections are certainly lower than the α-emission cross sections. The experimental trends for ^{3}H, ^{3}He and ^{7}Be emission follow a definite pattern, i.e. the higher the charge of the emitted complex particle, the lower is its emission probability. The α-particle emission is an exception due to its extremely high binding energy; its probability of emission is comparable to those of nucleons.

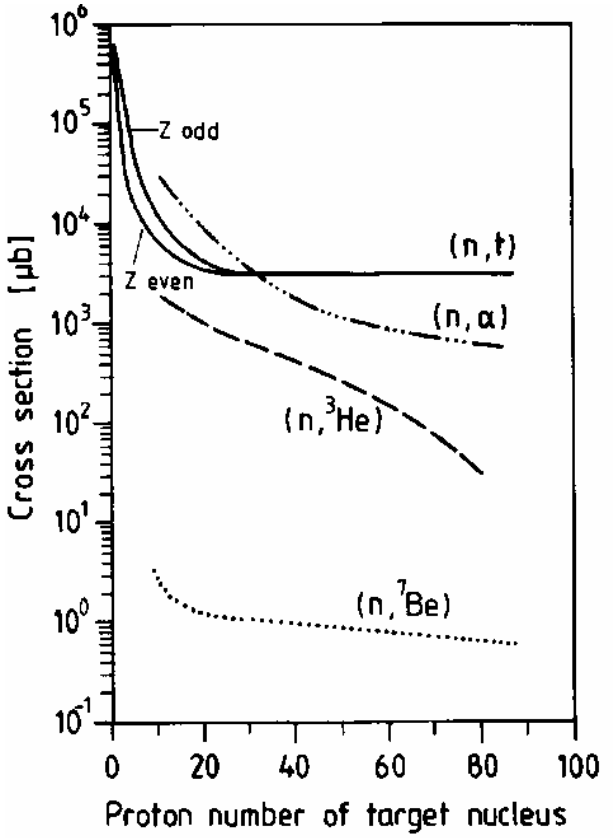

Figure 3 Systematic trends in (n,t), (n,^{3}He), (n,α) and (n,^{7}Be) reaction cross sections induced by 53 MeV d(Be) breakup neutrons [9, 15].

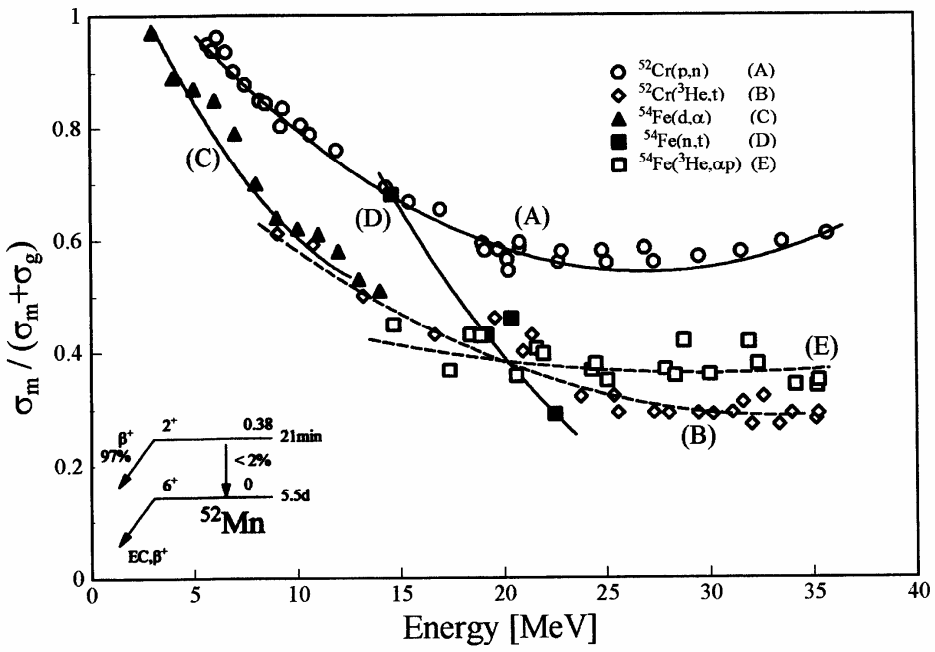

Figure 4 Experimental isomeric cross section ratios for the formation of ^{52m,g}Mn in several nuclear reactions, plotted as a function of the incident particle energy [18].

3.2 Isomeric cross sections

In comparison to the total cross section of a reaction channel, the partial cross section for the population of a particular nuclear level is more difficult to measure and to calculate. As mentioned above, the radiochemical technique is ideally suited for the measurement of isomeric cross sections. Over the last 20 years extensive investigations have been performed on several isomeric pairs, e.g. ^{52m,g}Mn, ^{58m,g}Co, ^{73m,g}Se, ^{94m,g}Tc, ^{120m,g}I, etc., involving different combinations of target, projectile, ejectile, and level structure of the product nucleus. It was found that the isomeric cross section ratio depends on the spins of the two concerned levels of the product nucleus and not on their separation energy [16, 17]. Furthermore, the yield of the high-spin isomer increases with the increasing projectile energy. Nuclear model calculations were successfully performed using the code STAPRE (incorporating statistical and pre-compound models) in combination with a very careful choice of the input parameters.

The experimental results for the formation of a typical isomeric pair ^{52m,g}Mn are given in Fig. 4 [18]. The important role of the spins of the states involved is confirmed. The cross section of the low-spin isomer in comparison to that of the high-spin ground state initially decreases with the increasing incident projectile energy, but becomes almost constant at high excitation energies. The magnitudes of the cross section ratio differ considerably. It is concluded that the reaction channel influences the isomeric cross section ratio appreciably, particularly when the channels differ widely, e.g. (p,n) and (^{3}He,t) processes. Presently more attention is being paid to the formation of high-spin isomers [19, 20].

3.3 Fusion reactor technology

As mentioned above, neutron reaction data from a few MeV upto about 20 MeV are of prime importance in fusion reactor technology. The radiochemically determined data are of significance in estimation of hydrogen and helium gas production in first wall constituents, tritium breeding in blanket materials, and in making an inventory of total radioactivity, i.e. a summation of all activation products, in various components of the reactor system. In 1970s and 1980s considerable attention was devoted to neutronics problems of a fusion system. But in 1990s those activities were considerably reduced. With the upcoming demonstration experiment ITER, those activities are expected to be rejuvenated. For a detailed discussion of radioactivity in fusion energy, especially the contributions made by the Juelich group, the reader is referred to [21].

3.4 Medical radionuclide production

The activities at Juelich have mainly concentrated on charged particle induced reaction

cross section data for production of medical radionuclides at cyclotrons. Here thin sample preparation plays a very special role. The determination of radioactivity, however, could be done in many cases via conventional γ-ray spectrometry (without chemical separation), but occasionally clean radiochemical separations were absolutely necessary to obtain accurate data (e.g. while investigating ß⁻ ray, X-ray and low-energy γ-ray emitters). Considerable efforts were devoted to optimisation and standardisation of data for production of commonly used SPECT radionuclides ^{123}I ($T_{1/2}$ = 13.2 h) [22 – 25] and ^{201}Tl ($T_{1/2}$ = 73.1 h) [26], as well as the PET radionuclides ^{11}C ($T_{1/2}$ = 20.3 min) [27], and ^{18}F ($T_{1/2}$ = 110 min) [28], and ^{82}Sr ($T_{1/2}$ = 25.3 d) which is the parent of the positron emitter ^{82}Rb ($T_{1/2}$ = 1.2 min) [29]. The major activity over more than two decades, however, has been directed to developing novel radionuclides for PET and internal radionuclide therapy.

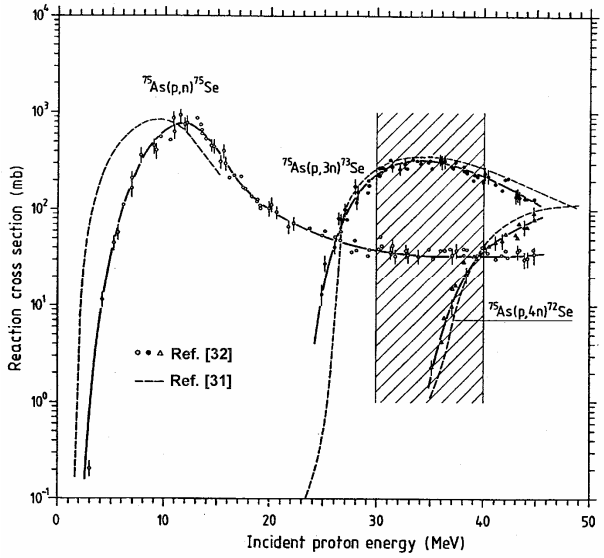

Figure 5 Excitation functions of ^{75}As(p,xn)^{72,73,75}Se reactions as reported in [31, 32]. The shaded area shows the optimum energy range for the production of ^{73}Se.

In development of a novel radionuclide using a cyclotron the reaction cross section data play a very important role [30]. They are needed to optimise the production process, which means a selection of the projectile energy range that will maximise the yield of the product and minimise that of radioactive impurities. Occasionally several nuclear reactions may lead to the same product radionuclide. The choice of a process for production is then made considering the yields of the product and impurities. Finally, to validate the experimental data it is often desirable to perform nuclear model calculations.

Novel PET radionuclides

The list of novel PET radionuclides whose production methods were developed at Juelich includes ^{38}K ($T_{1/2}$ = 7.5 min), ^{51}Mn ($T_{1/2}$ = 46.2 min), ^{55}Co ($T_{1/2}$ = 17.6 h), ^{64}Cu ($T_{1/2}$ = 12.7 h), ^{73}Se ($T_{1/2}$ = 7.1 h), ^{75}Br ($T_{1/2}$ = 1.6 h), ^{76}Br ($T_{1/2}$ = 16.0 h), ^{82m}Rb ($T_{1/2}$ = 6.2 h), ^{83}Sr ($T_{1/2}$ = 32.4 h), ^{86}Y ($T_{1/2}$ = 14.7 h), ^{94m}Tc ($T_{1/2}$ = 53 min), ^{120g}I ($T_{1/2}$ = 1.3 h) and ^{124}I ($T_{1/2}$ = 4.18 d). Among them ^{64}Cu, ^{86}Y and ^{124}I are finding worldwide attention. We discuss a typical case below.

The radionuclide ^{73}Se is a sulphur analogue and is thus of great potential interest. It can be produced via several routes, the reactions ^{75}As (p,3n), ^{75}As(d,4n), ^{72}Ge(^{3}He,2n) and ^{70}Ge(α,n) being most promising. The data for the ^{75}As(p,3n) process [31, 32] are shown in Fig. 5, together with the information on the other competing (p,xn) reactions. For the production of ^{73}Se the energy range E_p = 40 → 30 MeV appears to be most useful: the yield of ^{73}Se is high and the level of the long-lived impurities ^{72,75}Se is < 0.2 %. Similar studies were performed on the other three reactions as well [32, 33]. The results for all the four investigated processes are summarised in Table 1 for comparison. Evidently, the ^{75}As(p,3n)^{73}Se reaction is most suitable for production of ^{73}Se. On the other hand, if an intermediate energy cyclotron is not available, the ^{70}Ge(α,n)^{73}Se reaction can also be utilised for production purposes [34], though with much lower yield. For all the reactions studied, nuclear model calculations were also performed [16]. In general, good agreement was found between experimental data and results of model calculations. This validated and strengthened the data base.

Table 1. Comparison of production routes of ^{73}Se.

Nuclear process	Energy range (MeV)	Thick target yield of ^{73}Se* MBq (mCl)/μAh	% impurity*	
			^{72}Se	^{75}Se
^{75}As(p,3n)	40→30	1406 (38)	0.11	0.05
^{75}As(d,4n)	45→33	651 (17.6)	0.07	0.20
^{nat}Ge(^{3}He,xn)	36→13	37 (1.0)	1.77	0.24
^{nat}Ge(α,xn)	28→13	26 (0.7)	0.49	0.44

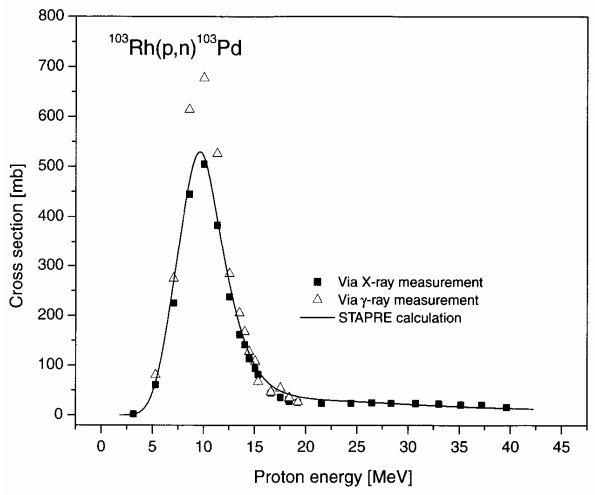

Figure 6 Excitation function of the ^{103}Rh(p,n)^{103}Pd reaction as reported in [36]. The data were obtained via both X-ray and γ-ray counting; more confidence was, however, placed on the X-ray data. The calculation using the code STAPRE is based on the statistical model incorporating precompound effects [36].

Novel therapeutic radionuclides

Therapeutic radionuclides are generally produced in a nuclear reactor since they are mostly ß$^-$ emitters. In recent years the cyclotrons have also been increasingly utilised, especially for production of radionuclides emitting low-energy ß$^-$ particles, Auger electrons, X-rays and α-particles. At Juelich novel production routes were investigated for the therapeutic radionuclides ^{67}Cu ($T_{1/2}$ = 61.9 h), ^{103}Pd ($T_{1/2}$ = 16.96 d), ^{140}Nd ($T_{1/2}$ = 3.37 d) and ^{193m}Pt ($T_{1/2}$ = 4.33 d). The radionuclide ^{67}Cu emits low-energy ß$^-$ particles whereas the other three radionuclides emit Auger (or conversion) electrons and X-rays. We discuss a typical case below.

The radionuclide ^{103}Pd is almost a pure X-ray and Auger electron emitter. It is being increasingly used in treatment of prostate cancer. For its production in no-carrier-added form several reactions have been investigated, e.g. ^{103}Rh(p,n) [35, 36], ^{103}Rh(d,2n) [37], ^{102}Ru(^{3}He,2n) [38], ^{101}Ru(α,2n) [38], ^{nat}Ag(p,x)^{103}Pd [39], ^{nat}Ag(p,x)^{103}Ag → ^{103}Pd [40], etc. However, the ^{103}Rh(p,n)^{103}Pd reaction is most commonly used. The data measured at Juelich are shown in Fig. 6. Some of the data were determined using a γ-ray at 357.5 keV [35, 36]. However, its abundance is only 0.022 %. Other measurements were done via high-resolution X-ray spectrometry using the $K_{α1}$ and $K_{ß1}$ lines at 20.22 and 22.72 keV, respectively. Evidently, the data obtained via conventional γ-ray spectrometry were too high. More reliance was therefore placed on the data obtained via X-ray spectrometry. Detailed nuclear model calculations using the code STAPRE were also performed [36]. An excellent agreement was obtained between experiment and theory over the whole investigated energy range. The data base for production of this radionuclide was thus strengthened. Furthermore, it was shown that if a thin Rh sample irradiated with protons to produce ^{103}Pd is introduced as a seed into the prostate, it is mandatory that the proton energy does not exceed 13 MeV, so that the reaction ^{103}Rh(p,pn)^{102}Rh is not induced and the long-lived activity ^{102}Rh ($T_{1/2}$ = 207 d) is avoided.

4. Conclusions

The radiochemical method of nuclear data determination is well established; it complements on-line physical measurements. The radiochemical technique is advantageously used in investigations on low-yield reaction products, soft-radiation emitters and low-lying isomeric states. Combined with nuclear model calculations, the measured data can also lead to some mechanistic information on the nuclear reaction. The radiochemically measured data are of considerable practical importance, especially in nuclear technology and cyclotron production of medical radionuclides. It should be emphasised that nuclear data research demands interdisciplinary approaches and cooperative efforts. It constitutes an interesting science and useful technology.

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