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Non-Destructive Analysis of Nuclear Fuels by Radiative Neutron Capture

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Non-destructive analysis of nuclear fuels by radiative neutron capture

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Abstract

The main requirements of non-destructive methods for nuclear fuel analysis are adequate accuracy, sufficient penetrability and high isotope discrimination capacity. The present paper investigates the possible use of the radiative neutron capture process for the detection, identification and quantitative analysis of fissile materials. An apparatus that eliminates interfering effects from nuclear fission is proposed. It is shown that recording characteristic primary transitions from neutron capture should fulfill all requirements, provided that neutrons of appropriate energy are used.

Zusammenfassung

Zerstörungsfreie Analyse von Kernbrennstoffen durch Strahlungseinfang von Neutronen

Die wichtigsten Anforderungen, die an zerstörungsfreie Methoden bei der Analyse von Kernbrennstoffen gestellt werden, sind gute Genauigkeit, hinreichende »Transparenz« sowie ein hohes Trennungsvermögen für die ein zelnen Isotope. Die vorliegende Arbeit untersucht die mögliche Ausnutzung des Strahlungseinfangs von Neutronen für den Nachweis, die Identifizierung und die quantitative Bestimmung spaltbarer Materialien. Es wird eine Versuchsanordnung vorgeschlagen, die störende Effekte durch die Kernspaltung ausschließt. Dabei zeigt sich, daß die Registrierung charakteristischer Primärübergänge vom Neutroneneinfang alle gestellten Anforderungen erfüllen sollte, wenn Neutronen geeigneter Energie verwendet werden.

EURATOM KEYWORDS

NONDESTRUCTIVE TESTING FUELS RADIATIVE CAPTURE FAST NEUTRONS GAMMA RADIATION GAMMA DETECTION SCINTILLATION COUNTERS

1. Introduction

In order to prevent nuclear fuels from being used for other than peaceful purposes, all peaceful applications of nuclear energy have been brought under the present systems of nuclear control and security. The regulations lay down that the operator of nuclear installations is required to furnish the controlling authority (IAEA or Euratom) with the basic technical characteristics of the installations, and that the Authority's inspectors are entitled to enter individual installations at any time. This type of supervision was developed at a time when there were only relatively small quantities of fissile material-mainly for scientific purposes-in use in the different countries. The situation has however been fundamentally altered by the increasing commercial exploitation of nuclear energy. Fissile material now passes through several nuclear installations during fuel production and reprocessing, the quantities used are considerably larger, and under conditions of free competition commercial interests assume greater importance. It is therefore desirable that an alternative system of control based on instrumentation be developed, which, by supervision of the flow of fissile material at selected strategic points, will provide adequate security against the danger of misuse of nuclear fuels [1]. An important problem inherent in such a control is the non-destructive analysis of non-irradiated fuel elements either at the output of the production plant or at the input of the reactors.

The requirements to be satisfied in such non-destructive analysis are very stringent: the methods adopted must be tamperproof, permit sufficient accuracy of measurement, be immune from systematic errors and provide as much detailed information as possible about the isotopic composition of the fuel.

On account of their short range, detection of alpha and beta particles from the natural radioactive decay of heavy nuclei is basically impracticable in a non-destructive procedure. Neither does the gamma radiation from electromagnetic transitions in the daughter nuclei in general ensure sufficient "transparency", since the energy of these gamma quanta is too low. For example, the half-thickness for the 184 keV radiation from the decay of ²³⁵U is only 0,23 mm in uranium metal. At best, the gamma groups of very low intensity ($\approx 10^{-5}$ per disintigrating parent nucleus) from 239 Pu at about 376 and 414 keV reveal half-thicknesses at 1,0 and 1,2 mm, respectively. Hence in general, procedures based on this method-such as the determination of the enrichment of uranium [2] or of the ²³⁹Pu content [3]-only reach the outermost layer of the fuel element, and are therefore only suited for the routine quality control carried out by the operator of a production plant, but certainly not for a nuclear control system.

In recent years consideration has been given to other possible methods of overcoming these difficulties. If, for instance, a fuel element containing ²³⁵U is irradiated with neutrons, part of these will induce fission processes in ²³⁵U releasing on the average 2,46 fast neutrons per event. If these fission neutrons are detected, the number of fissions that have taken place and hence the ²³⁵U content can be measured [4]. Under certain conditions, transmission measurements with X-rays enable the total uranium content (including ²³⁸U) to be determined [4]. The induced fission method is also applicable in principle to ²³⁹Pu and other fissile isotopes. If however there are several fissile nuclides in the fuel element, this method on its own does not yield a definitive result. To summarize, it can be said that none of the procedures at present known satisfies all the conditions specified.

Recently the author and colleagues [5] suggested certain new methods that make use of gamma-ray emission induced by neutrons or photons. In the present article, the applicability of the particular case of radiative neutron capture will be examined in more detail.

2. Principle of analysis by (n, γ) reactions

If the sample under test is irradiated by thermal or near thermal neutrons, then a fraction of these neutrons, depending upon the individual cross-sections of the nuclei present in the sample, will be captured. The compound nuclei produced are excited with the respective binding energies which lie between 4 and 11 MeV. De-excitation occurs by emission of prompt characteristic gamma rays, either by a direct transition to the ground state or by two-and multiple-step cascades. This de-excitation takes place preferably as a result of electric dipole radiation. Since the level density at low excitation energies of the nuclei is small, and as only a few of the states satisfy the parity and angular momentum selec-

KERNTECHNISCHE MESSVERFAHREN · NUCLEAR MEASUREMENTS



Fig. 1: High-energy gamma-ray spectrum from neutron capture in a nickel sample. Energies in $\ensuremath{\mathsf{keV}}$

Fig. 1: Hochenergetisches Neutroneneinfangspektrum einer Nickelprobe. Energien in keV

tion rules for electric dipole radiation from the capture state, the (n, γ) spectrum has a very simple structure in the higher energy region, although with modern equipment 100 or more gamma transitions per nuclide can be detected in the total spectrum. If a mixture of isotopes is present, it is therefore convenient to use the higher energy range for purposes of analysis. The separate isotopes are thus identified by the energy of the observed transitions and the line intensity is a measure of the number of nuclei of the particular nuclides present in the sample. Analysis of high-energy gamma radiation by means of semi-conductor counters presents no difficulty. Lithium-drifted germanium diodes are superior to all other spectrometers in respect of both energy resolution and detection efficiency. Fig. 1 illustrates the situation using neutron capture in a sample of nickel (1,62% ⁵⁸Ni, 5,18% ⁶⁰Ni, 92,11% 61Ni, and 1,08% 62Ni) as an example.

The method outlined here is applicable to nuclei that are not fissionable by slow neutrons. If the excitation energy of the compound nucleus is high enough to permit fission with appreciable probability, then the (n, γ) spectrum will be perturbed by the prompt gamma spectrum from fission. This spectrum decreases rapidly with increasing gamma-ray energy, but nevertheless extends up to energies of 10 MeV [6; 7]. Since the fission cross-sections for ²³⁵U, ²³⁹Pu, and ²⁴¹Pu are greater than the (n, γ) cross-sections, the structure of the spectrum for uranium and plutonium is primarily determined by the fission spectrum. In actual fact, measurements made with Nal(TI) detectors on uranium and plutonium show a very similar shape of the spectrum [8]. Although the possibility cannot be excluded that high-resolution counters may be able to show characteristic differences, it seems unlikely that an analysis of nuclear fuel could be carried out in the presence of the fission spectrum. It is therefore suggested that the fast neutrons released by nuclear fission should be detected in 4π geometry, and that the prompt gamma rays caused by fission be excluded from the measurements by means of an anticoincidence circuit. In this way the above considerations may also be applied to fissile materials. The possibility of effective suppression of the fission spectrum will be more closely examined in section 3, together with the influence of the gamma radiation emitted by fission products.

Non-destructive analysis by means of neutron capture reactions offers a number of worth-while advantages, viz.:

1. The individual nuclides show very characteristic transitions with low line densities.

- 2. In principle all isotopes of uranium and plutonium can be covered.
- 3. By suitable choice of neutron energy an adequate transparency of the method can be ensured. ²³⁸U contributes practically nothing to the attenuation of the inducing particles (in contrast to the attenuation of gamma rays in radioactive decay). Absorption of high-energy capture radiation is very small (15 to 20 barn atom⁻¹).
- Any additional matter in the fuel can, in certain circumstances, also be detected. The canning material has practically no effect on the analysis.
- 5. The gamma-ray source strength is independent of the radioactive half-lives and for a given neutron intensity it is determined only by the cross-sections and the isotope content. The cross-sections of the most important nuclides are of the same order of magnitude. The small capture crosssection of ²³⁸U is an advantage in the analysis of fuels with low ²³⁵U or ²³⁹Pu content.
- 6. Relatively high gamma-ray source strengths can be obtained.

3. Quantitative considerations

The most important data for the isotopes of uranium and plutonium are given in the Table, but as there has so far been no systematic nuclear spectroscopic research on fissile nuclei using the (n, γ) reaction, the information is still incomplete. The following conclusions can be drawn from the Table:

- The highest-energy characteristic gamma transitions are to be expected in the region of about 4 to 6,5 MeV. For positive parity of the compound state the highest-energy electric dipole transition leads in fissile nuclei to the octupole vibrational band of the product nucleus. For negative parity E1 radiation leading to the ground-state rotational band is to be expected.
- For these energies, according to the referenced works [6; 7], the number of photons per MeV and fission lies between 10⁻¹ and 5 · 10⁻³.
- 3. If the neutron energy is increased from thermal values to about 0,1 eV, then not only the transparency is increased as a result of reduced cross-sections, but for fuel elements containing Pu it is also to be expected that the ratio $\sigma_{n\gamma}/\sigma_{nf}$ will be influenced favourably.

To make an estimate of the counting rates and measuring times to be expected, an imaginary fuel element plate composed of $15^{0/0} {}^{239}$ PuO₂ and $85^{0/0} {}^{238}$ UO₂ is taken as a basis¹. Let the plate thickness be 6 mm. The theoretical density of the oxide mixture is 11,2 g cm⁻³ (the value reached in practice is about $85^{0/0}$ of this). Let the available neutron flux density be 10^7 cm⁻² s⁻¹ and the neutron energy be 0,1 eV. If 10 cm^2 of the fuel plate are irradiated, then the number of neutrons striking the plate per second is $N_0 = 10^8$. Of these

$$N = N_0 (1 - e^{-\Sigma_a d})$$

neutrons are absorbed, where $\Sigma_a = \Sigma_a^{239} + \Sigma_a^{238}$ is the total macroscopic absorption cross-section and d is the thickness of the plate. The influence of the oxygen can be neglected. The Table shows that

 $\Sigma_{a}^{239} = 2,226 \text{ cm}^{-1}$ $\Sigma_{a}^{238} = 0,036 \text{ cm}^{-1}$ $N = 7,42 \cdot 10^7 \text{ s}^{-1}$

and

 $^{^{\}rm t}$ Corresponding to the approximate composition of fuel for a fast breeder reactor.

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The number of fissions is calculated as

$$N_{f} = \frac{\Sigma_{f}^{239}}{\Sigma_{a}} N$$

where $\varSigma_{\rm f}^{239}$ is the macroscopic fission cross-section for $^{239}{\rm Pu}.$ Thus $N_{\rm f}=4,70\cdot10^7~{\rm s}^{-1}.$

For the (n, γ) processes the equations are

and

$$N_{\mathbf{n}\gamma}^{238} = \frac{\mathcal{Z}_{\mathbf{n}\gamma}^{238}}{\mathcal{Z}_{\mathbf{n}}}$$

 $N_{n\gamma}^{239} = rac{\Sigma_{n\gamma}^{239}}{\Sigma} N$

From the Table it follows that

and

$$U_{238}^{238} = 1.19 \cdot 10^6 \, \mathrm{s}^{-1}$$

 $N_{n_{N}}^{239} = 2.61 \cdot 10^7 \, \text{s}^{-1}$

Since there are as yet no measurements on the (n, γ) reactions, reasonable assumptions must be made for the absolute intensities of the available gamma transitions. The total number of primary transitions from the capture state is distributed over an energy range with a maximum of 6,5 MeV. It is therefore not unrealistic to assume that at least 0,05 photons per MeV and per capture process are emitted in the measuring range. The number of available transitions is thus

	$\left(N_{n\gamma}^{239} ight)' = 1,31 \cdot 10^{6} \ { m s}^{-1} \ { m MeV^{-1}}$
and	
	$\left(N_{n\gamma}^{238} ight)' = 5,95 \cdot 10^4 \ { m s}^{-1} \ { m MeV}^{-1}.$

If the solid angle of the gamma detector is $2 \cdot 10^{-3}$, then

and

$$(N_{n\gamma}^{239})_{det} = 2,62 \cdot 10^3 \text{ s}^{-1} \text{ MeV}^{-1}$$

 $(N_{n\gamma}^{238})_{det} = 1,19 \cdot 10^2 \text{ s}^{-1} \text{ MeV}^{-1}$

will be the number of photons striking the detector.

If a lithium-drifted germanium counter is used as a highresolution detector, then the double-escape pair lines are utilized for spectroscopy. For a counter with 12 mm depletion depth there is a probability of about [13] $1,5 \cdot 10^{-2}$ in the energy range from 4 to 6,5 MeV that the incident quantum will be absorbed by pair production, the positron-electron pair will lose all its energy in the counter, and both annihilation quanta will leave the detector. The peak counting rates associated with this are about

$$\left(\begin{array}{c} N_{n\gamma}^{239} \end{array}
ight)_{ extsf{peak}} = 40 \ extsf{s}^{-1} \ extsf{MeV}^{-1}$$

and

$$N_{n_{\gamma}}^{238}$$
 = 2 s⁻¹ MeV

For a statistical accuracy of 1 %, the measuring times required will therefore be approximately

5 min for ²³⁹Pu and 100 min for ²³⁸U.

Thus the calculation shows that the measuring times required for application of the procedure are in a reasonable order of magnitude. By optimising the counter and by using two or more detectors in combination with modern data processing equipment, these times may be further reduced. An important restraint in the detector geometry is the maximum permissable total counting rate of the detector for given energy resolution. If a multiplicity of 4 is assumed for radiative capture, and bearing in mind that fission leads to the emission of an average of 9 gamma quanta, then the



Fig. 2: Intensities of the components contributing to the gamma-ray spectrum Fig. 2: Intensitäten der einzelnen Komponenten des Gammaspektrums

above estimates give a figure of about 10⁶ photons per second falling on the detector. A significant part of these quanta has low energies and may be filtered out by a few mm of lead. Nevertheless, overall counting-rates of 10⁵ cps or more are to be expected. Counting-rate effects originate mainly in the electronics, but with present day techniques (pole-zero cancellation, base-line restoration, anti-pileup discrimination and gate methods) counting rates of up to 10⁵ cps may be used without seriously impairing the energy resolution [14]. Further improvements should be technically possible.

Measurement of the neutron capture spectrum is disturbed by two effects: the prompt gamma rays from fission, and-to a lesser extent-the delayed gamma rays from the fission products. Fig. 2 shows the results of a quantitative estimation of the individual components. The intensities per MeV and fission for the most important fissile isotopes ²³⁵U, ²³⁹Pu and ²⁴¹Pu were deduced from the Table, together with the energy position of the most energetic primary transitions. In calculating the intensities is has again been assumed that 0,05 available photons are emitted per MeV and per capture process. The prompt spectrum from fission can be taken directly from Refs [6; 7]. As will be shown later, a reduction of this spectrum in the anticoincidence method by a factor of 20 appears to be realistic. The intensity curve for the interfering spectrum arising from beta decay of the fission products is derived from experimental works [15; 16], in which all the half-lives between 350 ms and 1 h were included. Anticoincidence has no effect on the delayed component. The upper limit of 1 h is reasonable for this assessment as measuring times of this order are the highest considered in practice. Should measuring times of substantially less than 1 h be possible-which on the basis of the above considerations and the conservative estimate of the intensity per capture seems to be feasible-then the situation will be more favourable than is assumed in Fig. 2 It is worth-while to note that the effective counting rate, in contrast to the other components, is concentrated in a few peaks. One can therefore conclude from Fig. 2 that the (n, γ) process may be expected to give promising results for the most important fissile isotopes. The question remains whether ²³⁸U can be detected quantitatively when using thermal or near thermal neutrons (see section 5 on this topic).

KERNTECHNISCHE MESSVERFAHREN · NUCLEAR MEASUREMENTS

Table: Neutron capture in uranium and plutonium isotopes ¹⁾							Tabelle: Neutroneneinfang in Uran- und Plutonium-Isotopen						
Isotopes	Thermal neutrons ²⁾				En				 E.	·	$E_{\rm n}=0,1~{\rm eV}$		
	σ _{ny} barn	^ơ nf barn	$\sigma_{n\gamma}/\sigma_{nf}$	$\frac{1}{\nu}$	keV ³⁾	J _T	J _c	J _p	keV	Ja	σ _{nγ} barn ⁴⁾	^σ nf barn⁴)	$\sigma_{n\gamma}^{\ \ \sigma_{nf}}$
²³³ U	49,4	525,9	0,094	2,51	6783	5/2+		0+					
. ²³⁴ U	95		~	_	5267	0+	1/2+	7/2-					
²³⁵ U	100,9	578,3	0,175	2,44	6467	7/2-	(4~)	0+	150	4+	50	250	0,17
²³⁶ U	6	-	80	_	5304	0+	1/2+	(1/ 2 +)					
²³⁸ U	2,73	_	80	-	4784	0+	1/2+				~ 2	-	∞
²³⁹ Pu	265,8	743,1	0,358	2,88	6455	1/2+	(1+)	0+	597	(1-)	250	450	0,6 ⁵⁾
²⁴⁰ Pu	250	<0,1	>2500		5412	0+	1/2+	5/2+			150		
²⁴¹ Pu	425	950	(0,39)	3,00	6219	5/2+		0+			300	600	0,5
²⁴² Pu	19	<0,2	>95		5047	0+	1/2+						

1) Where

 $\sigma_{\mathbf{n}\gamma}$ radiative capture cross section $\sigma_{\mathbf{n}\mathbf{f}}$ fission cross section

 \bar{v} fission neutrons per fission

E_B neutron binding energy

 $J_{\rm T}^{-}, J_{\rm C}^{-}, J_{\rm p}^{-}$ spin of target nucleus, of compound nucleus and of product nucleus in ground state

 $E_{\rm a}$, $J_{\rm a}$ excitation energy and spin of the first state which can be populated by E1 radiation

The best anticoincidence shield for fission neutrons is a liquid or plastic scintillator. Since an immediate response is required, detection cannot be effected by the slowing down of the neutrons and their subsequent absorption in boron. Rather must the recoil protons be counted directly. The light yield should be as efficient as possible (65 to 80% anthracene) to ensure a low value for the counting threshold. Suitable substances are commercially available (e.g. NE 213, NE 224, or NE 102 A) or can be prepared in the laboratory. For the detection efficiency, use can be made of the fact that each fission process releases on average at least 2,5 neutrons. This means that a detection efficiency of 70% for the individual neutron is sufficient to detect a fission event with 95% efficiency. The theoretical response is approximately

 $W = \left(1 - e^{-\frac{L \varrho}{M} n \sigma_{s} d} \right)$

where

L = Loschmidt number

e = density of scintillator

M = molecular weight

n = number of hydrogen atoms per molecule

 $\sigma_{\rm s}$ = scattering cross-section for hydrogen

d = thickness of scintillator

Taking pseudocumol (1, 2, 4-trimethylbenzole) as an example, $\varrho=$ 0,88 g cm $^3,~M=$ 120,2 and n = 12.

The mean energy of the fission neutrons is about 1,8 MeV. The scattering cross-section for hydrogen at this energy is 3 barn. Thus with a thickness of 15 cm, a theoretical detection efficiency of about 90% is obtained. This does not take into account losses due to the finite detector threshold, for which values of about 50 keV are obtainable. Then a detection efficiency of 70% can certainly be achieved. The reduction of the fission spectrum by a factor of 20 assumed above is therefore justified.

Provided that the high-energy transitions do not lead directly from the capture state to the ground state, they occur in coincidence with low-energy photons. The neutron detector must be shielded by several centimetres of lead to prevent these gamma quanta from producing any anticoincidence.

2) See [9; 10; 11]

³⁾ See systematics of the heavy elements [12]

4) Estimated values using [10]

⁵⁾ See [10]

For 500 keV radiation 4,2 cm are sufficient to achieve an attenuation of two orders of magnitude. The optimum dimensions for the lead shielding depend on the level schemes which are not yet known. In some circumstances the shielding can be kept thin, and some of the (9 ± 2) quanta occurring on the average in nuclear fission can also be used for the anticoincidence. The mean energy of the prompt gamma rays from fission is $(1,0 \pm 0,2)$ MeV. The lead shield has no specific significance for the fast fission neutrons. Inelastic scattering in the lead will not appreciably affect the detection efficiency of the scintillator.

Finally, the transparency of the method for the imaginary fuel element assumed above is compared with other methods. The halfthickness for 0,1 eV neutrons is calculated from Table 1 to be 3,1 mm. Compared with this, absorption of the gamma rays is negligible. During the measurement a fuel element would be rotated in such a manner as to ensure optimum irradiation of all parts of the rod. For thermal energies the half-thickness is 2,1 mm. If the plutonium content is analysed by means of the natural radioactive decay through the characteristic gamma radiation between 333 and 414 keV, a half-thickness between 1,9 and 2,9 mm is calculated for the assumed composition. A major step forward in the direction of improved transparency can be made by inducing the (n, γ) process by neutrons of substantially higher energy. (See section 5.)

4. Measuring arrangements

Fig. 3 shows a rough schematic sketch of one possible measuring arrangement. In the centre of the apparatus a collimated neutron beam falls on the fuel element, which is moved in a plane perpendicular to that of the drawing. ⁶Li shielding (in the form of hydride or carbonate) prevents the penetration of primary or scattered slow neutrons into the neutron detector and the gamma counter. The lead shield and the scintillator completely surround the measuring zone, and a number of photomultipliers are mounted on the scintillator. The gamma detector is shielded at the sides by lead, and "sees" virtually only the filtered gamma rays from the fuel element.



Fig. 3: Schematic view of an experimental set-up Fig. 3: Schematische Darstellung einer Meßanordnung

Such an arrangement can most conveniently be set up on a reactor. Practically monoenergetic neutrons (of 0,1 eV for example) are obtained by means of Bragg reflection from a single crystal (lead, for example). This method has frequently been found to be useful in (n, γ) spectroscopy (see Refs. [17; 18] and others).

5. Use of an electron linear accelerator as a neutron source

An electron linear accelerator offers various advantages over a reactor as a neutron source, viz.:

- 1. The transparency can be substantially improved. A neutron beam filtered by ²³⁵U, ²³⁹Pu etc., which keeps the crosssections small enough for all incident neutrons, appears to be most suitable.
- 2. The values for the capture-to-fission ratio are considerably higher than in the thermal region.
- 3. Since the cross-section of ²³⁸U exhibits a clear resonance structure, the intensities of the gamma lines of ²³⁹U compared with those of the other isotopes can be considerably improved. ²³⁸U can also be covered with certainty by this method.

With the electron linear accelerator (and a suitable filter), all neutron energies below about 5 keV can be utilized. The upper limit is set by the requirement that the line width in the gamma spectrum must not be appreciably affected by the kinetic energy of the captured neutrons. For good germanium counters the full width at half-maximum for 7 MeV gamma-ray energy is in the region of 7 keV. Photon emission induced by neutrons with energies above 5 keV can be excluded by means of the time-of-flight method. The demands made on the accelerator are conventional.

The ratio $\sigma_{n\gamma}/\sigma_{nf}$ is only approximately known in the keV region [19]. Experimental values for ²³⁹Pu vary between 0,3 and 4,0 with indication of certain structures. Most of the data relating to the energy range from 100 eV to 5 keV lie around 1,0 or above this value. For ²³⁵U a mean value of about 0,5 is found in this region.

KERNTECHNISCHE MESSVERFAHREN · NUCLEAR MEASUREMENTS

6. Final conclusions

The considerations outlined in the preceeding sections lead to the conclusion that it is possible to carry out a non-destructive analysis of unirradiated fuel elements by means of neutron capture reactions. The efficiency of the method should now be further investigated by some experiments on a reactor. Since systematic studies of radiative capture in fissionable nuclei have not yet been performed, it is particularly necessary to analyse the (n, γ) spectra in detail by high-resolution spectrometers, using separated isotopes as far as possible. Of greatest interest are the energies and intensities of the primary transitions from the capture state, together with their coincidence relationships. On the technical side it would be desirable to develop a semi-conductor spectrometer for extremely high input counting rates. As a further step, more research on neutrons from a electron linear accelerator should be carried out.

To what extent the procedure may be applied to irradiated fuels (after a sufficient cooling time) would require a further detailed investigation.

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