ANNUAL·REPORT·90

Central Bureau FOR Nuclear Measurements

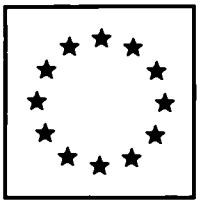
JOINT RESEARCH CENTRE

COMMISSION OF THE EUROPEAN COMMUNITIES

EUR 13456 EN

N N U A L · R E P O R T · 90

Central Bureau FOR Nuclear Measurements



JOINT RESEARCH CENTRE

COMMISSION OF THE EUROPEAN COMMUNITIES

EUR 13456 EN

CATALOGUE N° : CD-NA-13456-EN-C

TABLE OF CONTENTS

Page

WORK FOR THIRD PARTIES

GENERAL INFORMATION

Editor: H.H. Hansen

Note : For further information concerning the Programme Nuclear Measurements and Reference Materials, please contact W. Müller, Programme Manager.

PROGRAMME : NUCLEAR MEASUREMENTS AND REFERENCE MATERIALS

Executive Summary

W. Müller

This report covers CBNM's activities during the third year of the multiannual programme 1988-91.
The main task of CBNM as covered by the European Communities Framework Programme is defined as the specific programme "Nuclear Measurements and Reference Materials".
The activities of the CBNM - like for the other institutes of the JRC - are only in part funded as Specific Programme. A small proportion of the specific programme budget is allotted to Exploratory Research, in preparation of possible extensions of existing competences or of potential new activities. Parts of the funding are coming from Support to Other Commission Services and from Work for Third Parties on the basis of contracts.
Mobility and rejuvenescence of staff were fostered by contacts with the academic world, national and international research institutions and industry, leading to an increased number of fellowships and grants, including possibilities for visiting scientists and detached experts.
Despite severe understaffing due to (mainly) early retirement departures and delays in recruiting and budget difficulties during the reporting period, there are again examples of impressing achievements in both projects "Nuclear Measurements" and "Reference Materials".

In the field of neutron data for standards, for fission and for fusion application, (Project "Nuclear Measurements"), the nuclear charge distribution and odd-even effects for mass, charge and neutron number in the cold spontaneous fission of ^{252}Cf were determined. X- and γ-ray emission probabilities were evaluated in the frame of an IAEA coordinated Research Project.
The subthermal fission cross section measurements of ^{235}U, ^{233}U and ^{239}Pu, were finalised and reported - together with η of ^{235}U - at PHYSOR 90.
The dependence of the experimental weighting function of C_6D_6 detectors on thickness of several ^{56}Fe samples was determined.

Fusion data studies involved the development of a light-ion telescope with improved time - and energy resolution. Double differential cross-sections of ^{9}Be were analysed.

Radionuclide metrology dealt with the response of silicon detectors to electrons, deuterons and α-particles, as well as with the standardization of ^{192}Ir sources (EUROMET project).

Project "Reference Materials" reports the EC Certification of nuclear reference materials 210 (PuO_2), 523 (Al), 525 (Nb) and 526 (Nb), the latter three ones for neutron dosimetry.
Progress was achieved in the preparation of dried solid spikes of uranium and plutonium for undiluted reprocessing input solution analysis.
^{10}B and ^{6}Li deposits were prepared and characterized for a successful international redetermination of the neutron lifetime.
Additional intercomparisons were organized and executed within the CBNM guided Programmes REIMEP and "Esarda-LEU".
Preliminary studies on speciation of trace metals in biological fluids were successful.
More than 200 samples and targets were prepared for CBNM and other customers, biological and environmental RMs were prepared for the Community Bureau of Reference (BCR).

Upon request, radioactive waste barrels were analysed by γ-scanning (NIRAS/ONDRAF) and blood samples were irradiated with 0.6 MeV neutrons (SCK/CEN).

Exploratory research resulted in first measurements of transition radiation properties in cooperation with ALS (Saclay).

SPECIFIC RESEARCH PROGRAMME

PROJECT 1 : NUCLEAR MEASUREMENTS

Introduction

A. J. Deruytter

In the 1988-1991 multiannual research programme the project Nuclear Measurements contributes to the Specific Programme, Exploratory Research and Work for Third Parties.

The contribution to the Specific Programme is in the area of Nuclear Data and Nuclear Metrology, where the main objectives are summarized as follows :

- to improve the well-defined neutron standards data set relative to which partial cross-sections or other quantities important in fission or fusion technology are determined,
- to improve radionuclide decay data needed for standards application,
- to develop nuclear measurement techniques for nuclear (e.g. neutron flux and dose) and non-nuclear (e.g. environmental, medical) applications.

In 1990 the efforts for the improvement of the set of **standard neutron cross-sections** and other quantities selected whithin the INDC/NEANDC Standards File continued. In particular additional information on the nuclear charge distribution and the odd-even effects for mass, charge and neutron number in the cold spontaneous fission of ^{252}Cf was obtained and the results of an evaluation of X- and γ-ray emission probabilities of 23 radionuclides used in detector calibration were reported in the frame of an IAEA-Coordinated Research Project. An investigation to improve the knowledge of the ratio of two important standard cross-sections $^{235}U(n,f)/H(n,n)$ based on the use of octacosanol layers, as hydrogen containing component, was started.

In the field of **nuclear data for fission technology** work was concentrated on European requests in the NEA High Priority Request List. The final results for the subthermal fission cross-sections of ^{233}U, ^{235}U and ^{239}Pu and η of ^{235}U were reported at the Physics of Reactors (PHYSOR) Meeting in Marseille, April 1990. They were used in JEF2. Also new subthermal fission cross-sections were obtained for ^{241}Pu and the g_f factor calculated. In the framework of the NEANDC Task Force on the 1.15 keV resonance of ^{56}Fe the experimental weighting function of C_6D_6 n-capture detectors which solved the discrepancy between capture and transmission data was investigated for its dependence on the sample thickness. This experimental weighting function could then be used in the analysis of the capture cross-section of ^{60}Ni below 300 keV neutron energy. Parameters were obtained for 178 resonances of which 19 were observed for the first time in these high resolution measurements. Also a measurement station was made operational for the measurement of γ-ray decay

spectra from single resonances and first observations were made for capture in ^{53}Cr.

In the field of **nuclear data for fusion technology** measurements continued aiming at an improvement of relevant data for neutron transport calculation in the blanket and for prediction of gas production, in particular double differential neutron-emission cross-sections of ^{9}Be were analysed and the results transmitted for use in EFF. Also a light ion telescope with improved time- and energy resolution was developed and tested for (n, charged particle) reaction studies.

The **radionuclide metrology** subproject follows three lines : determination of decay-scheme data, preparation of special standards and the improvement of measurement techniques including international comparisons. In 1990 the detailed study of the response of silicon detectors to electrons, deuterons and α-particles was finalised and published. Also an international cooperation in the frame of an EUROMET project was started to provide air kerma rate and activity standards of ^{192}Ir wires and to develop a scanning device for the assessment of the longitudinal homogeneity of such ^{192}Ir wires used in radiotherapy.

In the area of **neutron metrology**, neutron flux and dose determinations were provided for neutron irradiations in radiobiological studies at one of the Van de Graaff accelerators.

Exploratory Reasearch concerned the study of transition radiation (TR) generated when energetic electrons cross the boundary between two media. Several properties of transition radiation were measured at a 550 MeV electron beam of ALS and microlithography with 300 nm resolution was achieved.

Work for Third Parties consisted mainly of (1) irradiations of blood samples for the Radiobiology Department of SCK/CEN with a neutron beam from the Van de Graaff accelerator and (2) analysis of several waste barrels for the Belgian organisation responsible for treatment and storage of radioactive waste (NIRAS/ONDRAF) by using a γ-ray scanning device.

GELINA was mainly used with short burst-widths (< 1 ns) for high resolution neutron production. It was also operated with large burst-widths (2000 ns) for low-energy neutron work and for transition radiation studies. The **CN-7 MV Van de Graaff** was mainly used for neutron data measurements and also for neutron irradiations in radiobiological studies. The **central computer** (IBM/4381/2) was chiefly used for batch processing and interactive work for the analysis of experimental data.

NUCLEAR DATA

NUCLEAR DATA FOR STANDARDS

Neutron Data for Standards

Standard Cross Section Ratio $^{235}U(n,f)/H(n,n)$

F.-J. Hambsch, R. Vogt, G. Willems*

As already outlined in the previous progress report[1], an investigation has started aiming at an improved determination of the cross section ratio $^{235}U(n,f)/H(n,n)$.

In the present reporting period a highly polished backing material could be supplied. The selection of a suitable hydrogen compound was made by way of electron microscope pictures of all considered compounds of the same thickness, of which octacosanol (CH_3-$(CH)_{26}$ - CH_2OH) looked most promising. Several samples have been prepared and three of them have been used for a first test measurement to check the homogeneity by means of the cosine-dependence of the ionization chamber signals. The schematical view of the experimental set-up is shown in Fig. 1. During the experiment many problems with electronics and vibrations of the experimental platform have been encountered. Due to this data acquisition has been delayed. A conclusion as to whether the new component is suitable or not could therefore not be drawn yet.

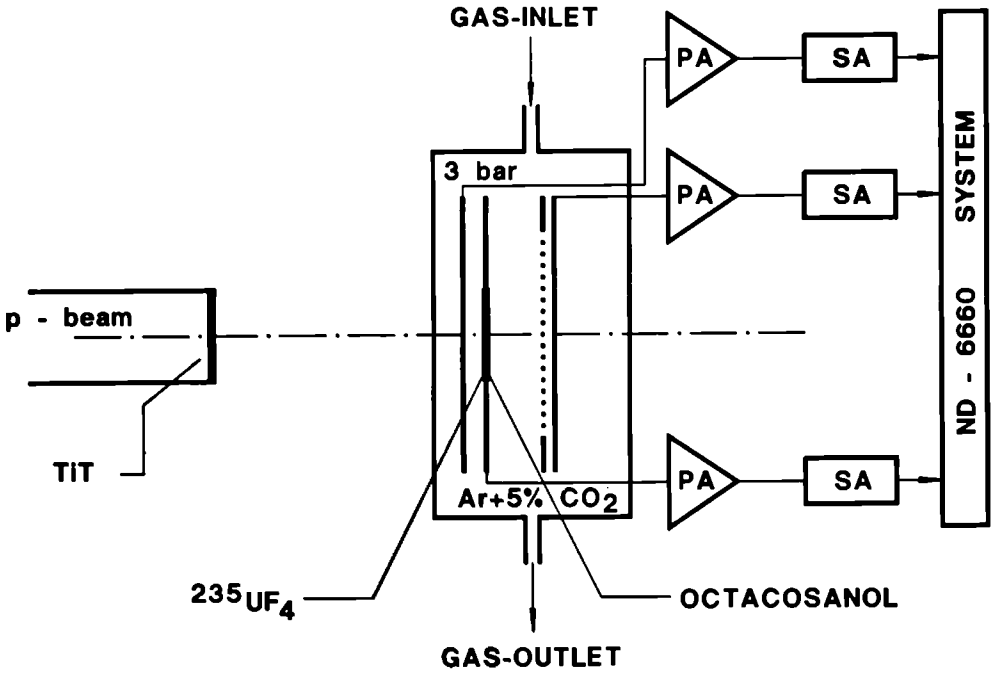


Fig. 1. Schematic experimental set-up

* Scientific Visitor from KU Leuven, Belgium

(1) CBNM Annual Report 89, EUR 12615 EN

Investigation of the Correlation Between Prompt Gamma-Ray Emission and Fission Fragments from $^{252}Cf(sf)$

F.-J. Hambsch, R. Vogt, B. Jäckel*

In the present reporting period the set-up described in the last progress report[1] has been completed and tests have been performed on the fission fragment detector together with the γ-ray detector. It has been found that the available γ-ray detector could not be used due to a neutron damage which results in a poor energy-resolution. This delayed the whole measurement and only recently data acquisition could be started. Until now, approximately $9 \cdot 10^6$ events were recorded in coincidence with the γ-ray detector and about the same number of events to be used for calibration purposes.

New Results from Cold Spontaneous Fission of ^{252}Cf

F.-J. Hambsch, H.-H. Knitter

In addition to the evaluation mentioned in the previous progress report [1], further investigation has been performed on the spontaneous fission of ^{252}Cf in the so-called cold fragmentation region, were the reaction Q-value is nearly exhausted by the total kinetic energy, TKE, of the fission fragments.
The nuclear charge information has been obtained from the experimental double-ratio

$$R(Z_L) = \frac{(P_{anode,L} - P_{sum,L})/P_{anode,L}}{(P_{anode,H} - P_{sum,H})/P_{anode,H}} = \frac{\overline{X}_L(E_L, A_L, Z_L)}{\overline{X}_H(E_H, A_H, Z_H)}$$

X is the distance between the centre of gravity of the charge distribution and the fragments' ion trace. P_{anode} is the anode and P_{sum} the sum signal of anode and grid[2]. Fig. 2 shows the experimental nuclear charge distribution for a total excitation energy, TXE, of 11 MeV and for two selected masses A_L = 106 and 109, together with the convolution of individual charges (dashed lines). Since the nuclide yields for each bin are measured, it is possible to sum up the nuclides with even-even, odd-odd, even-odd and odd-even proton and neutron numbers respectively. Also the odd-even effects for the mass δ_A, nuclear charge δ_Z and neutron number δ_N can be obtained. These numerical values are given in Table 1.

---

* Scientific Visitor from Philipps University, Marburg, Germany

(1) CBNM Annual Report 89, EUR 12615 EN

(2) C. Budtz-Jørgensen, H.-H. Knitter, Ch. Straede, F.-J. Hambsch and R. Vogt, Nucl. Instr. Methods A258 (1987) 209

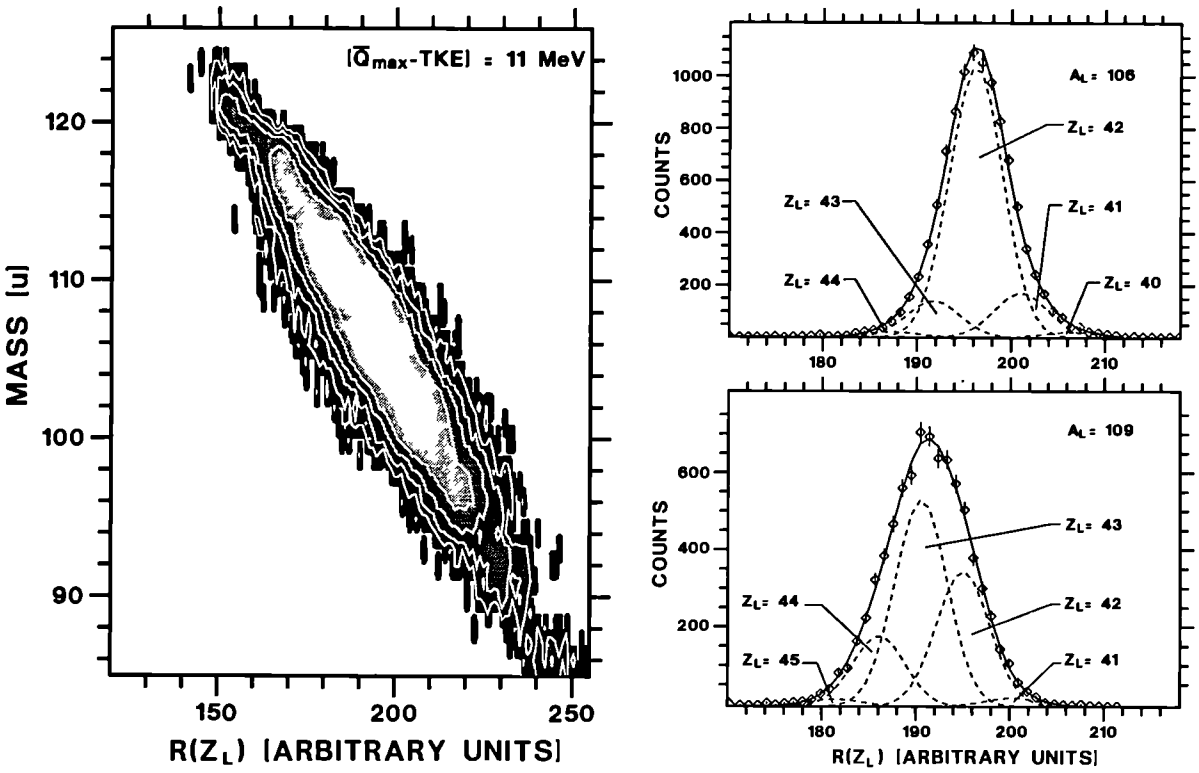


Fig. 2 . *Experimental nuclear charge distribution for Q_{max}-TKE = 11 MeV. Left : contour - image representation of the mass-charge correlation. Right : cut through the left picture for two selected masses and convolution of individual charges (dashed lines).*

Table 1. *The odd-even effects for fragment mass, nuclear charge and neutron number as well as the nuclear charge variance, are given for the different TKE-evaluation bins. The four lower lines give the relative yields for even-even, odd-odd, even-odd and odd-even nuclear charge and neutron number respectively*

	BIN 1 (0-2)MeV	BIN 2 (2-4)MeV	BIN 3 (4-6)MeV	BIN 4 (6-8)MeV	BIN 5 (8-10)MeV	BIN 6 (10-12)MeV	BIN 7 (12-14)MeV	BIN 8 (14-16)MeV
δA	0.19 ± 0.06	0.06 ± 0.03	0.014 ± 0.014	0.007 ± 0.008	0.000 ± 0.005	0.009 ± 0.003	0.000 ± 0.002	0.003 ± 0.002
δZ	-	0.48 ± 0.04	0.45 ± 0.01	0.506 ± 0.007	0.370 ± 0.005	0.304 ± 0.003	0.238 ± 0.002	0.183 ± 0.002
δN	-	0.13 ± 0.05	0.06 ± 0.02	0.068 ± 0.008	0.045 ± 0.005	0.033 ± 0.003	0.018 ± 0.002	0.013 ± 0.002
$<\sigma_Z^2>$	-	0.16 ± 0.09	0.32 ± 0.11	0.33 ± 0.66	0.40 ± 0.04	0.45 ± 0.03	0.48 ± 0.02	0.50 ± 0.02
EE[%]	(59)	37.6 ± 4	36.9 ± 1.2	40.7 ± 0.6	35.1 ± 0.3	33.6 ± 0.2	31.4 ± 0.2	29.7 ± 0.2
OO[%]	(0)	6.7 ± 2	11.4 ± 0.5	12.0 ± 0.3	14.8 ± 0.2	16.8 ± 0.1	18.6 ± 0.1	19.8 ± 0.1
EO[%]	(33)	36.7 ± 3	35.6 ± 1.0	34.6 ± 0.5	32.9 ± 0.3	31.6 ± 0.2	30.5 ± 0.2	9.5 ± 0.1
OE[%]	(7)	19.8 ± 1	16.1 ± 0.6	12.7 ± 0.3	17.1 ± 0.2	18.0 ± 0.2	19.5 ± 0.1	21.0 ± 0.1

δ_A is essentially zero giving indication for the randomness of the neck-rupture at scission even at very small TXE. δ_Z and δ_N on the contrary show linear dependence with TXE, however the magnitude of δ_Z is about five times larger than that of δ_N.

The odd-even effects for proton and neutrons have been also evaluated as function of mass-split, an example of which is shown in Fig. 3.

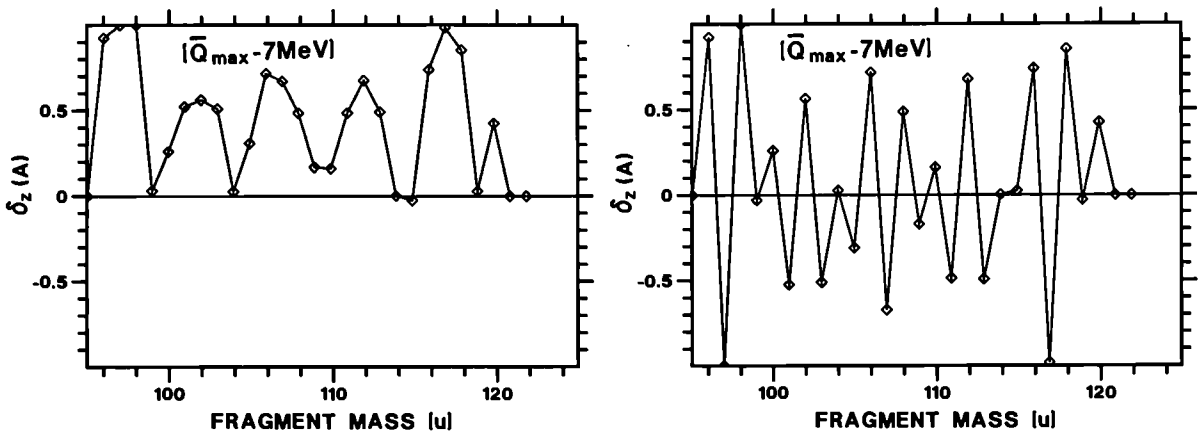


Fig. 3. ***Local odd-even efffects for protons (left) and neutrons (right) as function of fragment mass.***

A clear undulatory structure with a period of five mass units is seen in $\delta_Z(A)$, whereas $\delta_N(A)$ shows strong fluctuations from one mass to the other. The measured nuclide yields are a direct picture of the structures visible in the Q-value energy surface Q(A,Z) (Fig. 4), which accounts for the undulatory structure seen in $\delta_Z(A)$ and other parameters. Following the behaviour of Q(A,Z) which means cutting not parallel to the highest Q-value but parallel to individual Q-values for single charges, changes the picture drastically as seen in Fig. 5. The undulatory structure seen in the picture on the left essentially disappears in the picture on the right, where $\delta_Z(A)$ is evaluated for a constant excitation energy TXE(A, Z). The remaining structure in the picture on the right can be understood by level density considerations. The level densities close to the ground state are larger for odd-odd fragments than for even-even fragments and therefore fragmentations with broken nuclear pairs are favoured. Furthermore δ_Z or δ_N can no longer be interpreted as being a measure of the excitation energy of the fissioning nucleus at the moment of scission, because these values are close to zero or even slightly negative. This would give intrinsic excitation energies close to infinity, if they are calculated as in proposed models (TXE ~ ln δ)[1].

---

(1) H. Nifenecker, G. Marolopoulos, J.P. Bocquet, R. Brissot, Ch. Hamelin, J. Crançon and Ch. Ristori, Z. Phys. A308 (1982) 39

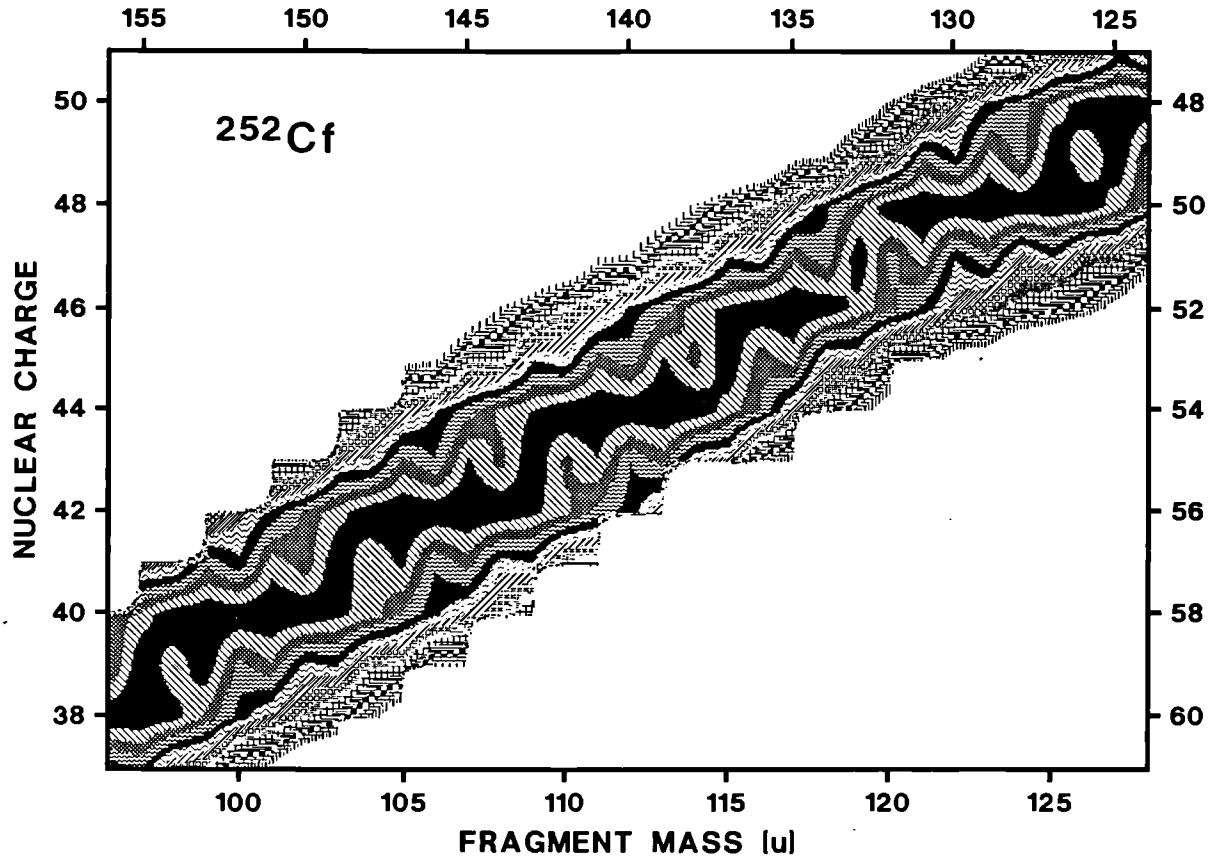
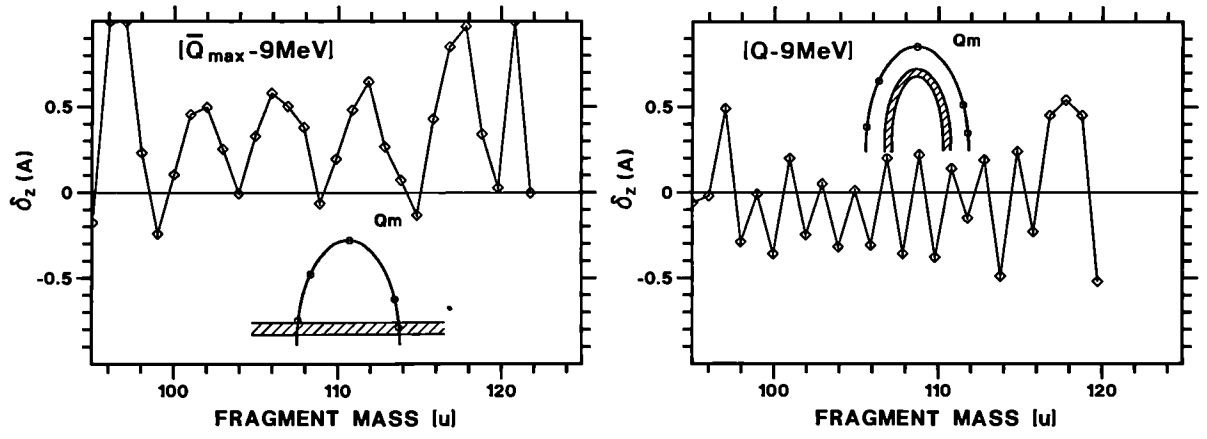


Fig. 4. *The Q-value energy surface Q(A,Z) in a grey-shaded representation.*



Fig; 5. *Local proton odd-even effect at (Q-9 MeV). Left : for cuts parallel to Q_{max}. Right : for cuts with constant distance from Q(A,Z).*

Non-Neutron Nuclear Data For Standards

Half Life of ^{125}I

T. Altzitzoglou

The evaluation of the measurements to determine the half life of ^{125}I has been finished. The data, corrected for background, dead time and pile-up, were fitted using a weighted least-squares fitting routine. Table 2 shows the results obtained from this work. They are in good agreement with the most recent results from the literature.

Table 2. Results of the ^{125}I half-life measurements

Source detector distance [mm]	Number of data points	Measuring time t / Halflife $T_{1/2}$	$T_{1/2}$ [d]
16	52	1.0	59.37 ± 0.04
54	163	1.7	59.37 ± 0.02
94	72	0.7	59.35 ± 0.06

The uncertainties quoted are those resulting from the weighted fitting. In the final calculation of the uncertainty, the following components were added to the statistical uncertainty : dead-time correction 0.01 %, background 0.05 %, timing 0.005 %, accidental coincidences 0.1 %, and threshold setting 0.1 %. An attempt was made to fit the data to a mixture of ^{125}I and ^{126}I, but with no success, which is in agreement with no detection of ^{126}I impurity. In order to check the results further, the data were divided into subgroups and each subgroup was analyzed independently by the method described by Walz et al. in 1983[1].
The adopted value for the half life of ^{125}I is (59.37 ± 0.06) d. A publication describing these measurements is in press.

Evaluation of X- and Gamma-Ray Emission Probabilities

W. Bambynek

In the frame of an IAEA Coordinated Research Project (CRP) on X- and Gamma-Ray Standards for Detector Efficiency Calibration the evaluated values of decay data for 23 radionuclides were communicated to the IAEA to be printed in the final document.

---

(1) K.F. Walz, K. Debertin and H. Schrader, Int. J. Appl.Radiat. Isot. 34 (1983) 1191

Reorganization of Atomic Shells after Radioactive Decay

W. Bambynek

The principles to produce inner-shell vacancies after nuclear decay by (a) orbital electron capture of the nucleus and (b) by internal conversion of gamma rays were summarized. In addition, the principles of the reorganization of atomic shells by radiative and radiationless (Auger and Coster-Kronig) transitions after radioactive decay were reviewed. Two invited lectures were given at the Second International Summer School on Low-Level Measurements of Man-Made Radionuclides in the Environment, La Rábida, Spain.

NUCLEAR DATA FOR FISSION TECHNOLOGY

Neutron Data of Actinides

Subthermal Fission of the Common Fissile Isotopes

C. Wagemans*, P. Schillebeeckx**, A.J. Deruytter, R. Barthélémy, J. Van Gils

The measurements of the subthermal fission cross-section of ^{233}U, ^{235}U, ^{239}Pu and ^{241}Pu have been finalized. For these experiments, a liquid nitrogen cooled methane moderator has been installed at GELINA to enhance the neutron production below 20 meV neutron energy. The accelerator was operated at a 40 Hz repetition frequency with 2 μs burst width and with an average electron current of 15 μA. An evaporated layer of 25 μg $^{6}LiF/cm^2$ for the neutron flux determination and a thin fissile layer were mounted back-to-back in the centre of a large vacuum chamber. The fission fragments and the particles from the $^{6}Li(n,\alpha)t$ reaction were detected with two large surface barrier detectors placed outside the neutron beam.

For the $^{6}Li(n,\alpha)t$ reaction cross-section, a 1/v-shape was assumed in the energy region below 20 eV. The ratio of the background corrected fission and $(\alpha+t)$ counting-rates yields the $\sigma_f(E)\sqrt{E}$-shape, which still needs to be normalized. This normalization was done in the thermal region relative to the σ_f°-value of the ENDF/B6 file.

Final results for ^{233}U, ^{235}U, and ^{239}Pu and partial results for ^{241}Pu have been presented at PHYSOR 90 in Marseille. These data had a strong impact on the JEF-2 evaluated data file for the actinides, as illustrated in Fig. 6 for $^{239}Pu(n,f)$.

In the first series of measurements of the $^{241}Pu(n,f)$ cross-section a 8 $\mu g/cm^2$ ^{241}Pu layer (deposited as an acetate) was used. For the present experimental campaign, a 27 μg ^{241}Pu layer has been prepared. In both cases, a chemical Am/Pu separation was performed prior to the sample preparation. Two runs have been performed with the neutrons bombarding a Li/Pu respectively a Pu/Li sample configuration. This was done to make sure that no absorption phenomena occurred. Since the results obtained in both runs were in agreement, they were converted into a single $\sigma_f(E)$ data set.

These new data have a much better statistical accuracy than those obtained with the 8 $\mu g/cm^2$ sample, with which they agree however within the experimental uncertainties. Fig. 7 shows the $\sigma_f(E)$ data from 0.002 eV up to 20 eV obtained in the present experiments. These data confirm the interference effect near 2 eV recently observed [1].

---

* Rijksuniversiteit Gent, Belgium
** Present address : JRC, Ispra, Italy

(1) H. Derrien and G. de Saussure, ORNL/TM-11123 (1989)

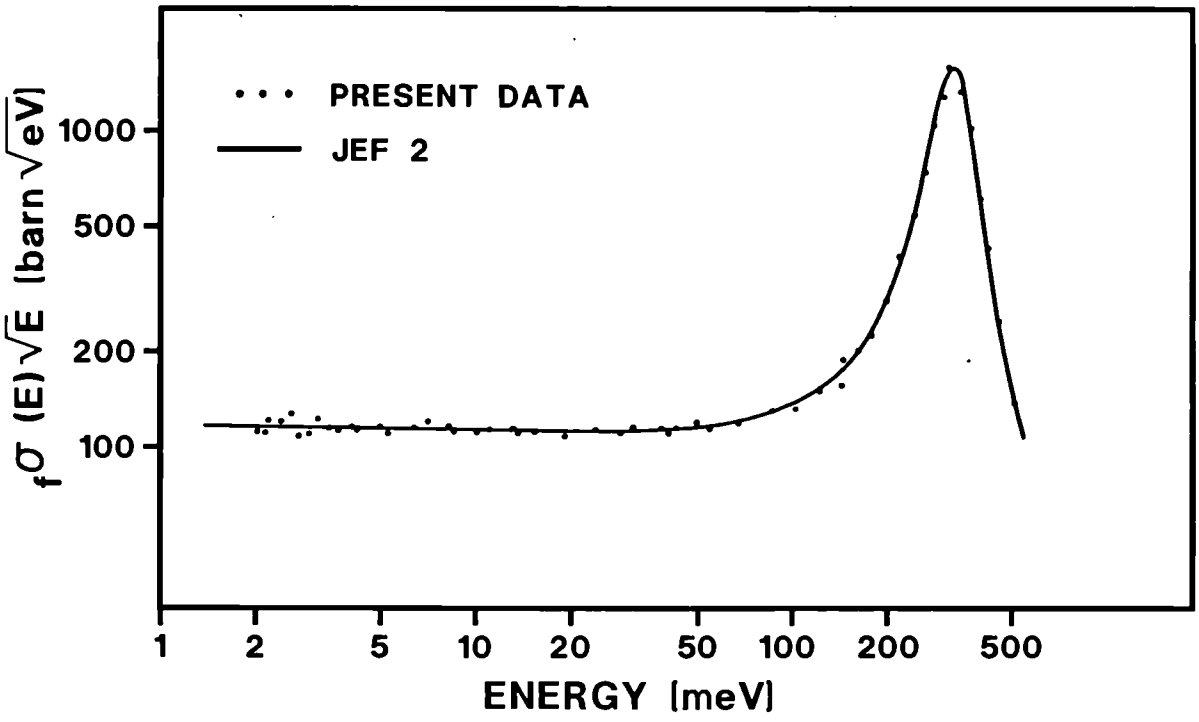


Fig. 6. *Comparison of the present ^{239}Pu(n,f) cross-section data with the JEF-2 data file*

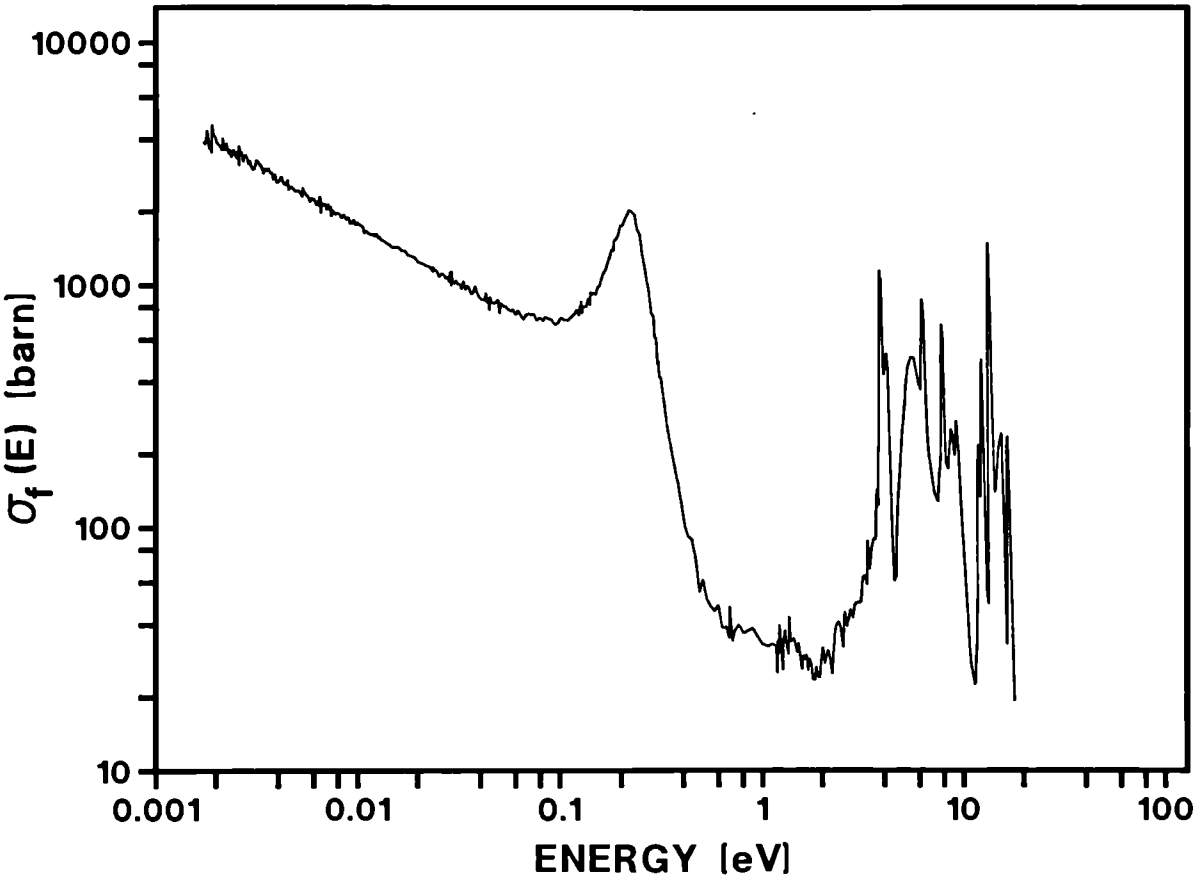


Fig. 7. *The ^{241}Pu(n,f) cross section from 2 meV up to 20 eV*

From the present σ_f(E)-data, a value of 1.041 ± 0.003 was calculated for the Westcott g_f-factor, which is lower than generally adopted. This is due to the fact that the older g_f-values were based on a very poor and discrepant σ_f(E)-data base for < 1 eV.

Development of a Multi-Purpose Charged Particle Detection System

C. Wagemans*, F. Verhaegen*, S. Druyts**, R. Barthélémy, J. Van Gils

During the ternary fission process as well as in some neutron induced reactions, light particles with different charge (Z) are emitted.
To investigate these phenomena, a detection system has been developed consisting of two gridded gas-flow ionization chambers and a 20 cm^2 large, 1000 µm thick surface barrier detector.
Using only one gridded ionization chamber, the detector can be applied e.g. to the study of (n,p) and (n,α)-reactions as a function of the incident neutron energy. As an extreme case, the 0.6 MeV protons produced in the ^{35}Cl(n,p) reaction have been studied.
By combining one ionization chamber with the surface barrier detector, a ΔE-E telescope is formed enabling the separation of $Z = 1$ and $Z = 2$ particles.
By activating the second ionization chamber finally, a F- detector is obtained, enabling a correlation of the charge-identified ternary particles with one of the accompanying fission fragments (F).

Capture to Fission Ratio, α, of ^{235}U

H. Weigmann, J.A. Wartena, C. Bürkholz

The detailed energy dependence of neutron cross sections and related parameters of fissile nuclei for sub-thermal neutron energies has recently found considerable attention because of its effect on the temperature coefficient of reactivity of thermal reactors.
Among others, measurements of η, the number of fission neutrons emitted per neutron absorbed, of ^{235}U, have shown a decrease with decreasing neutron energy in the sub-thermal region, in rough agreement with conclusions drawn from integral experiments.

---

* Rijksuniversiteit Gent, Belgium
** EC Fellow

The measured effect on eta is small, about 2 %, which is only about twice the experimental uncertainties. Furthermore, it has been argued that the apparent variation of η might have been produced by a solid state effect - coherent backscattering of the incident neutrons from the metallic sample used. Therefore, an independent verification is desirable.

Under the assumption that the average number of neutrons per fission, $\bar{\nu}$, is constant, the decrease of eta implies an increase of the capture to fission ratio, α, by a much larger fraction. Therefore, a measurement of α, although experimentally more difficult, might be as sensitive to the effect under investigation.

We have set up an experiment to measure α of ^{235}U by measuring specific low energy capture γ rays and prompt fission γ rays with a germanium detector. The liquid methane moderator of GELINA is used as a pulsed source of sub-thermal neutrons, and the neutron energy is determined by time-of-flight. The method requires that the relative yields of the measured γ rays per capture and fission event, respectively, do not vary over the energy range considered.

A series of measurements has been performed during a three weeks period when GELINA was operated with the liquid methane moderator. Analysis of the data is in progress.

Neutron Data of Structural Materials

The ^{56}Fe 1.15 keV Resonance Task Force

F. Corvi, G. Fioni*, A. Mauri**, K. Athanasopoulos

A point not sufficiently clarified in the previous works on the weighting method to be applied to neutron capture detectors[1] was the dependence of the weighting function on the sample thickness. To investigate this effect, we performed a series of measurements of the 1.15 keV ^{56}Fe resonance normalized to capture in gold for a variety of iron samples. The thickness of the reference gold sample was 0.1 mm and normalization has been done on top of the 4.9 eV resonance according to the saturated resonance method. Two different detector arrangements normally used in neutron capture were studied : if θ is the angle between the neutron beam direction and the axis of the cylindrical C_6D_6 scintillators, the first consisted of two detectors at $\theta = 90°$ and the second of four detectors at $\theta = 125°$.

---

* EC Fellow

** National Expert from ENEA, Bologna, Italy

(1) F. Corvi, A. Prevignano, H. Liskien and P.B. Smith, Nucl. Instr. Meth. Phys. Res. A265 (1988) 475

The results, uncorrected for γ-ray and electron self-absorption, are listed in Table 3. For the θ = 90° case, the measured neutron width stays approximately constant and agrees with the transmission value Γ_n = 61.7 ± 0.9 meV up to a thickness of 0.8 g/cm^2 corresponding to a 1 mm metallic sample. Then it decreases steadily.

Samples thicker than 1 mm were used in neutron capture of ^{56}Fe and ^{60}Ni but in these cases the data were normalized to the capture area of the lowest energy resonance of each isotope (E_0 = 1.15 and 2.25 keV, respectively). These parameters had been independently determined. The question was, however, whether the weighting function used is still able to deal correctly with the degraded γ-ray spectral shapes emerging from a thick sample.

Table 3. Values of the neutron width of the 1.15 keV resonance obtained from capture measurements normalized to the 4.9 eV gold resonance

Sample		Γ_n [meV]	
Thickness [g/cm^2]	Chemical/Isot. Composition	90° Geom.	125° Geom.
0.3874	^{56}Fe	60.1 ± 1.7	62.0 ± 1.6
0.7973	^{56}Fe	61.3 ± 1.8	62.7 ± 1.8
1.1847	^{56}Fe	56.2 ± 1.7	
1.987	Fe_2O_3	53.8 ± 1.8	
1.970	)22.7 % Fe))77.3 % Ni	52.3 ± 1.7	
1.826 [a)]	)93.5 % Fe)) 6.5 % Au	63.6 ± 2.0	65.3 ± 2.0
ORNL Transmission		61.7 ± 0.9	

a) Self-calibration sample

This was investigated using a mixed sample of iron and gold : the neutron width obtained by normalizing to capture in the gold contained in the sample was only 5 % larger than the thin-sample value. This is acceptable as the differences in spectrum shapes amongst the various ^{56}Fe resonances are much less than that between ^{56}Fe and gold spectra. Tests for θ = 125° give similar results as for θ = 90°. It shows that the weighting function, which was experimentally determined only for the θ = 90° geometry, can also be applied to the θ = 125° case.

Resonance Neutron Capture in ^{60}Ni

G. Fioni*, F. Corvi, A. Mauri**, K. Athanasopoulos

Neutron capture measurements of ^{60}Ni were performed at GELINA in the energy range from 1 to 700 keV. Two metal samples in the form of disks of 8 cm diameter were prepared with nickel enriched to 99.07 % ^{60}Ni : a sample of N = 0.0180 atoms/b and of 90.953 g was used in the high resolution run at a 58.6 m flight path ; a sample of N = 0.0041 atoms/b and 20.51 g was used at 28.4 m distance for calibration purposes and for measurements with energies of up to 100 keV. A new detector set-up was employed, consisting of four C_6D_6 scintillators viewing the sample at an angle of 125° with respect to neutron beam direction. This system was designed to minimize possible systematic errors due to the anisotropy of radiation emitted from resonances with spin larger than J = 1/2.

The data were normalized to the capture area of the 2.25 keV resonance which was measured relative to gold capture at 4.9 eV, using the thin sample. Values of the neutron width obtained at fixed Γ_γ = 0.60 eV, are given in Table 4 for two different detector geometries and are compared with ORNL transmission results. The most recent of these values is in reasonable agreement with capture.

Table 4. ***Values of the neutron width of the 2.25 keV resonance of ^{60}Ni obtained from capture measurements normalized to gold, and from transmission measurements***

Measurement			Sample Thickness [atoms/b]	$g\Gamma_n$ [meV]
Lab-Year	Type	Geometry		
CBNM-'89	Capture	θ = 90°	0.00410	60.2 ± 1.4
CBNM-'90	Capture	θ = 125°	0.00410	62.2 ± 1.4
ORNL-'82	Transmission(1)			53.0
ORNL-'90	Transmission(2)		0.0393, 0.0837	59.3 ± 0.8

* EC Fellow

** National Expert from ENEA, Bologna, Italy

(1) C.P. Perey et al., Phys. Rev. C27 (1983) 2556

(2) F.G. Perey, Private Communication (1990)

Since preliminary γ-spectroscopy measurement had indicated a spin J = 3/2 for the 2.25 keV resonance, the value measured at θ = 125° was chosen to normalize all ^{60}Ni(n,γ) data.

At low energy, capture in ^{60}Ni is dominated by the broad s-wave resonance at 12.42 keV. A fit obtained with the FANAC code for the thin sample run is shown in Fig. 8. Two p-waves sitting on its top are also shown.

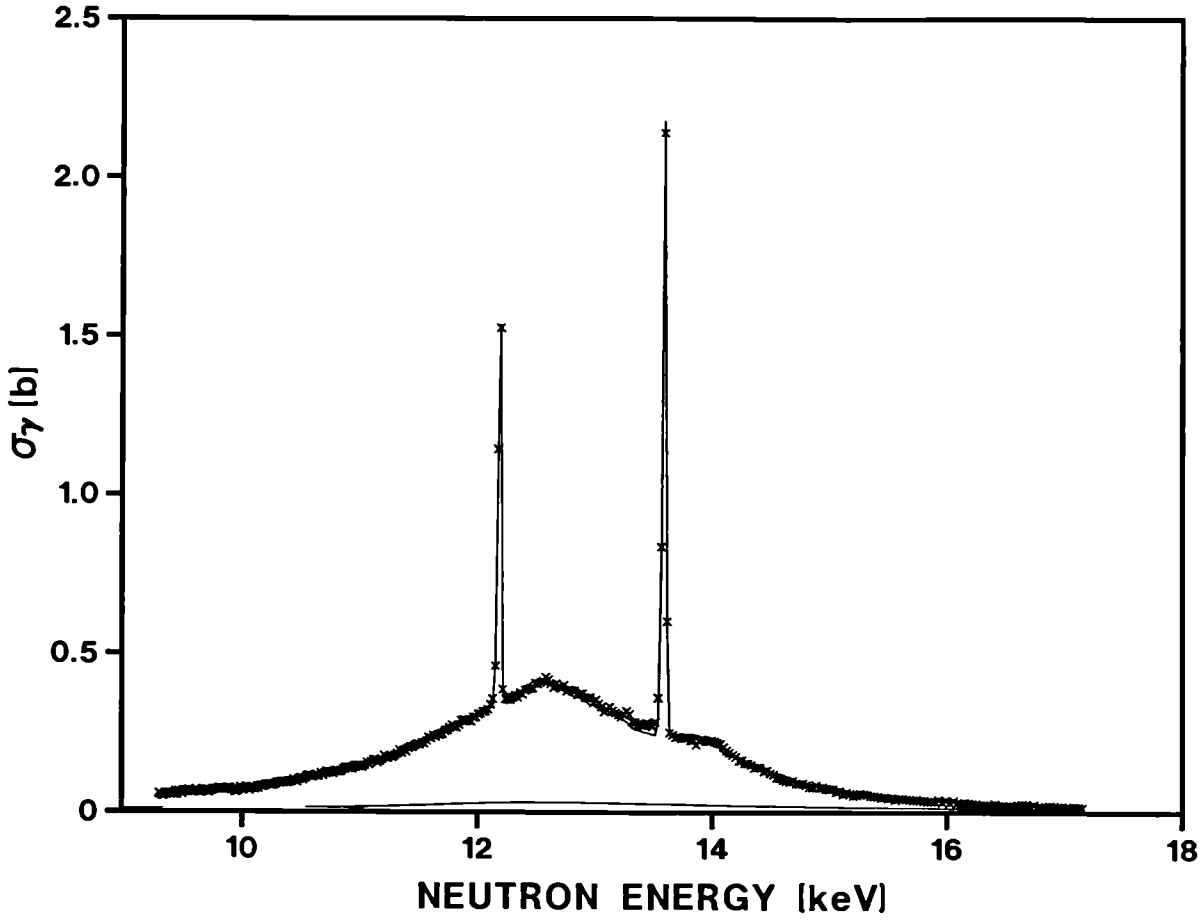


Fig. 8. Shape fit of the triplet of resonances at 12.22, 12.42 and 13.62 keV

Values of the capture width of the 12.42 keV resonance from the present and previous works are compared in Table 5. The two results from the present work are in good agreement in spite of the fact that the multiple scattering correction factor for the thick sample run is of the order of 4 to 5.

The energy region from 1 to 300 keV was analyzed with the area code TACASI and/or with the shape code FANAC. The parameters of 178 resonances were determined, 19 of which seen for the first time. From this data set the ^{60}Ni capture cross section was calculated averaging over a Maxwellian neutron energy distribution centred around various kT values in the keV region. The results, which are of interest for the study of stellar nucleosynthesis, are reported in Table 6.

Table 5. Values of the capture width of the E_0 = 12.42 keV s-wave resonance obtained in the present and in previous works

Author	Γ_γ [eV]
Present work - thin sample	2.33 ± 0.20
Present work - thick sample	2.22 ± 0.22
Stieglitz et al. (1971)	3.3 ± 0.3
Fröhner (1977)	2.73 ± 0.50
Perey (1983)	2.6 ± 0.8
Wisshak (1984)	2.92 ± 0.19

Table 6. Maxwellian-averaged (n,γ) cross sections of ^{60}Ni for various kT values

kT [keV]	$<\sigma_\gamma V>/V_T$ [mb]
12	48.1 ± 3.5
20	34.3 ± 2.0
25	29.4 ± 1.6
30	26.0 ± 1.4
40	21.5 ± 1.1
52	18.2 ± 0.9

Measurement of γ-Ray Decay Spectra from Single Levels Excited by Neutron Capture in ^{53}Cr

C. Coceva*, A. Spits**, A. Mauri***

The Ge telescope detector[1] was used to study the spectrum of gamma rays emitted upon neutron capture in resonances of ^{53}Cr. A 32.4 g sample of isotopically enriched ^{53}Cr and the Ge detector were placed on a 25 m flightpath at the GELINA facility. Time of flight and amplitude of pulses from the central

---

* Scientific Visitor from ENEA, Bologna, Italy
** Scientific Visitor from SCK/CEN,Mol, Belgium
*** National Expert from ENEA, Bologna, Italy
(1) CBNM Annual Report 89, EUR 12615 EN

Ge crystal, satisfying the anticoincidence conditions, are analysed simultaneously, with a resolution Δt = 15 ns and ΔE = 4.8 keV at 6 MeV, respectively.
Neutron resonances can be resolved up to about 110 keV ; new resonances are observed at 24.3, 47.8, 55.2 and 64.8 keV. From a rough examination of the spectra being collected, it can be deduced that the spin of the 8.17 keV resonance is J = 1, in disagreement with the value reported by Allen et al.[1].
In J = 1 resonances a close correlation is apparent between the E1 primary transitions to the ground state and to the first excited state of ^{54}Cr, roughly in the same ratio as the population of these two final states by the (d,p) reaction. This strongly hints to an effective valency capture mechanism.
It is believed that significant spectra can be seperately collected for 8 s-wave and 3 p-wave resonances below 30 keV. At neutron energies between 30 and 70 keV, an average gamma spectrum can be obtained over 15 p-wave resonances. The measurements are still in progress.

Transmission measurements of ^{58}Ni, ^{60}Ni and ^{61}Ni

A. Brusegan, C. Van der Vorst

The total cross sections of ^{58}Ni, ^{60}Ni and ^{61}Ni have been measured. Three independent high resolution transmission runs, one for each isotope, have been performed at GELINA running with 1 ns burst width.
The sample thicknesses and enrichments were 0.044 atoms/b (99.927 %), 0.074 at/b (99.07 %) and 0.019 atoms/b(91.78 %) for ^{58}Ni, ^{60}Ni and ^{61}Ni, respectively.
Neutrons were detected at about 49 m flight distance by a 0.5 cm thick sintered $^{10}B_4C$ slab, which was viewed by 4x(10 cm x 5 cm) C_6F_6 liquid scintillators, kept in a 10 cm thick lead screen.
Those detectors have been chosen instead of NaI(Tl) crystals because, in spite of their relatively low efficiency, they show very good timing properties and moreover a quite low sensitivity to neutrons. This last property helps in reducing those energy dependent systematic errors arising from local and not detectable fluctuations in the background, when this is mainly due to neutrons captured in the detectors and scattered by the boron slab itself.
The total cross section was measured in the energy range from 16 eV up the 1 MeV : the extension to low energy, where no resonances of the nickel isotopes are present, allows a consistent determination of the potential scattering radius together with the resonance parameters of the negative energy levels. In order

(1) B.J. Allen, A.R. de L. Musgrove, Neutron Data of Structural Materials for Fast Reactors, Geel, 1977, K.H. Böckhoff (ed.), p. 447

to estimate the systematic error induced by the measurement technique, the background and the data reduction procedure, a measurement of the transmission of a carbon sample (0.143 atoms / b thick), absorbing on average 50 % of the neutron beam, has been added to each.
The carbon cross section is a standard and the comparison to our results states a deviation on the transmission curves of 5 % from 100 up to 700 eV, of 2.5 % above 700 eV and below 1 keV and of 1.3 % up to 600 keV.

Development of a Li-Glass Detector for Neutron Transmission Experiments
A.Brusegan, C. Van der Vorst

In neutron time of flight spectroscopy, when the detector needs to register only the arrival time of the neutron, the $^{6}Li(n,\alpha)t$ reaction is probably as popular as the $^{10}B(n,\alpha\gamma)^{7}Li^{*}$ reaction.
In the ^{10}B reaction, the detection of the 478 keV gamma ray, emitted by the excited ^{7}Li nucleus, demands at least 2 relatively large, fast scintillators having moreover a low sensitivity to neutrons, which are scattered mainly by the boron itself.
Below a few hundred keV neutron energy, a lithium-glass scintillator may simplify the detection set-up, facilitate to solve stability problems, even keeping good detection efficiency and good time and amplitude resolution.
The lithium detector (NE912, 7.5 % enriched in ^{6}Li, low background), consists of a glass cylinder, with a diameter of 15 cm and a thickness of 0.6 cm, kept in the equatorial plane of an aluminium sphere of 20 cm diameter and 0.06 cm thickness.
The internal surface of the sphere is coated with barium sulphate. The circular surface of the glass is placed orthogonally to the neutron beam and to the photocathode of a 12.5 cm photomultiplier (EMI 9823 KQB, boron free quartz window),which is positioned in the aluminium sphere surface (Fig.9).
Fig. 10 shows the overall detector response to neutrons with energies above 30 eV in the moderated GELINA beam. It is the measured convolution of the actual Linac neutron flux with

a) the intrinsic efficiency of the detector;
b) the change of the total energy available to the reaction with the change of the neutron kinetic energy;
c) non linearity effects in the light output associated with high energy neutrons;
d) gamma-ray and neutron background.

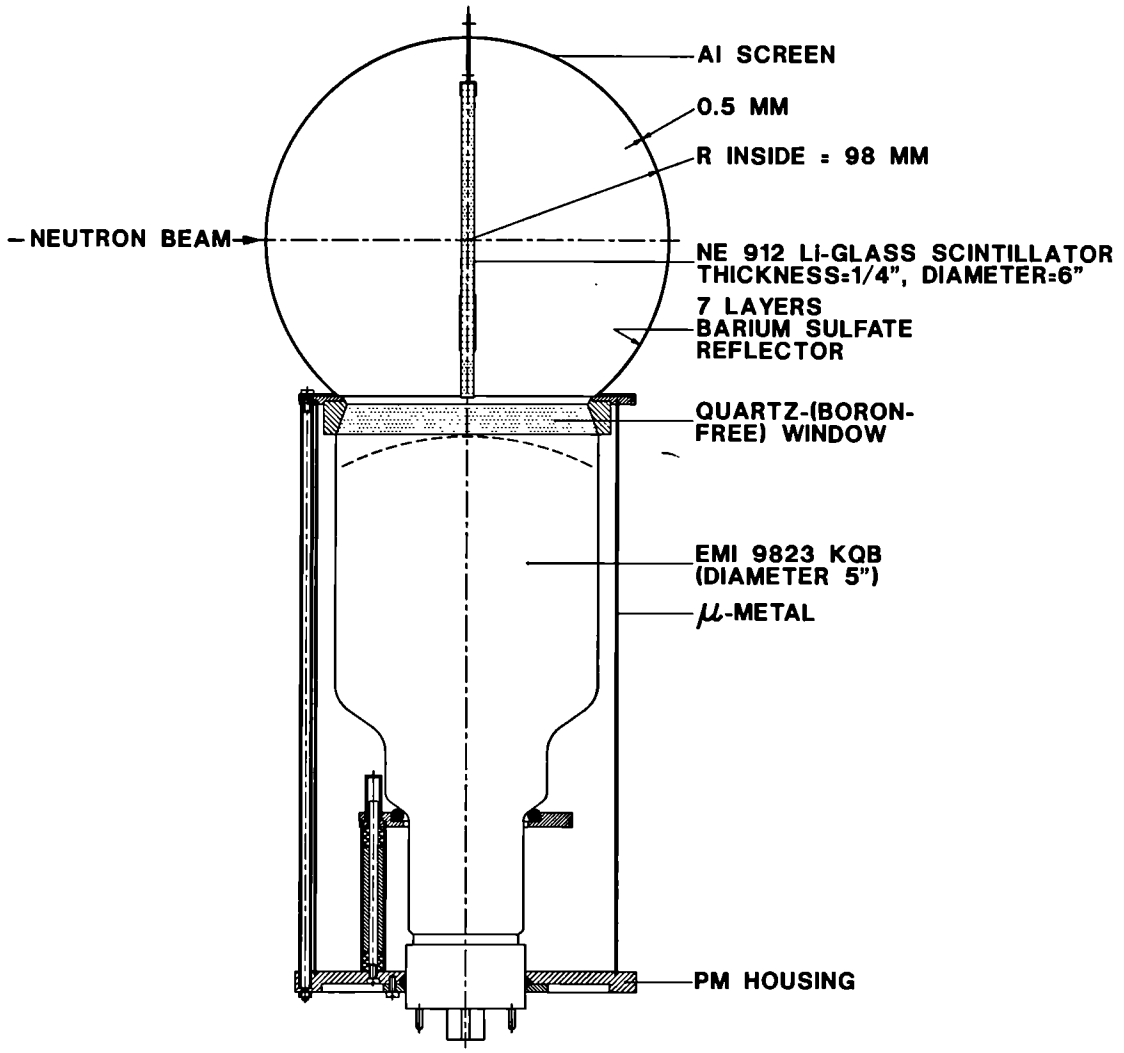


Fig. 9. Longitudinal cross section of detector showing the lithium-glass, the aluminium sphere, the photomultiplier and its housing

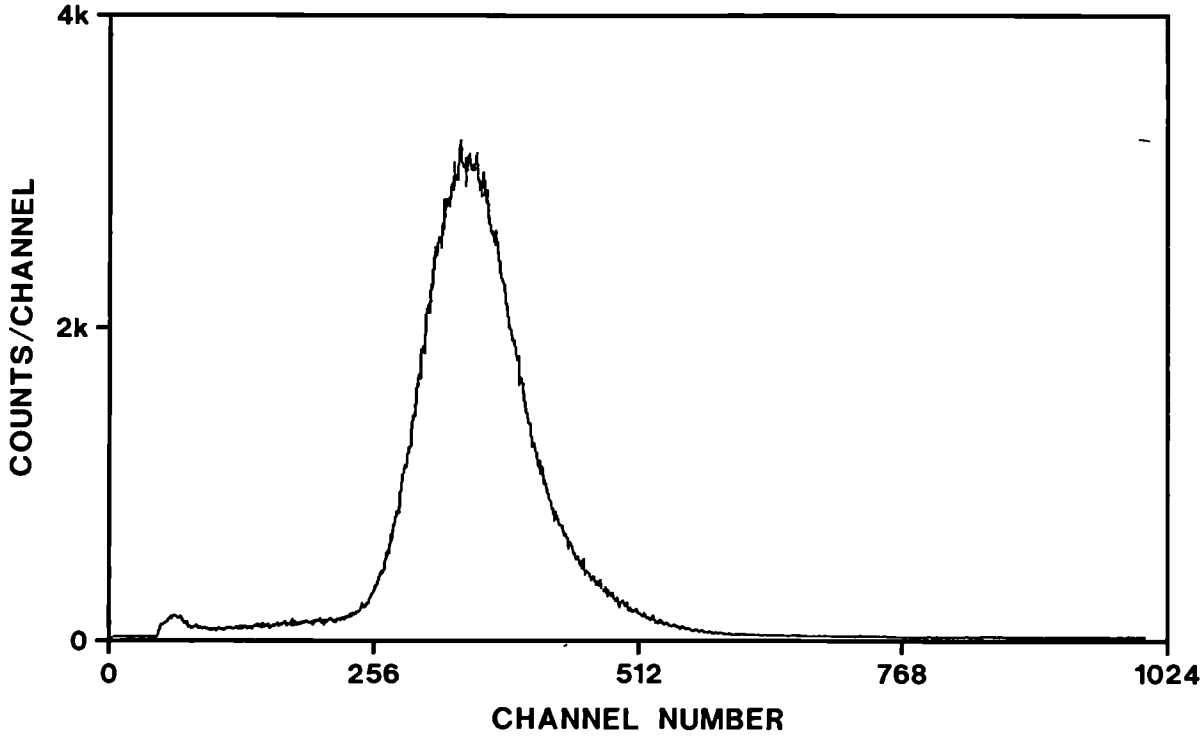


Fig. 10. Pulse height of NE912 in the moderated GELINA beam

The pulse height resolution is 29 %. The time resolution of the detector is about 3 ns, when measured on the spread of the attenuated gamma-ray flash and with 1 ns beam burst width.
Multiple scattering effects are of the order of 5 % and the total count rate at 100 m flight distance is about the same as that measured with a 0.5 cm thick sintered $^{10}B_4C$ slab viewed by the C_6F_6 liquid scintillators placed at 50 m flight distance.

High Resolution Transmission Measurements for Sc and Sc+Mn* Thick Composite Filter

A. Brusegan, C. Van der Vorst

Intense filtered neutron reactor beams, applied in the fields of neutron physics, health physics, dosimetry and radiobiology, demand a modest resolution, but can tolerate only a low contribution from neutrons outside the energy range of interest.
Large amounts of scandium remove practically all the neutrons from the impinging beam except in 'windows' where the total cross section approaches zero, i.e. where a strong resonance-potential scattering interference is present.
Neutron transmission measurements on thick scandium and scandium + manganese filters have been carried out at the 50 m flight path station of GELINA (1 ns burst width).
The measurements covered the neutron energy range from 15 eV up to 0.5 MeV.
The detection system consisted in a 0.5 cm thick $^{10}B_4C$ slab viewed by 4x(10 cm x 7.5 cm) NaI(Tl) scintillators.
Transmission data have been obtained for the scandium and for the scandium (32 cm thick) + manganese (1,5 mm thick) composite filters.
The scandium + manganese attenuates the 2 keV main line of the scandium filter, but does hardly effect other lines in the spectrum.
The present transmission experiment allows a better and higher resolution description of the neutron spectrum effectively transmitted by the scandium and scandium + manganese filters, i.e. it defines possible systematic errors associated with the filter 'difference' technique.

---

* Filter supplied by PTB, Braunschweig, Germany

NUCLEAR DATA FOR FUSION TECHNOLOGY

Double-Differential Neutron-Emission Cross-Sections of ^{9}Be

F.Poortmans*, J.Wartena, H. Weigmann, C. Bürkholz

Because of its large (n,2n) cross section beryllium is a preferred candidate material for future fusion reactor blankets. For the calculation of neutron transport in beryllium containing blankets, a detailed knowledge of the neutron-emission cross section of beryllium, including the secondary neutron energy and angular distributions, is required. Therefore measurements of the double-differential neutron-emission cross sections of ^{9}Be have been performed. The experimental methods applied in these measurements have been previously described [1]. Extensive measurement series had been conducted in 1986 and 1989, including measurements on ^{12}C which serve for normalization of the data obtained for ^{9}Be, to the differential elastic-scattering cross section of ^{12}C below 2 MeV.

The analysis of the data from both experiments is almost complete. Apart from dead-time and background corrections, the analysis mainly includes the unfolding of the experimental pulse-height distributions to yield secondary neutron energy spectra. These have to be corrected for self screening and multiple scattering in the sample. The Monte-Carlo code written earlier[1] for the multiple scattering corrections has been extended to include the (n,2n) reactions ; an approximate description of the secondary neutron energy distributions associated with these processes is used as an input to the code.

The results will be available in the form of double-differential neutron-emission cross sections as a function of secondary neutron energy and laboratory emission angle, for incident neutron energies from about 2 to 11 MeV. Above 11 MeV the incident neutron flux drops too quickly to give statistically meaningful results. It is expected that with the uranium-beryllium sandwich target available only recently, the useful energy range can be extended.

As an example of the results obtained, Fig. 11 shows double-differential neutron-emission cross sections for four emission angles and for the incident neutron energy interval from 6.5 to 7.1 MeV.

---

* SCK-CEN, Mol, Belgium

(1) CBNM Annual Progress Report 1988, EUR 12267 EN

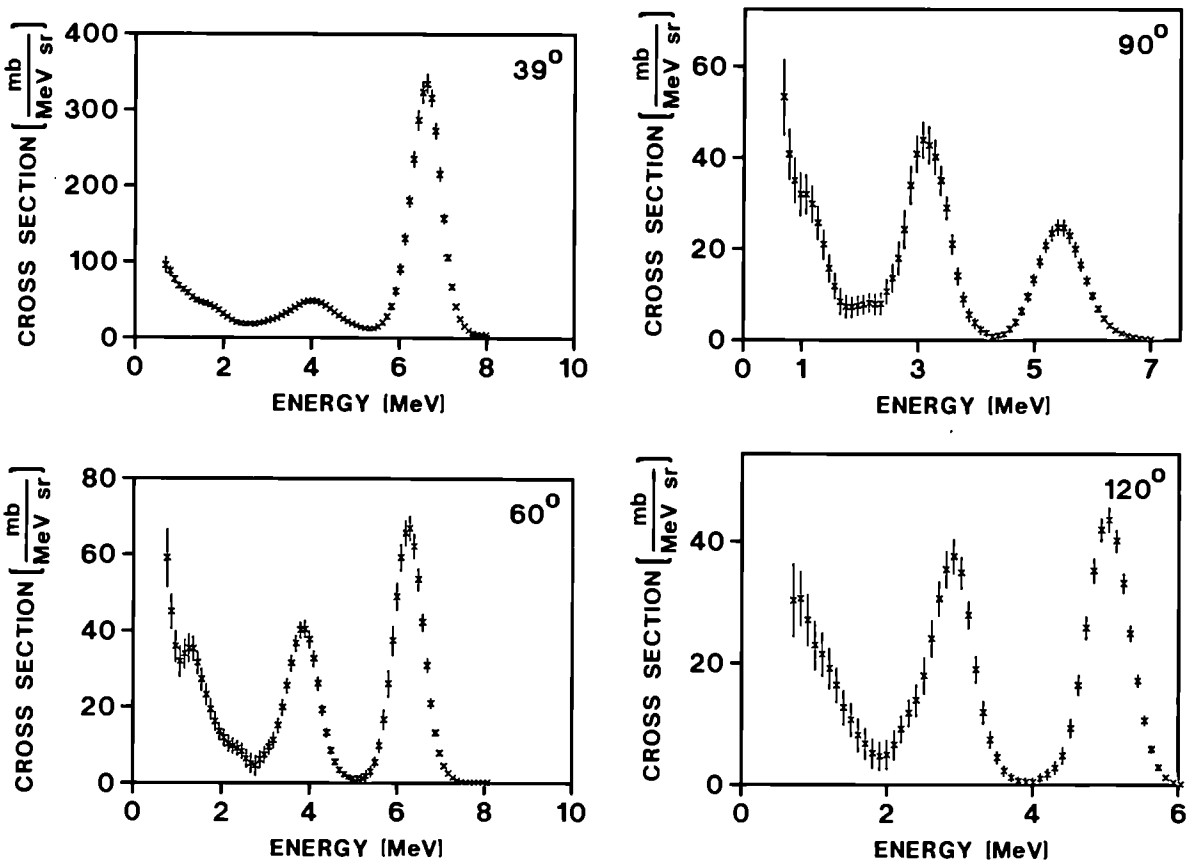


Fig.11. Double-differential neutron-emission cross section of ^{9}Be for incident neutron energies from 6.5 to 7.1 MeV

Excitation Functions of $^{nat}Ti(n,x)^{46,47,48}Sc$ Processes in the Energy Range of 12.5 to 19.6 MeV

N.I. Mollar*, S.M. Qaim*, H. Liskien, W. Widera

Titanium is an important component of structural materials used in fission and future fusion reactors. Cross sections were measured for the $^{nat}Ti(n,x)^{46,47,48}Sc$ processes over the neutron energy range of 12.5 to 19.6 MeV. Use was made of the activation technique in combination with high resolution γ-ray spectroscopy. The neutron fluence rates were determined using two independent methods, viz. proton recoil and $^{27}Al(n,\alpha)^{24}Na$ monitor reaction. The present results strenghten the data base above 15 MeV, especially for the formation of ^{47}Sc.

* KFA, Jülich, Germany

A Scintillator for Use in a Light Ion Telescope with Improved Time- and Energy-Resolution

E. Wattecamps, G. Rollin

Neutron induced light ion particle-production cross-section data are needed in fusion reactor design. A telescope was built at the Van de Graaff laboratory to measure such data. The telescope consists of three detectors in line : two multiwire parallel plate avalanche counters (MWPPAC) of circular area with 5 cm diameter, and an hexagonal pilot-U scintillator of approximately 6 cm side length and 1 mm thickness which is located at 38 cm distance from the first MWPPAC[1]. Two opposite scintillator sides are viewed by two photomultipliers (Fig. 12a and b).

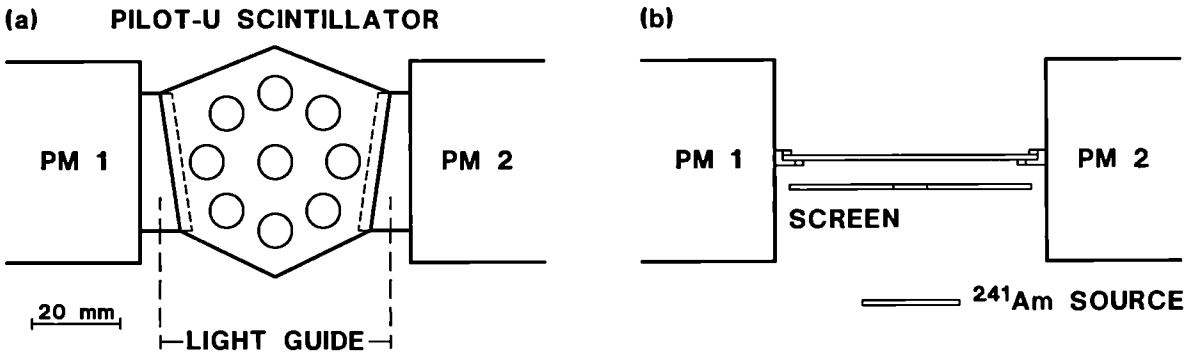


Fig. 12. Schematic view of the scintillator mounting : (a) vertical section, (b) horizontal section

In order to improve the energy- and time-resolution of the pilot-U scintillator a systematic investigation of time- and energy-response behaviour was made. Energy- and time-spectra of α-particles from a small ^{241}Am source have been measured for the entire scintillator as well as for nine spots of 9 mm diameter across the surface of the scintillator.

Typical results of the measurements of the energy, of the energy-resolution and of the start-stop time distribution for the nine spots across the surface are illustrated in Fig.13a, b and c.

---

(1) CBNM Annual Report 89, EUR 12615 EN

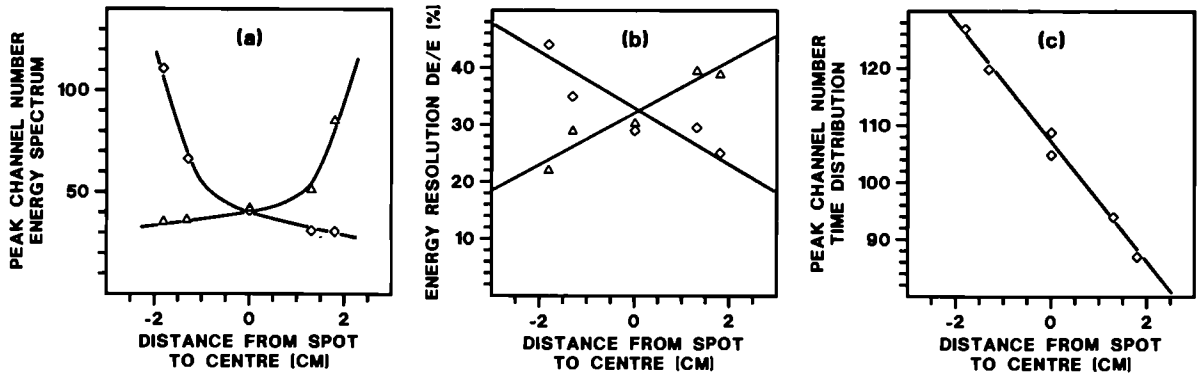


Fig. 13. ***Energy (a), energy resolution (b) and start-stop time (c) versus the location of the irradiation on the scintillator***

On the basis of the light attenuation and the time delay across the scintillator determined experimentally, a correction programme COMMND2. EETP has been written to correct the energy and time information of each event.
A detailed description of the experiment and correction procedure will be published. The energy- and time-spectra for total surface irradiation before and after correction are shown in Fig. 14a and b. It can be seen that the energy resolution improves from 31 % to 25 % and that the time resolution reduces from 988 to 355 ps. This result is quite satisfactory having in mind that the burst width of the accelerator is about the same value.

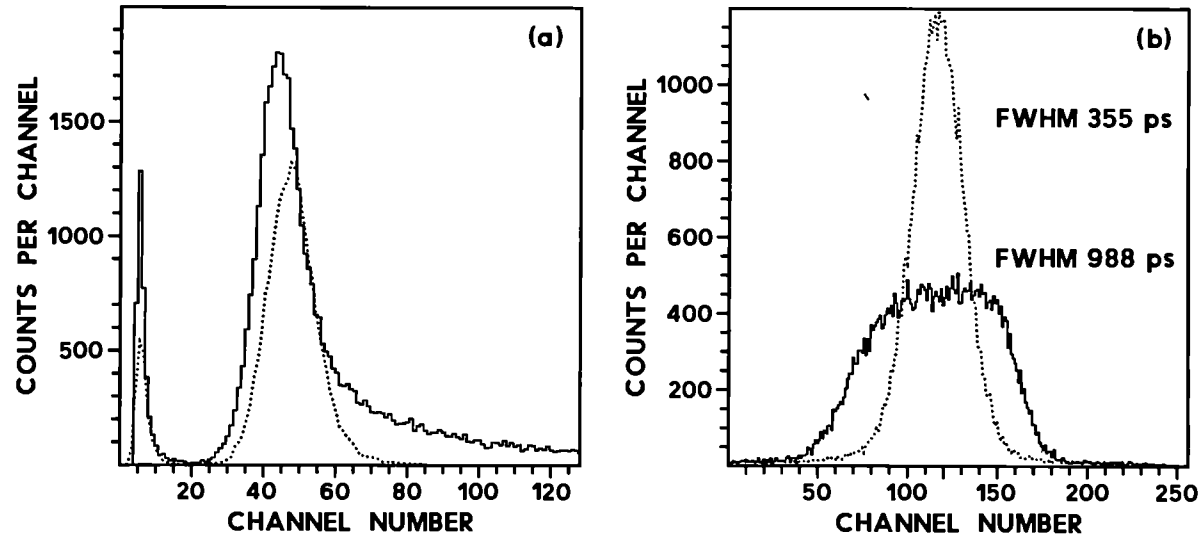


Fig. 14. ***(a) energy spectrum and (b) time spectrum for total scintillator irradiation ; full line : uncorrected, dotted line : corrected.***

NUCLEAR METROLOGY

RADIONUCLIDE METROLOGY

Response of Silicon Detectors to Electrons, Deuterons and Alpha Particles

P. Bauer*, G. Bortels

The response of silicon detectors to electrons from the decay of ^{109}Cd, ^{233}Pa and ^{137}Cs, to deuterons in the energy range 35 to 440 keV from a Van de Graaff accelerator and to alpha particles from a mixed ^{239}Pu, ^{241}Am, ^{244}Cm source has been measured. The results were found to be in agreement with the model proposed by Lennard at al. (1986)[(1)] which describes the nonlinear detector response in particular in the case of light ions. For electrons, the non-linearity was found to be negligible within experimental uncertainties. For deuterons, the non-linearity observed in Rutherford- backscattering measurements at low energies was used to accurately measure the window thickness of the detectors. For alpha particles in silicon it has been confirmed that the energy required to create an electron-hole pair depends on the stopping power. The non-linearity parameter in the Lennard model has been measured. This offers a reliable way of measuring the energy corresponding to peaks in alpha-particle spectra from a calibrated semiconductor system. The work was presented at the 7th Symposium on Radiation Measurements and Applications, Ann Arbor.

Low-Energy X-Ray Standards

B. Denecke, C. Ballaux**, W. Bambynek, G. Grosse, A. Srivastava***, U. Wätjen

After the successful demonstration of the use of the indirectly excited fluorescence sources for the efficiency calibration of a Si(Li) detector the production of ten sets of fluorescence layers was ordered and is nearly finished. In this type of reference sources many different fluorescence layers can be used with the same high-activity ^{55}Fe excitation source. The procedure to produce such ^{55}Fe sources as deposits on thin copper rings has been established using an inactive iron-chloride solution. The fabrication of the active sources is in progress. In addition, the prototype system for the counting-gas regulation was rebuilt using better equipment for the gas-flow control and the pressure regulation.

---

* Scientific Visitor from the Johannes Kepler Universität, Linz, Austria

** SCK/CEN, Mol, now at AIB Vinçotte, Brussels, Belgium

*** Scientific visitor from the University of Mainz, Germany

(1) W. Lennard, H. Geissel, K.B. Winterbon, D. Phillips, T.K. Alexander and J.S. Forster, Nucl. Instr. and Meth. A248 (1986) 460

Efficiency Calibration of Photon Detectors

T. Altzitzoglou, L. Delfosse

An efficiency re-calibration of the photon detectors currently in use for X- and gamma-ray measurements is in progress. The spectra analysis is performed using the program RETEOH and the data reduction and analysis are done by custom-made spreadsheets. The fitting of the final results is made using spline or polynomial fitting routines.

A High-Purity-Germanium Detector System for the Measurement of Low-Level Radioactivity in Environmental Samples

D. Mouchel

A high-purity-germanium detector system designed to measure low-level radiation has been set up 25 m above sea level in an area where the exposure rate is about 60 nSv/h. It is operated in a normal laboratory without special provisions with respect to the building construction. Nevertheless, by careful selection of the materials for the detector assembly and the shielding with respect to their very low radioactivity, the background could be reduced by a factor of 70. The system has been described in a seminar lecture at the Second International Summer School on Low-Level Measurements of Man-Made Radionuclides in the Environment, La Rábida.
At present, the detector system is being calibrated with suitable radionuclides emitting gamma rays in the energy range 50 to 2000 keV. Well-defined matrices have been prepared in Marinelli beakers of the type IEC-450 and spiked with standard sources at various environmental radioactivity levels. Measurements of various organic samples are in progress.

Measurement of K-Shell Fluorescence Yields

A. Solé*, B. Denecke, D. Mouchel, W. Bambynek

Following a review on K-shell fluorescence yields for elements with atomic numbers between 20 and 30, the feasibility to measure fluorescence yields by means of (gamma-ray)-(X-ray) coincidence methods has been investigated. It is estimated that these methods will yield results with an accuracy below 2 % [1]. Sources of ^{58}Co were prepared by evaporation in vacuum to minimize attenuation of the X rays in the source. The source purity was checked by

---

* EC fellow

(1) A. Solé, CBNM Internal Report GE/R/RN/11/90

gamma- and X-ray spectrometry, and the source homogeneity by auto-radiography. A gas-flow proportional-counter system for the measurement of the X rays and a NaI(Tl) scintillation detector to measure the gamma rays were set up. First measurements with ^{58}Co were performed. Data evaluation is in progress.

Metrology of ^{192}Ir Brachytherapy Sources

D.F.G. Reher, T. Altzitzoglou, B. Denecke, E. De Roost

On request of the Belgian Association of Hospital Physicists and in the frame of the EUROMET project Nr. 219 an international co-operation was started to provide air-kerma rates and activity standards of ^{192}Ir wires and their traceability to national and international standards, and to develop a scanning device for the assessment of the longitudinal homogeneity of such ^{192}Ir wires used in radiotherapy.

During the reporting period, an intercomparison of measurements of eight ^{192}Ir wires of 10 mm length was organized. NPL, RIVM, and RUG did air-kerma rate measurements, CBNM and NPL did ionization-chamber measurements, and CBNM additionally gamma-ray spectrometry.

Two solutions of ^{192}Ir were standardized and used for calibration of an ionization chamber and a Ge detector. Ampoules of these solutions were exchanged between NPL and CBNM. Each of the two laboratories sent a set of ampoules to the BIPM as input to SIR.

A study for the development of a ^{192}Ir wire scanner started at CBNM in collaboration with the Belgian Association of Hospital Physicists. NPL made relative efficiency measurements using external sources and an ionization chamber.

A study (CBNM and NPL) about the decay scheme of ^{192}Ir revealed that the accuracy of the half life, the gamma-ray emission probabilities and some other decay-scheme data need to be improved.

The air-kerma rate measurements and the activity standardization have shown that the achieved accuracies need to be improved. Furthermore an effort to improve the decay-scheme parameters is necessary.

PROJECT 2 : REFERENCE MATERIALS

Introduction

R. Lesser

In the current multiannual JRC Research Programme 1988 - 1991 the project Reference Materials is divided into a number of sub-projects. Nuclear and non-nuclear activities are concerned.
The main objectives of the **nuclear activities** are :

- to provide **high quality reference** materials required for accurate analytical measurements in all fields of the nuclear fuel cycle, including safeguards,

- to secure **European independence** in the above field of high public sensitiveness,

- to facilitate **progress and harmonization of analytical measurements** by adequate development activities.

Non-nuclear activities are guided by the idea :

- to make **use of nuclear measurement experience** and preparation techniques for other applications.

In this Annual Report contributions related to reference materials are also included in the chapters on Exploratory Research, Scientific and Technical Support to the Commission, and Work for Third Parties. This is due to the fact that some activities have multiple aims or the results find different applications.

Two achievements may be underlined :

- Good progress was made in the development and preparation of **dried nitrate solid spikes** for the accurate assay of uranium and plutonium in undiluted fuel **reprocessing input solution** by IDMS. First applications in collaboration with Safeguards authorities and reprocessing plants led to very promising results.

- By intensive collaboration between scientists from the University of Sussex (UK), NIST (USA), the Scottish Universities Research and Reactor Center, Glasgow (UK), and CBNM it was possible to perform new measurements on the **free neutron lifetime** which resulted in a value of (893.6 ± 5.3) sec. CBNM's contribution consisted in the extremely careful preparation and very accurate characterization of sets of ^{10}B and ^{6}Li deposits on silicon wafers.

Summarized information on available nuclear reference and spike materials are given in Table 7.

Table 7. Nuclear and Isotopic Reference Materials presently available at CBNM

Code	Material	Application	Certified Quantities and Uncertainties (95% confidence level)	Certificate	Single Unit Size	Price 1991 ECU/unit
EC-NRM 101	Uranium metal (0.5 - 1g)	element analysis	999.85 ± 0.05 g U·kg^{-1}	EC	1 g	50
EC-NRM 110	Uranium dioxide pellets (1g)		881.34 ± 0.13 g U·kg^{-1}	EC	25 g	200
CBNM-106	Uranium dioxide pellets (10g)		881.43 ± 0.24 g U·kg^{-1}	CBNM	150 g	180
EC-NRM 210	Plutonium dioxide powder (5 g)		880.26 ± 0.44 g Pu·kg^{-1}	EC	5 g	5000
EC-NRM 171	Set of 5 Al cans with U_3O_8 of different $^{235}U/U$ abundances	$^{235}U/U$ abundance assay by gamma spectrometry	0.3206 ± 0.0002 atom % ^{235}U 0.7209 ± 0.0005 atom % ^{235}U 1.9664 ± 0.0014 atom % ^{235}U 2.9857 ± 0.0021 atom % ^{235}U 4.5168 ± 0.0032 atom % ^{235}U	EC	5 x 200 g	5500
CBNM-271	Set of 3 stainless steel cans with PuO_2 pellets of different Pu isotopic composition and ^{241}Am concentration	Pu isotope abundance/ratio and ^{241}Am concentration assay by gamma spectrometry	84.3985 ± 0.0084 atom % ^{239}Pu 73.4248 ± 0.0098 atom % ^{239}Pu 62.6562 ± 0.028 atom % ^{239}Pu and corresponding values for ^{238}Pu, ^{240}Pu, ^{241}Pu, ^{242}Pu and ^{241}Am.	CBNM	3 x 6.65 g	5100
CBNM-011	Boric acid	isotope analysis	19.824 ± 0.020 atom % ^{10}B 80.176 ± 0.020 atom % ^{11}B	CBNM	1 g	145
CBNM-015	Lithium carbonate		95.610 ± 0.025 atom % ^{6}Li 4.390 ± 0.025 atom % ^{7}Li		50 mg	145
CBNM-016	Lithium carbonate		7.525 ± 0.029 atom % ^{6}Li 92.475 ± 0.029 atom % ^{7}Li		1 g	145
CBNM-017	Silicon		92.233 ± 0.014 atom % ^{28}Si 4.675 ± 0.011 atom % ^{29}Si 3.092 ± 0.008 atom % ^{30}Si		50 mg	135
CBNM-018	Silicon dioxide		92.214 ± 0.014 atom % ^{28}Si 4.688 ± 0.011 atom % ^{29}Si 3.098 ± 0.008 atom % ^{30}Si		5 g	440
CBNM-021			0.43842 ± 0.00022 atom % ^{235}U			
CBNM-022			0.72009 ± 0.00036 atom % ^{235}U			
CBNM-023	Uranium hexafluoride		3.2743 ± 0.0016 atom % ^{235}U		20 g	530
CBNM-024			5.0506 ± 0.0024 atom % ^{235}U			
CBNM-040a	Spike solution; $^{233}U/U$: 0.980	element assay by isotope dilution mass spectrometry (IDMS)	0.9382 ± 0.0026 mg$^{233}U.g^{-1}$	CBNM	10 ml	260
CBNM-042a	Spike solution; $^{244}Pu/Pu$: 0.979		0.9150 ± 0.0019 µg$^{244}Pu \cdot g^{-1}$		10 ml	
CBNM-043	Spike solution; $^{242}Pu/Pu$: 0.88		44.25 ± 0.13 ng$^{242}Pu \cdot g^{-1}$		10 ml	
CBNM-044	Spike solution; $^{242}Pu/Pu$: 0.999		9.213 ± 0.014 µg$^{242}Pu \cdot g^{-1}$		10 ml	
CBNM-049	Spike solution; $^{242}Pu/Pu$: 0.999		91.23 ± 0.18 µg$^{242}Pu \cdot g^{-1}$		10 ml	
CBNM-050	Spike solution; $^{235}U/U$: 0.999		0.99996 ± 0.00025 µg$^{235}U.g^{-1}$		10 ml	
CBNM-060	Spike solution; $^{230}Th/Th$: 0.9985		40.38 ± 0.30 µg$^{230}Th \cdot g^{-1}$		10 ml	
CBNM-610	Spike solution; $^{10}B/B$: 0.949		36.8778 ± 0.0088 µg $^{10}B.g^{-1}$		5 ml	
CBNM-611	Spike solution; $^{11}B/B$: 0.802		44.31 ± 0.44 µg $^{11}B.g^{-1}$		5 ml	
CBNM-615	Spike solution; $^{6}Li/Li$: 0.956		23.18 ± 0.16 µg $^{6}Li.g^{-1}$		5 ml	
CBNM-618	Spike solution; $^{87}Rb/Rb$: 0.9799		9.743 ± 0.015 µg $^{87}Rb.g^{-1}$		5 ml	

Table 7 (continued).

Code	Material	Application	Certified Quantities and Uncertainties (95 % confidence level)	Certificate	Single Unit Size	Price 1991 ECU/unit
CBNM-1027a	Solid spike (dried); $^{235}U/U$: 0.196 $^{239}Pu/Pu$: 0.971	element assay by IDMS	For each individual unit : amount of spike isotope, certified to ≤ 0.1%	CBNM	50 or 100 mg (U + Pu)	100
CBNM-047a	Dry nitrate; $^{239/244}Pu$ mixture	mass spectrometer linearity or isotopic abundance ratios calibration	$^{239}Pu/^{244}Pu$: 1.2984 ± 0.0025	CBNM	0.15 mg	480
CBNM-072	Set of 15 solutions, different $^{233}U/^{235}U$, equal $^{235}U/^{238}U$ atomic ratios		various $^{233}U/^{235}U$ and $^{235}U/^{238}U$ ratios ±0.03% of values	CBNM	15 x 1 ml	850
CBNM-199	Uranyl nitrate		$^{233}U/^{238}U$: 1.00001 ± 0.00030 $^{235}U/^{238}U$: 1.00015 ± 0.00020	CBNM EC in preparation	5 ml	520
EC-NRM 501	^{238}Uranium dioxide spheres 0.5 and 1.0 mm diameter,		879.4 ± 2.8 g U · kg^{-1} 10.4 ± 0.5 mg ^{235}U · kg^{-1} U 999.9896 ± 0.0005 g ^{238}U · kg^{-1} U	EC	100 mg (0.5 mm ⌀) 200 mg (1.0 mm ⌀)	155 155
CBNM 502	^{237}Neptunium dioxide spheres 0.5 and 0.8 mm diameter		873 ± 7 g Np ·kg^{-1}	CBNM EC in preparation	100 mg (0.5 mm ⌀) 200 mg (0.8 mm ⌀)	280 280
EC-NRM 521	Nickel 0.1 mm foil 0.5 mm ⌀ wire		< 0.1 mg Co · kg^{-1}	EC	100 cm^2 1 m	230 125
CBNM 522	Copper 0.1 mm foil 1 mm foil 0.5 mm or 1 mm ⌀ wire	reactor neutron dosimetry	< 0.05 mg Co·kg^{-1} 0.95 ± 0.04 mg Ag·kg^{-1}	CBNM EC in preparation	100 cm2 20 cm2 1 m	190 190 100
EC-NRM 523	Aluminium 0.1 mm foil 1 mm foil 1 mm ⌀ wire		< 0.1 mg Na·kg^{-1}	EC	100 cm^2 20 cm^2 1 m	155 155 100
CBNM-524	Iron 0.1 mm foil 0.5 mm ⌀ wire		< 0.05 mg Co·kg^{-1} < 0.1 mg Mn·kg^{-1}	CBNM EC in preparation	100 cm^2 1 m	155 100
EC-NRM 525	Niobium 20 µm foil 0.1 mm foil 0.5 mm ⌀ wire		19.6 ± 1.8 mg Ta·kg^{-1}	EC	20 cm^2 20 cm^2 1 m	190 125 125
EC-NRM 526	Niobium 20 µm or 0.1 mm foil 0.5 mm ⌀ wire		0.30 ± 0.09 mg Ta·kg^{-1}	EC	20 cm^2 1 m	1100 550
CBNM-527	Al - 0.1% Co 0.5 mm ⌀ wire		1.00 ± 0.02 g Co · kg^{-1}	CBNM	1 m	100
CBNM-528	Al - 1% Co 0.5 mm ⌀ wire		10.0 ± 0.2 g Co · kg^{-1}	CBNM	1 m	100
CBNM-529	Rhodium 50 µm foil		Pt < 10 mg·kg^{-1} Ir 25 mg·kg^{-1}	provisional	20 cm^2	600
CBNM-530	Al - 0.1% Au 0.1 mm foil 0.5 mm or 1 mm ⌀ wire		1.00 ± 0.02 g Au·kg^{-1}	CBNM EC in preparation	100 cm^2 1 m	200 135

ACTINIDE REFERENCE MATERIALS

Elemental Analysis

Analytical Chemistry

Y. Le Duigou, A. Rodríguez, W. Leidert, A. Michiels, M. Bickel

In order to satisfy the needs for plutonium RMs within the Community the characterization of a batch of 250 g of high purity plutonium dioxide has been finalized. In its meeting of 1 February 1990 the Nuclear Certification Group (NCG) gave positive advice on the content of the certification report. Accordingly, the corresponding European certificate for EC-NRM 210 has been issued.

On basis of the results given previously[1] the plutonium mass fraction of (880.26 ± 0.44) $g \cdot kg^{-1}$ is certified. An overall uncertainty of the plutonium mass fraction of an individual one gramme sample was estimated by combining the standard deviation of the plutonium measurements, the standard deviation of the calibration measurements, the standard deviation of the plutonium content of the reference material and the standard deviation of the random variation of the plutonium mass fraction between samples (inhomogeneity) according to the law of error propagation. Information on the origin of the material, the measurements performed, the way in which the reference material must be used and the list of participating laboratories is given on the certificate. The technical and administrative certification processes have been finished, the certification report is published and the material is available for sale.

The certification report of two uranium ores (EC-NRM 113 and EC-NRM 114) has also been submitted to the NCG. The base materials were purchased from CEA with uranium contents certified on the basis of spectrophotometric results from seven laboratories. The NCG considered that the limited amount of additional measurements provide insufficient verification for the spectrometry data. Suggestions were made that a set of verification analyses should be performed to confirm the uranium content in the ores.

The report on the first phase of the interlaboratory comparison exercise for the determination of uranium in pure uranyl nitrate solutions by potentiometric titration, initiated by the ESARDA-LEU Working Group, has been drafted. This exercise is a method specific intercomparison and, therefore, different from the much broader REIMEP programme. It was led through as a precision experiment to be used as a test method to investigate the type of the variability of the data and the difference between the results and the certified

---

(1) CBNM Annual Report 89, EUR 12615 EN

value(s). The statistical treatment included : calculation of the intra-laboratory and the inter-laboratory variances, laboratory means, the repeatability, the reproducibility and comparison of the laboratory means with the certified values. The original data set consisting of 84 observations originating from 14 laboratories comprises statistical outliers for nearly all determinations. All the data coming from two laboratories have been discarded. Figure 15 shows an example for the results.

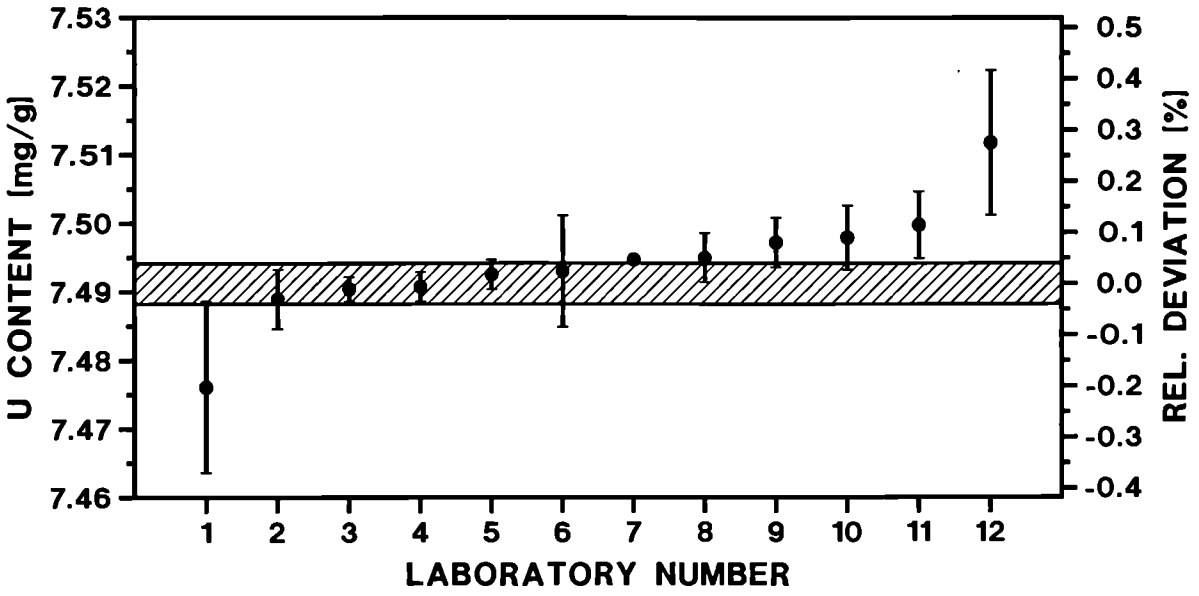


Fig. 15. Distribution of measured uranium concentrations around the certified value

A relative standard deviation of about 0.25 % described very well the total reproducibility variability in the interlaboratory comparison. Results indicate that an experienced laboratory will have an intra-laboratory standard deviation of as low as 0.08 %.

The methodology of the second phase of this ESARDA-LEU initiated experiment has been decided after consultation of other experts. Technical and organisational preparations are in progress.

In the course of the REIMEP programme further measurements have been performed on the **UO_2 powder** by constant potential coulometry leading to a further increase in certification quality. The value for the uranium content was (859.91 ± 0.36) $g \cdot kg^{-1}$. Two other REIMEP exercises, i.e. plutonium-nitrate solution and MOX pellets are in preparation.

A second campaign of **analyses of neptunium** in support of CBNM's activity "Reactor Neutron Dosimetry RM's" was carried out by a potentiometric titration method at the TUI, Karlsruhe. The analytical method has been described previously[1]. Results were slightly different from the first campaign. Experiments have begun for the uranium and **plutonium chemical assay of MOX.**

(1) CBNM Annual Report 89, EUR 12615 EN.

Two independent methods will be used for both elements : potentiometric titration (PT) and constant potential coulometry (CPC). While methods for CPC of uranium and plutonium as well as PT for uranium are already in operation, the following PT method for plutonium has been chosen : plutonium is quantitatively oxidised to plutonium (VI) with silver (II) oxide. The excess of silver (II) is destroyed by adding saturated sulphamic acid solution. The plutonium (VI) is reduced to plutonium (IV) with a small excess of iron (II) and the excess is titrated with standard dichromate solution, the end point being detected potentiometrically.
For the treatment of plutonium containing materials, a specially designed titration facility had to be installed including a weighing, a dissolution and a titration box. It is ready to go into cold test now.
Hot CPC experiments with simulated MOX solutions have started already.

Gamma-Ray Spectrometry
F. Quik, W. Nagel

A project "Determination of the **Uranium Content in Uranium Ore** EC-NRM-113 and 114" was started as the Nuclear Certification Group had indicated that further independent verification analysis would be meaningful.
The method consists in the determination of the ^{235}U abundance of an ore sample with known degree of infinite thickness relative to a quasi infinite thick metallic uranium reference sample applying the enrichment-meter technique.
Due to the fact that the isotopic composition of the uranium in the ore is natural, the uranium content can be determined. Calculations and some preliminary measurements have shown that this can be done with sufficient accuracy (1 % at the 1s confidence level).
The parameters to be known with best possible accuracy are:

- the matrix correction factor of the uranium ore relative to the reference sample,
- the correction factor for differences in the degree of infinite thickness of the used samples.

For that reason the mass attenuation coefficient, μ - valid for the applied measurement geometry - of the ore and the uranium metal were determined experimentally on a well defined additional ore and uranium metal sample for the used gamma-ray line energy.
Preliminary values are:

- μ (uranium ore, 163,4 keV) = $0.1684\ cm \cdot g^{-1} \pm 0.7\ \%$
- μ (uranium metal, 163.4 keV) = $2.017\ cm \cdot g^{-1} \pm 0.5\ \%$.

The 163.4 keV gamma-ray line has to be used because the other characteristic gamma-ray lines of ^{235}U are interfered by other gamma-rays.

To have a high degree of infinite thickness (> 95 % for the 163.4 keV gamma-ray energy) a large conical ore container has been designed and prepared at CBNM. The filled container accepts about 5 kg of uranium ore. The degree of infinite thickness will be determined by transmission measurement using the 661 keV gamma-ray line of ^{137}Cs.
During the past, users of the CBNM-271 plutonium pilot reference material have stated, e.g. in the frame of ESARDA and several ad-hoc working groups, the usefulness of such well characterized materials for calibration purposes.
Due to the increasing use of U, Pu mixed oxide as a reactor fuel ESARDA NDAWG members have expressed their interest in well characterized MOX reference materials.

Spike Reference Materials
A. Verbruggen, A. Lamberty, F. Hendrickx, K. Mayer, A. Alonso

The preparation of spike reference materials is an important function of CBNM. Certification procedures for two renewals are being accomplished : CBNM-040a (1 mg $^{233}U \cdot g^{-1}$ solution) and CBNM-046a (a mixed spike solution of 1 mg $^{233}U \cdot g^{-1}$ and 100 µg $^{242}Pu \cdot g^{-1}$ solution).
159 units, corresponding to 18 orders from 16 customers, of Isotopic and Spike RMs have been delivered to various laboratories within and outside the European Community in 1990.

REACTOR NEUTRON DOSIMETRY REFERENCE MATERIALS

CBNM is collaborating with the **Euratom Working Group on Reactor Dosimetry** (EWGRD) to produce a series of reference materials for neutron metrology and reactor surveillance. These are activation dosimeters of alloys, high purity metals or fission dosimeters of uranium or neptunium oxide. The preparation of the metallic reference materials is combined with co-ordinating and participating in the characterisation programme.

Sale of Reference Samples
C. Ingelbrecht

Although not all materials are officially certified yet, they have been put on sale on special request of EWGRD. During the last year 50 units have been sold to various customers within and outside the EC (see Table 8)

Table 8. Sale of reactor neutron dosimetry reference materials

No.	Material	Number of Units	Country
CBNM-502	$^{237}NpO_2$	1	B
EC-NRM 521	nickel	3	CH, USA
CBNM-522	copper	2	USA
EC-NRM 523	aluminium	2	USA
CBNM-524	iron	4	DK, NL, USA
EC-NRM 526	niobium	~10	NL, UK, USA
EC-NRM 527	Al-0.1 % Co	3	D, USA
EC-NRM 528	Al-1 % Co	15	B, D, F, UK
EC-NRM 530	Al-0.1 % Au	10	B

Preparation of New Reference Materials

C. Ingelbrecht, F. Peetermans

The analysis of CBNM-530 (Al-0.1 % Au) has been completed in collaboration with SCK/CEN, Mol and INW/RU, Gent, and a certified value of gold mass fraction of (0.100 ± 0.002) wt% (2s) has been adopted. A paper describing the preparation and certification of CBNM-530 was presented at the 15th World Conference of the INTDS in Santa Fe, September 1990. This reference material is useful both for **neutron metrology and for k_0 standardisation of neutron activation analysis.**

Encapsulation using quartz, aluminium and vanadium have been carried out on a trial basis and will be routinely available to customers. Much interest has been shown in other aluminium alloys for neutron dosimetry i.e. Al-Ag, Al-Sc and Al-In and the preparation of certified stocks of these materials will be considered. The certification analyses of CBNM-529 (rhodium) with iridium ~25 $mg \cdot kg^{-1}$ and platinum <10 $mg \cdot kg^{-1}$ are almost complete. Titanium metal suitable for neutron dosimetry (99.99 % purity, low scandium mass fraction) has been ordered.

Reporting and Certification

C. Ingelbrecht, J. Pauwels

The reference materials with numbers 523 (aluminium), 525 and 526 (niobium), have been awarded EC status after positive advice of the Nuclear Certification Group in its meeting of 1 February 1990, and the certification reports have been

printed and distributed to customers. The final drafts for certification of reference materials n° 522 (copper) and 524 (iron) have been prepared and will shortly be submitted to the participating laboratories for approval. A technical brochure giving details of all the dosimetry reference materials has been released and a poster presentation of the programme was made at the Seventh ASTM-EURATOM Symposium in Strasbourg, August 1990.

SAMPLES AND TARGETS FOR NUCLEAR MEASUREMENTS

Preparation and Characterisation of Samples and Targets

J. Pauwels, C. Ingelbrecht, R. Eykens, F. Peetermans, H. Mast, J. Van Gestel, C. Louvrier, A. Dean

158 samples and targets covering 33 different requests have been prepared and characterised in support to the CBNM specific programme. They comprise thin deposits, special plastic films and various bulk samples of metals, compounds and alloys (see Table 9).Three papers on the preparation of samples were presented at the 15th World Conference of the INTDS in Santa Fe, September 1990.

Characterisation of ^{10}B and ^{6}LiF Reference Deposits

J. Pauwels, R. Eykens, A. Lamberty

The characterisation of ^{10}B and ^{6}LiF reference deposits prepared last year [1] was completed. The ^{10}B and ^{6}LiF surface densities were determined on the basis of isotope dilution mass spectrometry on selected samples from a range of deposits, counting rates of which were compared in a neutron beam at the BR1 reactor at SCK/CEN, Mol. A set of ^{10}B IDMS determinations including a chemical purification step confirmed earlier results obtained without purification.
^{10}B and ^{6}LiF surface densities could finally be certified with accuracies of 0.30 % and 0.35 %, respectively. An intercomparison of the ^{10}B and ^{6}Li calibrations using the ^{10}B and ^{6}Li neutron cross sections showed their consistency at < 0.1 %.
In the frame of a collaboration between the University of Sussex, NIST Gaithersburg, SURRC Glasgow and CBNM one of the ^{10}B deposits was used as a reference for the accurate determination of the lifetime of the free neutron at the Institut Von Laue-Langevin, Grenoble. An improved neutron lifetime value $\tau_n = (893.6 \pm 5.3)$s was determined and jointly published. This value is

(1) CBNM Annual Report 89, EUR 12615 EN

Table 9. Preparation of thin deposits, films and bulk samples in support of the CBNM programme

Preparations	Number of Orders	Number of Samples	Preparation Methods (1)
Thin deposits			
^{10}B	2	7	VD
^{152}Gd	1	1	VD
^{157}Gd	1	1	VD
^{177}Hf	1	1	VD
Hydrogen	1	15	VD
^{241}Pu	2	2	SO
^{149}Sm	1	1	VD
^{235}U	2	2	VD
^{171}Yb	1	1	VD
Films			
Polyimide	1	2	CE
Gold coated polyimide	5	9	CE-VD
Polystyrene	1	16	CE
Bulk samples			
Aluminium	1	1	MA
Al_2O_3 - tubes	2	85	MA
^{53}Cr	2	3	CAN
Gold	2	2	MA
Iron	1	1	M-R-MA
^{7}Li	1	2	R-MA-CAN
Manganese	1	1	CAN
Melamine	1	1	CAN
^{58}Ni	1	1	R
Silicon	1	2	MA
Uranium	1	1	MA

(1) CAN canning
CE centrifuging
M melting
MA machining
R rolling
SO solution spraying
VD vacuum deposition

in good agreement with the value predicted by precise measurement of the β-decay asymmetry parameter A and the standard model : τ_n = (897.4 ± 3.7)s.

Preparation of U-Pu Metallic Spikes

C. Ingelbrecht, F. Peetermans, J.M. Orea-Rocha*

Alloys of ^{235}U - ^{239}Pu are required as metallic spikes for the accurate assay by IDMS of uranium and plutonium in undiluted input solution samples of reprocessing plants. However, problems exist with both homogeneity and workability of these alloys and a systematic study of the parameters of importance for their preparation is being carried out. A metallographic facility for α-active alloys consisting of two glove boxes for mounting/polishing and microscopy have been installed and a box for encapsulation under inert atmosphere is being prepared. Binary and ternary alloys of uranium with molybdenum, zirconium or niobium additions of 2 to 12 % have been prepared in order to stabilise the γ-phase and improve the ductility of the uranium matrix. Some success has been achieved and wires have been prepared by swaging. A study of the error propagation of IDMS indicates an optimum ^{239}Pu content of 10 % in 100 mg spikes. This is not compatible with good ductility in the alloy and some compromise in composition will have to be reached.

Preparation and Assay of Hydrogen Reference Deposits

J. Pauwels, J. Van Gestel, R. Eykens, A. Rodríguez, G. Willems**, F.-J. Hambsch

As elastic scattering on hydrogen H(n,n)H is an important standard reaction in neutron metrology, the effort to prepare homogeneous, stable and well characterised reference layers was continued. Last year, deposits of various compounds (tetracosane, tetracosanol, pentacosane, heptacosane, octacosane, octacosanoic acid and octacosanol) were evaporated onto several substrate materials. Mainly on the basis of electron microscopy studies, octacosanol was chosen as the most appropriate compound. Several evaporations of this compound on highly polished tantalum were carried out to prepare deposits of various thickness (300, 650, 1000 $\mu g \cdot cm^{-2}$) for further investigation, e.g. stability tests.

The determination of hydrogen in octacosanol has been carried out by the gravimetric method (mean: 1.421 $mg \cdot g^{-1}$; s : 0.003 ; n : 30). The mean value is close to the theoretical one of 1.423 $mg \cdot g^{-1}$.

---

* EC Fellow

** Scientific Visitor from KU Leuven, Belgium

AEROSOL AND SURFACE REFERENCE MATERIALS

Development of a Scanning Proton Microprobe and its Application to Single Aerosol Particle Analysis

G. Lövestam*, E. Swietlicki*, E. Louwerix, P. Rietveld, U. Wätjen

A scanning proton microprobe (SPM) has been developed to facilitate effective materials analysis at micrometer dimensions making use of Particle Induced X-ray Emission (PIXE) and/or Rutherford Backscattering Spectrometry (RBS). The unique features of an SPM are obtained by means of focusing a proton beam to a few μm and scanning it over the target surface. Thus not only the elemental composition of the specimen is detected but also the lateral distribution of the elements revealed.

A schematic view of the microbeam line indicating the main parts of the set-up is shown in Fig. 16. After passing through the object slits the beam enters the magnetic quadrupole doublet with an angular spread limited by the aperture slits.

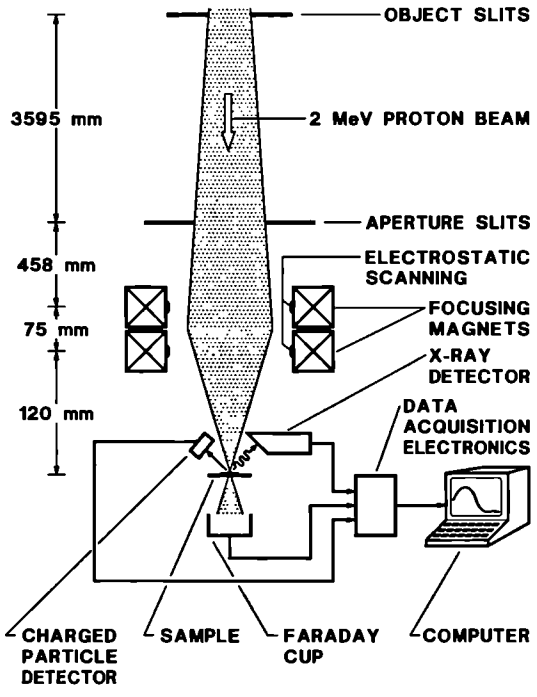


Fig. 16. Schematic drawing of the SPM set-up at the KN 3.7 MV accelerator

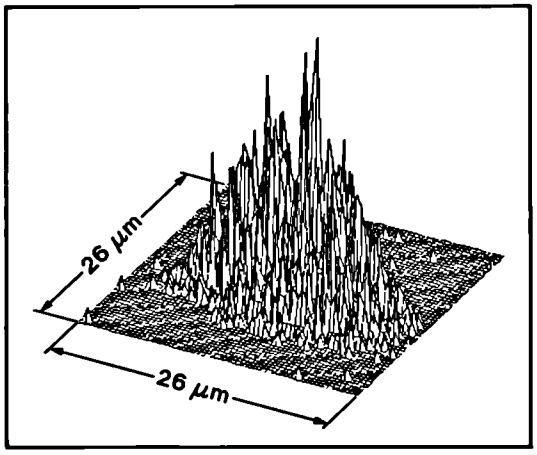


Fig. 17. Intensity map of iron from a rectangular scan over an ambient aerosol particle. The scan size is 26 x 26 μm^2

* Scientific Visitor from the Institute of Technology, Lund, Sweden

The magnetic quadrupoles function as an ion lens forming a demagnified image of the object slits in the target plane. A beam spot size of 3 x 3 μm^2 can routinely be achieved at an ion current of 100 pA, which is a practical value for the analysis of aerosol particles. Samples from other applications, e.g. in materials sciences, can be analysed with an even smaller beam spot at lower ion currents (e.g. 1.4 x 1.4 μm^2 at 10 pA).

By applying an adjustable voltage to the electrically isolated quadrupole poles, the microbeam is scanned over the sample. The scanning pattern can be rectangular in order to produce elemental maps of a selected area of the sample, or irregular in a pattern coinciding with the location of interesting sample spots. This irregular scan option reduces the required analysis time of aerosol particles considerably, because much less time is spent on merely analysing the blank collection substrate.

In pilot studies the SPM was applied so far to the PIXE analysis of laboratory aerosol and ambient aerosol particles collected by cascade impactors. As an example, Fig. 17 shows the intensity map of iron from a scan covering a single ambient aerosol particle. Similar maps were acquired for a total of 12 elements, among them chromium and manganese. It is planned for future activities to quantify such elemental maps yielding the absolute amounts of the respective elements present in single particles. It was demonstrated here that the SPM with PIXE analysis has far lower detection limits, rendering even some trace elements detectable, than other methods used so far in single particle analysis, among them the scanning electron microprobe. Thus this method is an important supplement to the analytical capabilities of aerosol chemistry.

Characterization of High-Dose Implants of Cobalt and Chromium in Silicon

A. Climent-Font*, U. Wätjen, H. Bax, L. Palmetshofer**

Single crystal silicon wafers implanted at high doses with chromium or cobalt at 300 keV, with nominal values ranging from (4.5 to 144) x 10^{15} atoms/cm^2, were prepared in order to study their potential use as working standards for Auger Electron Spectroscopy (AES) and Secondary Ion Mass Spectroscopy (SIMS). These samples were calibrated by comparison with primary standards [1] using RBS, yielding a relative inaccuracy of the determined implantation doses of about 5 %. The range and shape of the implantation were also measured by RBS within an accuracy of 20 nm (2s). The calibrated values

---

* Scientific Visitor from Universidad Autónoma Madrid, Spain
** Universität Linz, Austria

(1) I.V. Mitchell, H.L. Eschbach, L. Avaldi and W. Dobma, Nucl. Instr. Meth. 218 (1983) 91

of dose and depth profiles of implanted samples, as summarized in Table 10 for the chromium implants in silicon, will be used by other laboratories in establishing system parameters for more surface-sensitive AES and SIMS measurements.

Table 10. Implanted doses of chromium in silicon and its distribution profiles determined by RBS

Dose Nominal/Measured [10^{15} atoms/cm^2]	Projected Range [nm]	Gaussian HWHM External/Internal [nm]
4.5 / 3.8	289	104 /89
14.4 / 13.2	293	98 / 89
45 / 40	289	116 /89
144 / 147	284	117 / 85

Uniformity of a Batch of Implanted Bismuth Layers in Silicon

U. Wätjen, H. Bax

Implanted samples of bismuth in silicon had been made available as calibration standards after characterization of six samples out of a batch of 100 with the internal standard method, and testing the uniformity of the full batch[1]. After non-uniformities up to 5 % were reported [2],[3], a re-investigation by RBS of the remaining batch of 65 samples has been started.

Multi-channel scaling (MCS) data were taken of the energy window containing the bismuth signal while rotating the target wheel - loaded with 19 samples at a time - through the ion beam of 1 mm diameter (Fig. 18). So far 28 standard samples were examined. The determined non-uniformity given as the relative range of the bismuth content is < 4.0 %. This is in accordance with or even better than stated before [1], where a relative standard deviation of 1.6 % was reported at the 68 % confidence level. Nevertheless, the MCS technique will further be used to improve the quality of this reference material by assigning

(1) I.V. Mitchell, H.L. Eschbach, L. Avaldi and W. Dobma, Nucl. Instr. Meth. 218(1983)91

(2) J.A. Davies, T.E. Jackman, H.L. Eschbach, W. Dobma, U. Wätjen and D. Chivers, Nucl. Instr. Meth. B15(1986)238

(3) T.E. Jackman, J.A. Davies and D. Chivers, Nucl. Instr. Meth. B19/20(1987)345

calibration values with smaller uncertainty to the individual chip of bismuth in silicon together with an evaluation of the uniformity within each sample.

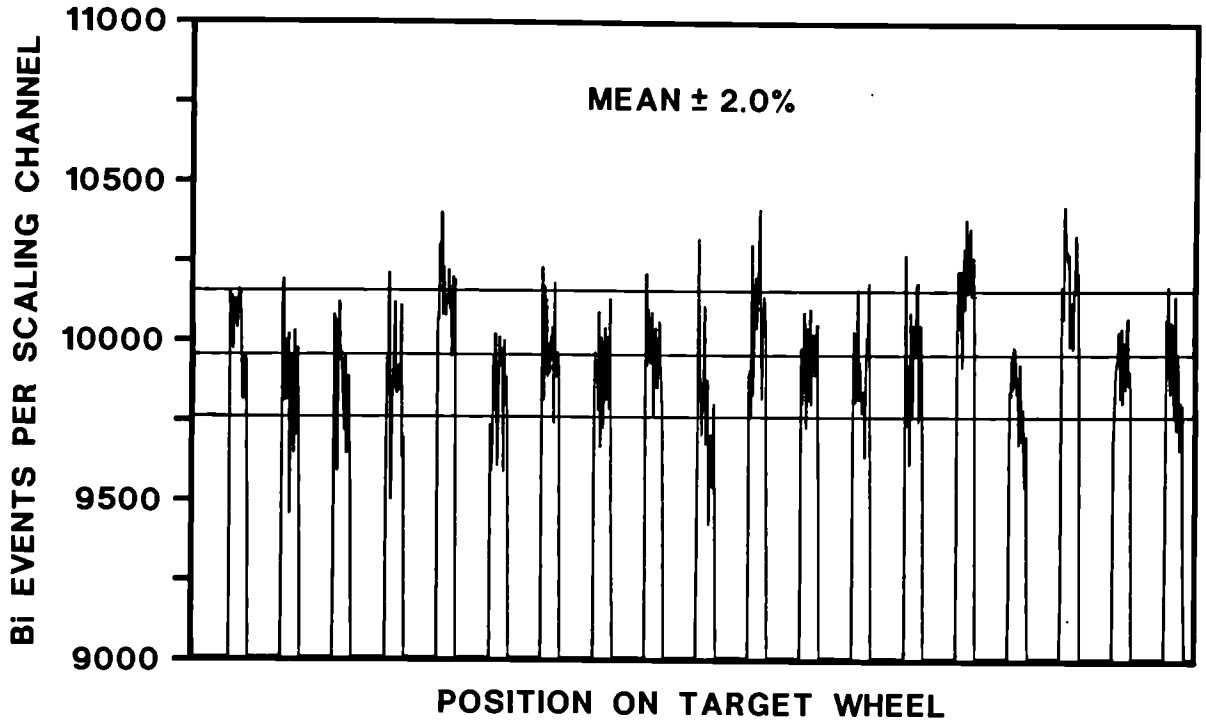


Fig. 18. MCS data of the bismuth content in 19 implanted silicon samples

ATOMIC WEIGHT DETERMINATIONS

Isotopic Measurements on Silicon

P. De Bièvre, S. Valkiers, W. De Bolle, G. Lenaers*, S. Peiser**, H. Ku**, T.J. Murphy**

This activity aims at the accurate determination of the atomic weight (molar mass), hence the isotopic composition of silicon, in single crystals by mass spectrometry. The measurements are part of a PTB coordinated project to realise a more accurate Avogadro Constant, which has a high potential significance for the International System of Units, e.g. the unit of mass and the volt.

The interrelationship of the different measurement parameters involved is :

$$N_A = \frac{A_r(Si) \cdot n}{\rho \cdot a_o^3}$$

where N_A is the Avogadro Constant, $A_r(Si)$ the atomic weight (molar mass) of the silicon single crystal, ρ the density of the crystal, a_0 the lattice constant of this crystal and n the number of silicon atoms per unit cell ($n=8$).

---

* Visiting Scientist from University of Antwerpen, Belgium

** Visiting Scientist from NIST, Gaithersburg, USA

The atomic weight value of 28.085 382 ± 0.000 023 (± 0.82 ppm, 1s) combined with

$$a_o = (543\,102.036 \pm 0.034)\ \text{fm}$$
$$\rho = (2.329\,080\,4 \pm 0.000\,001\,6)\ \text{g/cm}^3$$

as determined by PTB, led to a new value of the Avogadro Constant:

$$N_A = (6.022\,134\,1 \pm 0.000\,006\,6) \times 10^{23}\ \text{mol}^{-1}$$

A joint CBNM-PTB publication for Phys. Rev. is ready. Planning for future work is going on. The aim is to reduce the uncertainty on $A_r(Si)$, and hence on the Avogadro Constant, to 0.1 ppm.

The discussions about the aspects of modernisation and upgrading programme for the silicon mass spectrometer were continued. Analyser, collector and inlet system are ready and are being tested.

A 10 kg batch of high purity SiO_2 material, isotopic reference material CBNM-018, has been homogeneized and packed in teflon containers, in 5 g portions. Different samples have been sent to laboratories in the EC and USA, together with samples of CBNM-017, in the frame of an interlaboratory measurement evaluation programme on trace elements in semiconductor Si, organized by NIST and the ASTM Task Group on Nuclear Methods Chemical Analysis. This will allow CBNM to collect considerable information on its Si reference materials regarding impurities.

Isotope Abundance Measurements and Atomic Weight Determinations of Selected Elements

R. Mäck*, P. Taylor, P. De Bièvre

At present the adopted iron isotopic composition and the iron atomic weight A_r(Fe) are as follows [1]:

	mol %
^{54}Fe	5.8 ± 0.27
^{56}Fe	91.72 ± 0.27
^{57}Fe	2.2 ± 0.14
^{58}Fe	0.28 ± 0.06
A_r(Fe)	55.847 ± 0.003

* EC Fellow

(1) P. De Bièvre and I.L. Barnes, Int. J. Mass Spec. Ion Proc. 65(1985)211

During the previous years efforts have been directed towards the iron chemistry, e.g. the purification of enriched isotopes and checking the stoichiometry of the Fe_2O_3 used to prepare the synthetic isotope mixtures. In the present reporting period the work has mainly been focussed on the development of appropriate isotopic measurement procedures for iron. **Progress has been made to determine the absolute isotopic composition of iron**, which will result in a new determination of the Atomic Weight of Iron A_r(Fe).

A mass spectrometer was especially assigned to this work (MAT 261). Problems were encountered with the instrument's software (lack of reliability). Ad hoc solutions were implemented, but for the future a new software must be envisaged.

When determining isotopic compositions and for the preparation of the synthetic mixtures, widely ranging ion currents (from 10^{-11} to 10^{-15} A) need to be measured accurately over a very large range. Whereas in the past the linearity of the mass spectrometer has always been tested indirectly (e.g. electrically) and incompletely (e.g. not including the detector), this has now for the first time been done by a direct approach [1] using triple synthetic isotope mixtures of ^{233}U, ^{235}U, ^{238}U isotopes (CBNM Isotopic Reference Material 072/1-15) at different ion currents.

Thus, the linearity of the entire mass spectrometer from the detector to the output of the data via the software has been tested directly. As a result of this, it can be stated that the mass spectrometer used allows to measure isotope ratios down to 10^{-3} with a deviation from linearity of less than $3 \cdot 10^{-3}$ at a total ion current of 10^{-11} A.

A Thermal Ionisation Mass Spectrometric (ThIMS) procedure has been developed resulting in an increased production of iron ions (an ion current of approximately 10^{-10} A can be maintained over a 1 hour period). This is based on a carefully optimised loading procedure using a silicagel/boric acid chemical matrix on the filament.

The above procedure allows internal precisions for one filament on the isotope abundance ratios of 0.05 % and external precisions of 0.5 %. The latter is still unsatisfactory and needs further improvement.

Preliminary measurements were carried out on the enriched isotopes ($^{54}Fe_2O_3$) yielding an uncertainty of typically 1 % on ^{58}Fe in natural iron , which is far superior to the current ^{58}Fe uncertainty of about 20% (IUPAC).

---

(1) K. Mayer, R. Mäck, P. De Bièvre, "Checking the Linearity of Isotope Mass Spectrometers", presented at the Finnigan MAT Users' Group Meeting, Eindhoven

TECHNICAL APPENDIX

LARGE FACILITIES

Electron Linear Accelerator

J. M. Salomé

The GELINA electron beam was available during 3157 h for physics experiments. The parameters are shown in Table 11.

Table 11. Beam parameters of the GELINA

Pulse length [ns]	Repetition Rate [Hz]	Peak Current [A]	Mean current [μA]	Mean energy [MeV]	Time [h]	Time [%]
< 1	800	~100	65	100	2058	65.2
12	100	8	10	100	82	2.6
2000	40	0.2	16	100	588	18.6
Other parameters					429	13.6

Neutrons are produced in a rotary uranium target via (γ,n) and (γ,f) reactions. According to the requested neutron energies, moderators of water or liquid methane are placed on both sides of the target. Twelve flight paths are equipped for neutron time-of-flight experiments. On the average, 4.6 neutron beams were used simultaneously when GELINA was operated at very short bursts and 2.7 when operated at 40 Hz with the methane moderator.

A neutron measurement campaign with the liquid nitrogen cooled methane moderator was disturbed due to a leak in the isolated pipes. The concept of the circuit was slightly modified to minimize the repair time in case of defectiveness.

For limited periods of time, the accelerator was operated with uncommon parameters suited to the Transition Radiation (TR) experiments. Very low peak currents, down to 10 μA can be obtained with derated rf power and very low cathode temperature. In these conditions, the "dark currrent" and background are sufficiently low to carry out X-ray TR spectrum measurements.

A 35-MeV magnetic deflexion has been ordered in view of reconstructing the photo-activation facility. The three-position switching magnet will be installed in the target room. Following the requests, the electron beam will become available for the neutron cross-sections, transition radiation or photo-activation measurements.

Van de Graaff Accelerators

A. Crametz

The total working time of the two accelerators was 3063 hours. A break down of this total is given in Table 12.

Table 12. Exploitation of the Van de Graaff accelerators

	CN - 7 MV		KN - 3.7 MV	
	Time [h]	Time [%]	Time [h]	Time [%]
Adjustments	47	1.5	62	2.0
Experiments	842	27.5	869	28.4
Conditioning	333	10.9	-	-
Maintenance	655	21.4	36	1.2
Modifications	219	7.1		

- Modifications :

  Accelerators have been stopped during three weeks for installation of a new alarm and safety system in all the laboratories. During this time also the air conditioning system in the target hall has been modernized.

- CN - 7MV :

1) Maintenance : Twice the tank has been opened to replace the ion source. The high number of hours is due to modification of the experimental set-up on the four installed beam extensions and to test runs for experiments concerning work for third parties.
2) Conditioning : The determination of the shape of the neutron spectrum produced by a 7 MeV deuteron bombardment on a 1 mm thick Be target is in progress. For the measurements it is necessary to condition the accelerator during the night in order to maintain a stable voltage during the day.

- KN - 3.7 MV :

  The tank has been opened once to replace the ion source after 1320 hours of operation. Beside RBS and PIXE experiments the microbeam line has been put into working.

  The unique feature of the scanning proton microprobe (SPM) is obtained by focusing the beam to a few micrometers and scanning it over the target area. Thus, not only the elemental composition of the specimen is revealed like in RBS and PIXE, but the lateral resolution of every

individual element is also detected. Details of the SPM set-up are described under the heading "Development of a Scanning Proton Microprobe ...", p. 44.

The critical parameters that influence the beam quality at focus are the energy spread and the brightness of the proton beam. The energy spread gives rise to chromatic aberrations in the focusing magnets. The brightness, defined as the beam current per source area and steradian, determines the current density in the final beam spot. The energy spread has not yet been measured but is reported by the manufacturer to be about 0.14%. The brightness has been measured to be less than $1pA \cdot \mu m^{-2} \cdot msr^{-1}$ which is lower than expected.

The final beam-spot size is usually determined at a proton current of 100 pA. This is regarded to be the minimum beam current practical for the analysis of biological samples. Samples from other fields of science,e.g. material science, can however, be analyzed with a lower beam current (e.g. 10 pA). Here, a beam-spot size of 3 x 3 μm^2 with a beam current of 100 pA, a 2 x 2 μm^2 with 50 pA and a 1.4 x 1.4 μm^2 with 10 pA can routinely be achieved.

These values are particularly good for the brightness stated above and suggest that the actual brightness is higher than measured.

Mass Spectrometers

P. De Bièvre, K. Mayer, R. Damen, A. Alonso, P. Hansen, H. Nerb, R. Fiedler*, R. Perrin**

Twelve isotope mass spectrometers are operated and an (ultra-)Clean Chemical Laboratory (CCL) is being built.

1) Gas instruments : three are operational; one is being upgraded rather thoroughly in order to perform future silicon measurements (on SiF_4) for the Avogadro Constant project and one is under discussion to be returned to the manufacturer because of unsatisfactory performance.

2) Instruments for solids : five are operational , some of them specifically tuned to an element (U-Pu-B-Li-Fe). Testing of a new higher abundance sensitivity standard spectrometer has been going on : the gain in abundance sensitivity may be 10^2 (from 10^{-6} to 10^{-8}) ; this prototype was ordered by CBNM in 1982 and seems to initiate a new production line.

---

* Visiting Scientist from IAEA Safeguards Analytical Laboratory (SAL), Seibersdorf, Austria

** Visiting Scientist from Los Alamos National Laboratory, USA

A more accurate determination of the minor abundant isotopes in enriched isotopes is possible with this instrument. The reduced uncertainty on the minor isotopes in isotopically enriched material will lead to a lower overall uncertainty of the synthetic isotope mixtures prepared from higly enriched material. One instrument is undergoing a complete overhaul and will be equipped with a high accuracy ion counter development by Los Alamos National Laboratory staff; one 20 year old instrument is being discarded.

The software for isotopic measurements presently distributed by the manufacturer does not satisfy our needs. This position is shared by Los Alamos National Laboratory (LANL) and IAEA's Safeguards Analytical Laboratory (SAL) in Seibersdorf. A common isotope ratio measurement philosophy was worked out and the development of an appropriate software package was agreed. The programme will cover instrument operation, data acquisition and raw data transfer modules, as well as data treatment and uncertainty calculation. This software will be applied to the MAT product line and to the NBS instruments at CBNM, SAL and LANL.

3) - An (ultra-)Clean Chemical Laboratory (CCL) is being built; it will contain up to 6 simultaneously occupied working places; it will be of Class 100 [1] in the room and Class 10 [1] on the working benches and will contain CBNM's 2nd generation ultra-pure acid distillation laboratory; works have advanced to the point that the building is under roof.

---

(1) Corresponding to about 3500 and 350 particles ($\geq$ 0.5 µm) per m^3, respectively

DATA PROCESSING AND ELECTRONICS

CBNM Computer Network

T. Babeliowsky, C. Bernard, C. Cervini, H. Horstmann, C. Van den Broeck*, P. Van Roy

The CBNM computer network for scientific data processing and office automation has been upgraded, in particular for external communications.

- All IBM and XEROX terminals and all computers on Ethernet with 3270 emulation software can be used to send and receive electronic mail based on the UCLA/Mail 400 message handling system (CCITT X.400). By means of an X.400 relay (Namur) and gateways in Germany (DFN) and Belgium all important scientific computer networks can be reached (EARN, BITNET, ARPANET, EUNET, UUCP, JANET and many others).
- A connection to IXI (International X.25 Interconnect), the European backbone net for research, has been established. In this way telecommunication with JRC Ispra could be improved in speed and stability.
- Users of IBM 4381 and other computers on the network can access remote computer systems on X.25 networks reachable via the Belgian public data transmission network (DCS).
- Special software has been installed for the connection of a computer in the Mass Spectrometry to the ECSAM Office in Luxemburg via DCS.

Scientific-Technical Applications of Personal Computers

T. Babeliowsky, W. Stüber, L. Van Rhee

- The programs for analysis of densitometer data from spark source mass spectrometry have been adapted to the data format of the prototype microdensitometer constructed by CIT, Geel. Graphical inspection of raw data at an IBM/PC and of deconvoluted peaks at a graphical IBM 3270 terminal is possible.
- The software for the control computer of the constant current coulometry unit was further improved. Several hardware-software incompatibility problems, which caused unreliability, had to be identified and solved.
- Work has been carried on for the extensive data base of locations and types of fire-fighting products at CBNM.

---

* Comparex, Brussels, Belgium

Multiparametric Event Acquisition and Analysis Using Transputers

C. Bastian, S. de Jonge, J. Gonzalez

A multiplexer module combining the inputs of 4 ADC's to a (up to) 4-parametric event was built and tested. Such a module uses 2x16-bit transputers and may be combined with similar modules to build up events of up to 16 (possibly 64) parameters. The event stream is passed to an array of 32-bit transputers sorting the events by coincidence pattern and processing them in parallel. Event sorting and analysis is thus performed outside of the host machine- a microVAX in this case.

Bench tests of the multiplexer have shown that a throughput of more than 50000 events/s can be ensured. Nevertheless, the multiplexer is being improved by adding an input FIFO buffer in order to reduce the deadtime.

Interval Digitizer

S. de Jonge, B. Denecke

A prototype of an interval digitizer has been delivered and tested. The instrument is a single width NIM module, which converts the time interval between subsequent input signals into a binary value and is intended to measure distorsion in time distributions. The instrument contains a 512 word buffer memory, has a selectable channel width of 10 to 2560 ns, a range and offset of 4096 channels, and can resolve 3 input signals within 25 ns. It can be connected as a ND ADC.

EXPLORATORY RESEARCH

Study of Transition Radiation

X. Artru*, P. Dhez**, P. Goedtkindt***, M. Jablonka•, N. Maene••, F. Poortmans••, P. Rullhusen, J.M. Salomé, P. ter Meer, F. Van Reeth, L. Wartski•••

Transition Radiation (TR) is generated when energetic electrons cross the boundary between two media. The radiation can extend from microwave to X-ray frequencies and is emitted into a narrow cone in forward direction.
Different types of radiators have been studied by computer simulation in order to optimize the parameters such as thickness and number of foils, formation length and X-ray absorption for various materials. Successful experiments have been achieved using the 550 MeV electron beam of the Accélérateur Linéaire de Saclay (ALS). The X-ray angular distribution was measured for various radiators of one or several thin foils of kapton, mylar, aluminium or copper. Strong enhancement of the radiated intensity due to interference effects was observed using multi-foil radiators. Micro-lithography with 300 nm resolution was achieved. The results have been presented at two conferences.
Measurements of the radiation spectrum in the X-ray energy range are under current investigation at GELINA using a Si(Li) detector.
Optical transition radiation (OTR) will be used for electron beam characterization. A complete optical system has been set up and will be transferred to the target room in January 1991, in order to allow on-line energy and divergence measurements of the GELINA electron beam.

Trace Metals Analyses in Biological Samples

J. McCourt, F. Cordeiro-Raposo, B. Slowikowski, N. Soufi***, A. Muñoz***, G. Bordin, A. Rodríguez.

Many trace metals have been shown to have biochemical functions in living organisms. Besides the total concentration of an element, it has been recognized that a good knowledge of the chemical forms of the element (chemical speciation) is necessary to understand its role in biochemical and environmental processes. Within organisms (ranging from mammal tissues to mussels), a group of specific metalloproteins, called metallothioneins, appear to

---

* IPN-Lyon, Villeurbanne, France
** LURE and LSAI, Orsay, France
*** EC Fellow
• DPhN/STAS-CEN Saclay, France
•• SCK/CEN, Mol, Belgium
••• Université de Paris XI, Orsay, France

be active in the metabolism and detoxifying action of some metals, particularly copper, cadmium and zinc. Still open are questions such as whether or not these metallothioneins are present in all living organisms and, if so, under what conditions they are produced and whether these proteins are structurally similar to the first metallothioneins isolated in mammals.

The speciation scheme of an element is normally studied by using and comparing the results obtained by different analytical techniques, each one providing specific information. Owing to the small sample sizes normally available and the low metal concentrations present in most biological tissues, good sample preparations are necessary before analyses. To date, analyses have been carried out by three analytical techniques, namely electrothermal atomic absorption spectrometry (ETAAS), inductively coupled plasma spectrometry (ICP) and voltammetric methods. Equipment for liquid chromatography has been installed recently.

Research in this field of trace metals in biological media is progressing in three main directions:

1) Metallothionein isoforms and their metal composition as indicators of anemia and metal toxicity.

   After optimization of the different parameters, the concentrations of copper, cadmium, iron and zinc were measured by ETAAS in samples provided by the Complutense University of Madrid, (see Table 13.), as well as in commercial metallothioneins. On the latter proteins, fundamental studies on speciation of cadmium and zinc are being performed using electrochemical methods, e.g. differential pulse polarograms taken under different conditions allow the distinction between complex forms of Zn and Cd thioneins from Zn^{2+} and Cd^{2+}. From these results apparent stability constants have been estimated for each complex cation.

2) In collaboration with the Delta Institute for Hydrobiological Research in The Netherlands, a programme of investigation on trace metals in some marine bivalves has been initiated. The determination of total metal concentrations (copper, cadmium, iron) in the clam *Macoma balthica* - collected at regular intervals in the Schelde estuary - is well advanced. A complete procedure of sample preparation has been developed (sampling, freeze drying, digestion, etc.) and controlled with a certified reference material (BCR 278, mussel *Mytilis edulis*). Notably different digestion conditions have been checked to find the most suitable one. Analyses done by ETAAS and ICP show very good agreement. Concentrations of five sets of samples, collected every two months at two places,have been measured. These data are the averages of the results from measurements with calibration curves and standard additions.

Table 13. Determination of Metal Concentrations by ETAAS in Metallothioneins Isolated From Rat Livers

Sample		Cadmium [$\mu g \cdot l^{-1}$]	Zinc [$\mu g \cdot l^{-1}$]	Copper [$\mu g \cdot l^{-1}$]	Iron [$\mu g \cdot l^{-1}$]
Control	F - 30	< d.l.	2565 ± 40	5.4 ± 0.3	111.7 ± 6.0
	F - 32	< d.l.	3040 ± 12	< d.l.	< d.l.
	F - 33	< d.l.	1288 ± 25	< d.l.	< d.l.
	F - 34	< d.l.	1372 ± 21	< d.l.	< d.l.
Anaemia*	F - 30	< d.l.	136 ± 10	23.0 ± 3.4	< d.l.
	F - 32	< d.l.	343 ± 26	22.7 ± 4.8	< d.l.
	F - 33	< d.l.	306 ± 18	< d.l.	< d.l.
	F - 34	< d.l.	644 ± 118	< d.l.	< d.l.
Anaemia**	F - 30	1278 ± 322	< d.l.	< d.l.	< d.l.
+ Cd	F - 32	1385 ± 329	< d.l.	< d.l.	< d.l.
	F - 33	1234 ± 266	< d.l.	< d.l.	< d.l.
	F - 34	1132 ± 210	< d.l.	26.1 ± 3.5	< d.l.
Anaemia**	F - 30	< d.l.	1360 ± 42	9.9 ± 0.8	< d.l.
*	F - 32	< d.l.	1497 ± 312	7.1 ± 0.3	< d.l.
+ Zn	F - 33	< d.l.	<d.l.	57.9 ± 3.7	< d.l.
	F - 34	< d.l.	152 ± 7.5	34.4 ± 0.8	< d.l.

d.l. : detection limits for each element [$\mu g \cdot l^{-1}$] ; Cd : 3.5 ; Zn : 1.0 ; Cu : 5.9 ; Fe : 6.8.
* rats fed with a deficiency in iron
** anaemia plus injection of cadmium
*** anaemia plus injection of zinc

3) The inhalation of some metals (cobalt, nickel, copper, cadmium) is responsible for very serious pulmonary diseases. Hence, it is very important to be able to measure the concentrations of these metals in living tissue samples and in broncho-alveolar lavages (BAL). These lavages are being studied applying differential pulse adsorptive stripping voltammetry, which has been optimized for the determination of cobalt and nickel at the ppb level. Interferences from other metals like cobalt, zink, lead, copper and iron on the nickel determinations were found to be negligible. In parallel with this work the suspended matter isolated by

centrifugation of BAL has been analyzed by X-ray fluorescence leading to a qualitative identification of only two elements, zink and potassium, due to the lack of XRF reference materials.

Development and Application of Isotope Dilution Mass Spectrometry

A. Lamberty, G. Lapitajs*, A. Verbruggen, L. Van Nevel**, A. Götz***, M. Rener**

Isotope Dilution Mass Spectrometry (IDMS) is being developed as a reference method for assaying traces of toxic and essential elements in biological and environmental systems. In the frame of a collaboration with NIST, CBNM was asked to certify several elements (rubidium, boron, copper) in the drinking water reference material NIST-SMR 1643c. The rubidium concentration was determined by IDMS and certified to be $(11.31 \pm 0.08)^{\bullet}$ $\mu g \cdot g^{-1}$ after separation of the rubidium on a cation exchange column. $^{85}Rb/^{87}Rb$ isotope ratios were measured on a magnetic sector instrument and on a quadrupole instrument and agree within the measurement reproducibility (0.55 %).

A ^{11}B Spike Isotopic Reference Material was prepared and characterized by Reverse IDMS containing $(2.424 \pm 0.024)10^{21}$ atoms $^{11}B \cdot kg^{-1}$ of solution. The material is available as CBNM-611 in quartz ampoules containing approximately 5 g solution. This natural boron spike was used to certify the boron content of a series of ^{10}B reference deposits with different surface densities used in the determination of the lifetime of the neutron (collaboration NIST-Sussex-SURRC-CBNM)[1]. The total uncertainty is 0.97 % (series without chemical separation of the boron subsequent to the dissolution of the deposit) resp. 1.2 % (series with chemical purification of the boron) on the number of ^{10}B atoms in the deposits.

An older natural boron CBNM-reference deposit was recalibrated revealing a discrepancy of 2.3 % on the absolute surface density compared with the value obtained by the non-specific (100 – X) method [2]. The boron concentration on NIST SRM 1643c was certified to be $(0.117\,6 \pm 0.001\,4)^{\bullet\bullet}$ $\mu g \cdot g^{-1}$.

---

* EC Fellow

** University of Antwerpen, Belgium

*** Visiting Scientist from the Universität Regensburg, Germany

• The uncertainties (2s) include statistical and systematic uncertainty contributions related to the certification work done here but do not include long term evaporation effects through the polyethylene bottles.

(1) J. Byrne et al., Nucl. Instr. Meth. Phys. Res., A 284 (1989) 116

(2) H.L. Eschbach, CONF - 711002 (1971) 77

•• The uncertainties (2s) include statistical and systematic uncertainty contributions related to the certification work done here but do not include long term evaporation effects through the polyethylene bottles.

A **^{6}Li Spike** Isotopic Reference Material CBNM-615 was prepared in quartz ampoules (ca. 5 g) and characterized by Reverse IDMS. It contains (2.304 ± 0.016) 10^{21} atoms $^{6}Li \cdot kg^{-1}$ of solution. This spike was used to determine the lithium concentration in BCR 303 material $((0.517\ 1 \pm 0.005\ 7)\ m\ mol \cdot l^{-1})$ and in BCR 304 material $((0.987 \pm 0.014)\ m\ mol \cdot l^{-1})$.
The lithium content of a series of ^{6}LiF reference deposits with different surface densities used for the determination of the neutron lifetime (collaboration NIST-Sussex-SURRC-CBNM) [1] was certified with a total uncertainty of 0.50 % on the number of ^{6}Li atoms in the deposit.
IDMS has been developed

- for chlorine leading to the preparation and characterization of a **^{37}Cl Spike** which was used to certify the ^{35}Cl and ^{36}Cl content of a series of targets used for (n,p) cross section measurements. Also a ^{36}Cl abundance was certified. Evaluation is being finalized.
- for cadmium leading to the preparation and characterization of a **^{111}Cd Spike** used to certify the cadmium concentration in four plastic reference materials for the German car industry (VDA). Evaluation is being finalized.
- for copper including the preparation and characterization of a **^{65}Cu Spike** used to certify the copper concentration in NIST SRM 1643c. Evaluation is going on.
- for silicon using Negative Thermal Ionization (NTI) for the first time to determine the silicon content of a water doped with 15 ppb natural silicon. Using a previously characterized ^{29}Si spike, these silicon traces could be determined with an accuracy of 9.4 %.

---

(1) J. Byrne et al., Nucl. Instr. Met. Phys. Res., A 284 (1989) 116

SCIENTIFIC AND TECHNICAL SUPPORT TO THE COMMISSION

SUPPORT TO DG-I : INTERNATIONAL COLLABORATION - NUCLEAR SAFEGUARDS

Preparation of U-Pu Dried Spikes

P. De Bièvre, A. Verbruggen, K. Mayer, A. Alonso, F. Hendrickx

Development and preparation work continued. The IAEA has tested and ordered solid spikes in the dried nitrate form for the accurate assay of IDMS of uranium and plutonium in undiluted input samples of reprocessing plants : CBNM-1027a and 1027b. The aliquoting into glass vials (ca. 500 units in total) is carried out metrologically : a given amount (known to 0.05 %) of material is distributed in the vials. Verification measurements of the established certified values will be performed by IDMS.

Each batch will consist of two sub-batches, the first 200 units containing about 48.5 mg uranium and 1.5 mg plutonium, the second 50 units containing 97 mg uranium and 3 mg plutonium (certified to 0.1 % or better for the ^{235}U or ^{239}Pu content, respectively).
Abundances (in mole %) are as follows

	CBNM-1027a	CBNM-1027b
^{234}U	< 0.4	< 0.4
^{235}U	19.8	19.9
^{236}U	≦ 0.1	≦ 0.1
^{238}U	79.8	79.8

^{238}Pu	≦ 0.03
^{239}Pu	97.1
^{240}Pu	2.8
^{241}Pu	0.04
^{242}Pu	≦ 0.03

* EC Fellow

SUPPORT TO DG-XII : "BUREAU COMMUNAUTAIRE DE RÉFÉRENCE" (BCR)

Storage, Sale and Distribution of Samples

E. De Coninck

Support to the BCR is provided by storage and distribution of BCR reference materials and candidate RMs for sale and analysis. In this reporting period 7925 RMs were sold. In addition, 2027 samples were distributed for analytical intercomparisons and homogeneity studies covering 1130 dispatches (see Table 14.). Several new raw materials (e.g. orange juice, milk powder, soils, sediments, fly ashes, pig liver) were received for transformation and more than 15000 new reference samples prepared elsewhere were received for packaging, labelling, bookkeeping and storage under specific conditions for each material.

Table 14 . Breakdown of BCR RMs as handled by CBNM

	Sale	Samples for analyses	Dispatches
1986/1987	1768	1226	396
1987/1988	2407	1258	475
1988/1989	4775	1126	753
1989/1990	7925	2027	1130

Non-Ferrous Metals

C. Ingelbrecht, P. de Vos, A. Dean

The preparation of titanium reference materials BCR-89 and BCR-90 were completed. Samples (blocks 2mm x 2mm x 3mm) were made by slicing discs from round bars and subsequent dicing on a microprocessor-controlled cutting machine.

Biological Reference Materials

G.N. Kramer, J. Pauwels, K.H. Grobecker, L. De Angelis, M. Dürr

The preparation of a codfish candidate reference material (BCR-422) was carried out by cryo-grinding, freeze-drying and subsequent bottling (1300 samples of 7 g each) under dry controlled atmosphere. Microhomogeneity checks and production control were done by solid sampling Zeeman atomic absorption spectrometry. Two papers, one on the apparatus development and one on the preparation of the material, were presented at BERM-4 in Orlando, USA (Jan. 1990).

A feasibility study of jaw crushing at liquid nitrogen temperature of pig liver was started. The formation of meat balls between the jaws will require a redesign of the previously conceived jaw crusher.

Conservation tests on freeze-dried orange juice powder to be certified for amino acids and sugars were successfully continued in cooperation with Rijksuniversiteit Gent. In preparation of the production of a large batch of candidate reference material, an evaluation of the modified parameters of the new high capacity freeze-drier was started.

Freeze-drying experiments were further conducted for red wine, port, grape juice, red beet and milk contaminated with dioxine.

Environmental Reference Materials

G.N. Kramer, K.H. Grobecker, M. Dürr, S. Palmeri

A provisional laboratory for the production of environmental reference materials was set up.

A candidate reference material (BCR-424) of TBT-contaminated sediment was prepared (1000 bottles) and distributed for homogeneity control. The rest of the material (particle size > 70 µm) was jet-milled in cooperation with Alpine, Augsburg and also bottled (800 samples) as research material.

Two soils were sieved, and subsequently jet-milled to a particle size of < 70 µm in cooperation with Alpine, Augsburg.

A batch of 250 samples of fly ash contaminated with dioxine was prepared on behalf of BCR and supplied to SCK/CEN, Mol for an intercomparison.

SUPPORT TO DG-XIII :

Technology Transfer - Gas Encapsulation in Zeolites

P. De Bièvre, W. De Bolle, E. Vansant*

In the frame of a CEC license agreement, research on the current status and future developments of gas analysis by mass spectrometry has been continued in order to evaluate the achievements of present measurement methods and to identify needs for development. The study is made in the context of the patented gas separation and ultra-purification techniques using modified Zeolites. Two new EUR Patents were granted : EUR 0 189 607 (1990.05.23) and EUR 0 199 854 (1990.01.31) and one US Patent : 4 933 162 (1990.01.31) on

- A method for the separation of hydrogen isotopes
- Process for ultra-drying of gas.

A sensitive quadrupole spectrometer has been installed and tested. Sensitivities to 10 ppb level have been attained. Various fluoride gases have been measured for test purposes : SiF_4 - CF_4 - SF_6 - BF_3.
As a test, the isotopic composition of argon in air (~ 1 %), krypton in air (~ 1 ppm) and xenon in air (~ 80 ppb) could be determined within a few % in direct measurements, e.g. without separating the noble gases from the air.

SUPPORT TO DG-XVII :

Nuclear Safeguards - ECSAM

K. Mayer, A. Alonso, P. De Bièvre

In the context of the EURATOM Safeguards System, CBNM is supporting the Safeguards Directorate by different actions. One of central importance is the execution of destructive analysis of samples taken by the Safeguards inspectors for verification purposes at the various nuclear fuel installations and laboratories. About 50 U/Pu samples were handled. This activity is backed by a quality control programme. The results are confidential.
Consulting has been performed for the On-Site-Laboratory (OSL) concept including first ideas for "remote quality control" of such laboratories.

---

* University of Antwerpen, Belgium

WORK FOR THIRD PARTIES

Metrology of Radioactive Waste Barrels

D.F.G. Reher, B. Denecke

The Belgian organism responsible for the treatment and storage of radioactive waste (NIRAS/ONDRAF) contracted CBNM to analyse conditioned and non-conditioned radioactive waste containers. For this purpose CBNM is using a professional gamma scanning device, allowing to scan heavy conditioned waste drums. The operation of the scanner has been automatized and the efficiency for different concrete matrices has been measured. The uncertainty on the total activity of ^{60}Co and ^{137}Cs is about $\pm$ 15 % for homogeneously distributed waste and $\pm$ 30 % for heavily shielded inhomogeneously packed waste.
Several 400 l barrels containing filters, conditioned evaporator concentrates, or resins from Belgian power stations were analyzed.

Neutron Irradiations of Biological Samples

A. Crametz, F. Falque, J. Leonard

Two runs for irradiating blood samples were made with 0.6 MeV neutrons on request of the SCK/CEN Radiobiology Department. The doses have been determined for each irradiation with a 0.53 cm^3 thimble tissue-equivalent ionisation chamber operated in continuous gas flow. Small temperature and pressure correction factors are determined separately before irradiation of blood samples.
It is expected to get beginning of next year, silicon samples from the Roma University. A fluence of 10^{13} neutrons$\cdot cm^{-2}$ of 1 MeV is requested. Because of the technical limitation of the telescope (1 MeV is the lower limit), the long counter has been used and a calibration factor between telescope and long counter has been determined.

Preparation and Characterisation of Samples and Targets

J. Pauwels, C. Ingelbrecht, R. Eykens, F. Peetermans, H. Mast,
J. Van Gestel, C. Louvrier, A. Dean, A. Lamberty, A. Götz*, C. Hofmann**,
K.H. Grobecker***

In total 104 samples and targets corresponding to 42 orders from 12 customers outside CBNM have been prepared, characterised and supplied as shown in Table 15.
In the non-nuclear field, four candidate reference materials (more than 4000 samples) were prepared on behalf of VDA (Verband der Automobilindustrie e.V., Frankfurt). The homogeneity was controlled using solid sampling Zeeman atomic absorption spectrometry. Certification using isotope dilution mass spectrometry is in progress.

Regular European Interlaboratory Measurement Evaluation Programme (REIMEP)

P. De Bièvre, K. Mayer, A. Alonso, A. Rodriguez, M. Bickel, W. Leidert,
A. Michiels, F. Quik, W. Nagel, W. De Bolle, A. Verbruggen, F. Hendrickx

REIMEP 89/90 on uranylnitrate solution, UO_2 Powder, UO_2 pellets : incoming results were coded in order to prevent identification of the 21 participating laboratories. Graphs were generated displaying the results for isotopic composition and element concentration measurements. The results were presented and discussed with the participants during the meeting of the ESARDA Working Group on Destructive Analysis in Vienna. They are planned to be published in the near future. Problems occurred especially for the assay of the UO_2 powder where a weight change correction proved to be necessary.
The majority of the laboratories regarded themselves as "experienced" for the paricular analysis of the material concerned. **No correlation between this self assessment and the quality of results was found.**

REIMEP 90/91 on spent fuel, fission product free input material, MOX pellet, plutonium nitrate solution : the new rounds were announced to all interested laboratories. Due to the fact that all four materials contain plutonium, the number of participants is lower than that for the previous rounds. The characterisation of these materials is ongoing.

---

* Visiting Scientist from the Universität Regensburg, Germany
** EC fellow
*** Visiting Scientist from Universität Giessen, Germany

Table 15. Supply of thin deposits, films and bulk samples to external customers

Preparations	Applicants [1]	Number of Orders	Number of Samples	Preparation Methods [2]
Thin deposits				
^{239}Pu	(1)	1	6	SP
^{241}Pu	(2)	1	2	SO
^{232}Th	(2)	1	1	SU
^{233}U	(3)	1	2	VD
^{235}U	(1)	2	12	SP
^{236}U	(2)	1	2	SU
^{238}U	(4)	1	4	VD
Films				
Polyimide	(5)	1	6	CE
Bulk samples				
Al-Ag	(6)	1	6	LM-MA-CAN
Al-Au	(7)(8)(9)	3	5	LM-R-WD
Al-Lu	(8)	1	1	LM-WD
Al-Mn	(2)(8)	3	3	LM-R-WD
Al-Ni	(10)	1	1	LM-WD
Al-238U	(2)	2	2	LM-R-WD
Au-Fe	(11)	1	1	M
Iron	(11)	2	5	MA
Iron, nickel, cobalt, niobium	(6)	1	9	LM-MA-CAN
^{6}Li	(2)	1	2	R-MA-CAN
Ni-Al	(11)	1	1	M
Ni-Co	(6)(11)	2	7	M-MA-CAN
Ni-Fe	(11)	1	1	M
Ni-In	(11)	6	6	M
Ni-In-Nb	(11)	1	4	M-R
Ni-Pd	(11)	1	1	M
Ni-Rh	(11)	1	1	M
Ni-Si	(11)	1	1	M
Ni-Ta	(11)	1	1	M
Titanium	(11)	1	6	MA-CAN
Uranium	(12)	1	5	CAN

(1)
(1) ILL, Grenoble (F)
(2) Rijksuniversiteit, Gent (B)
(3) Univ. Tübingen (D)
(4) Los Alamos Nat. Lab. (USA)
(5) CEA, Bruyères-le-Châtel (F)
(6) HFR, Petten (NL)
(7) ECN, Petten (NL)
(8) KfA, Jülich (D)
(9) UI Antwerpen (B)
(10) Studsvik (S)
(11) KU, Leuven (B)
(12) IAEA, Wien (A)

(2)
CAN canning
CE centrifuging
LM levitation melting
M melting
MA machining
R rolling
SO solution spraying
SP spraypainting
SU suspension spraying
VD vacuum deposition
WD wire drawing

Isotopic and Compound Analysis of Gases

P. De Bièvre, W. De Bolle, S. Valkiers

A new gas mass spectrometer (quadrupole) was made operational to execute gas- and isotopic measurements. Especially the measurement procedures and software for isotopic analysis are now such that measurements can be done at the 1 % level or better without calibration with synthetic isotope mixtures and only based on the detailed knowledge of the measurement process resulting in adequate corrections for systematic errors.

Measurements were performed to certify UF_6 in-plant Reference Materials of a commercial uranium enrichment plant. Also a number of UF_6 measurements were carried out serving as an "external quality control" on their production materials.

Isotopic and compound analysis of about 10 mixtures were performed by high accuracy, calibrated gas mass spectrometry. Extensive attention was paid to proper calibration of these measurements.

GENERAL INFORMATION

LIST OF PUBLICATIONS

CONTRIBUTION TO CONFERENCES

ALTZITZOGLOU, T., BORTELS, G., DENECKE, B., GROSSE, G., MOUCHEL, D., REHER, D.F.G., BAMBYNEK, W.
Activities of the Radionuclides Metrology Group of the Central Bureau for Nuclear Measurements.
Intern. Conf. Applications of Nuclear Techniques, Crete, June 24-30, 1990

BAMBYNEK, W.
Reorganization of atomic shells after radioactive decay.
2nd Intern. Summer School on Low-Level Measurements of Man-Made Radionuclides in the Environment, La Rábida, Hùelva, June 24-July 6, 1990

BAMBYNEK, W.
Production of inner-shell vacancies by orbital electron capture of the nucleus and by internal conversion of gamma rays.
2nd Intern. Summer School on Low-Level Measurements of Man-Made Radionuclides in the Environment, La Rábida, Hùelva, June 24-July 6, 1990

BAMBYNEK, W.
CEC-JRC Central Bureau for Nuclear Measurements.
EUROMET Workshop Ion. Rad. Radioactivity, Teddington, October 7-10, 1990

BASTIAN, C., DE JONGE, S.
An event-coder module for multiparametric data acquisition in nuclear experiments.
Intern. Sem. Nucl. Electronics, Warsaw, September 25-28, 1990

BASTIAN, C., DE JONGE, S.
On-line analysis of nuclear reactions using arrays of transputers.
Intern. Conf. Supercomp. in Nucl. Appl., Mito, March 12-16, 1990

BAUER, P., BORTELS, G.
Response of Si(Li) detectors to electrons, deuterons and alpha particles.
7th Symp. Rad. Meas. and Appl., Ann Arbor, May 21-24, 1990
Nucl. Instr. Methods, Phys. Res. A299 (1990) 205

CLIMENT-FONT, A., WÄTJEN, U., BAX, H., PALMETSHOFER, L.
Characterization of high dose implants of Cr and Co in Si.
IB-SA 90, Namur, September 5-7, 1990

COCEVA, C.
Neutron decay of isolated nuclear levels excited by gamma-rays.
6th Intern. School on Neutron Physics, Alushta, October 8-18, 1990

CORVI, F., FIONI, G., MAURI, A., BABELIOWSKY, T.
The 30keV averaged neutron capture cross sections of ^{56}Fe and ^{60}Ni.
7th Intern. Symp. Conf. Gamma-Ray Spectrosc. and Rel. Topics, Asilomar, October 14-19, 1990

EYKENS, R., GOETZ, A., LAMBERTY, A., VAN GESTEL, J., PAUWELS, J., WAGEMANS, C., DRUYTS, S., D'HONDT, P.
Preparation and characterization of ^{35}Cl and ^{36}Cl samples for (n,p) cross-section measurements.
15th World Conf. Intern. Nucl. Target Dev. Soc., Santa Fe, September 10-14, 1990

FIEDLER, R., MAYER, K., DE BIÈVRE, P.
Measuring larger isotope ratios with a new commercial mass spectrometer at CBNM.
23. Diskussionstagung der ARG Massenspektrometrie, Konstanz, June 5-8, 1990

GOEDTKINDT, P., SALOMÉ, J.-M., ARTRU, X., DHEZ, P., JABLONKA, M., MAENE, N., POORTMANS, F., WARTSKI, L.
Interference effects in X-ray transition radiation with a 500 MeV electron beam.
11th Intern. Conf. Appl. Accelerators in Res. and Industry, Denton, November 5-8, 1990

GOEDTKINDT, P., ARTRU, X., DHEZ, P., MAENE, N., POORTMANS, F., SALOME, J.-M., WARTSKI, L.
X-ray microlithography with a transition radiation source.
Microcircuit Egineering 90, Leuven, September 18-20, 1990

HAMBSCH, F.-J., KNITTER, H.-H., BUDTZ-JØRGENSEN, C.
New results and interpretations of cold fission data from ^{252}Cf.
IAEA Consultants' Meeting on Neutron Data for Neutron Emission in the Fission Process, Vienna, October 22-24, 1990

HAMBSCH, F.-J., KNITTER, H.-H., BUDTZ-JØRGENSEN, C.
New results from ^{252}Cf spontaneous fission. Nuclear mass- and charge distributions in the cold fragmentation region.
6th Intern. School on Neutron Physics, Alushta, October 8-18, 1990

HOFMANN, C., VANDECASTEELE, C., PAUWELS, J.
Calibration in solid sampling Zeeman atomic absorption spectrometry.
4th Intern. Colloq. "Solid Sampling with Atomic Spectroscopy", Jülich, October 8-10, 1990.

INGELBRECHT, C., PEETERMANS, F., DE CORTE, F., DE WISPELAERE, A., VANDECASTELE, C., COURTIJN, E., D'HONDT, P.
Aluminium-gold reference material for the k_0-standardization of neutron activation analysis.
15th World Conf. Intern. Nucl. Target Dev. Soc., Santa Fe, September 10-14, 1990

INGELBRECHT, C., PEETERMANS, F., PAUWELS, J.
Reference materials prepared at CBNM for reactor neutron dosimetry.
7th ASTM-EURATOM Symp. Reactor Dosimetry, Strasbourg, August 27-30, 1990

KNITTER, H.-H., HAMBSCH, F.-J., BROSA, U., BUDTZ-JØRGENSEN, C.
New results in fission: fission modes and bifurcation points - inhomogeneous elasticity of a prescission nucleus-scission neutron production mechanism in ^{252}Cf (SF).
General Meeting Belgian Phys. Soc., Louvain-la-Neuve, May 30-June 1st, 1990

KNITTER, H.-H., HAMBSCH, F.-J., BUDTZ-JØRGENSEN, C.
New results from ^{252}Cf spontaneous fission.
DPG Spring Meeting of the Nuclear Physics Section, Strasbourg, March 26-30, 1990

KRAMER, G.N., PAUWELS, J.
The development of cryo-grinding equipment for the transformation of large quantities of biological reference materials with low heavy metal contents.
4th Intern. Symp. Biol. Environm. Ref. Mat., Orlando, February 5-8, 1990;
Fresenius' J. Anal.Chem. 338 (1990) 390

MOUCHEL, D.
A high-purity-Ge detector system for the measurement of low-level radioactivity in environmental samples.
2nd Intern. Summer School on Low-Level Measurements of Man-Made Radionuclides in the Environment, La Rábida, Hùelva, June 24-July 6, 1990

PAUWELS, J., KRAMER, G.N., DE ANGELIS, L., GROBECKER, K.H.
The preparation of codfish candidate reference material to be certified for Pb, Cd, Hg, Fe and Zn.
4th Intern. Symp. Biol. Environm. Ref. Mat., Orlando, February 5-8, 1990
Fresenius' J. Anal.Chem. 338 (1990) 515

PAUWELS, J., DE ANGELIS, L., GROBECKER, K.H.
Solid sampling Zeeman atomic absorption spectrometry in production and use of certified reference materials.
4th Intern. Colloq. "Solid Sampling with Atomic Spectroscopy", Jülich, October 8-10, 1990

PAUWELS, J., EYKENS, R., LAMBERTY, A., VAN GESTEL, J., TAGZIRIA, H., SCOTT, R.D., BYRNE, J., DAWBER, P.G., GILLIAM, D.M.
The preparation and characterization of ^{6}LiF and ^{10}B reference deposits for the measurement of the neutron lifetime.
15th World Conf. Intern. Nucl. Target Dev. Soc., Santa Fe, September 10.14, 1990

PAUWELS, J.
Specific nuclear target preparation and characterization techniques used at CBNM.
11th Intern.Conf. Appl. Accelerators in Res. and Industry, Denton, November 5-8, 1990

PINHEIRO, M.T., TAPPER, U.A.S., STURESSON, K., BROWN, A.
Experimental investigations on sample preparation of biological specimens to nuclear microprobe analysis.
Intern. Conf. Nucl. Microprobe Techn. and Appl., Melbourne, February 5-9, 1990

REHER, D.
Progress report on the EUROMET project n° 219 : "Metrology of ^{192}Ir Brachytherapy Sources".
EUROMET Workshop Ion. Rad. Radioactivity, Teddington, October 7-10, 1990

ROHR, G.
The nuclear reaction mechanism studied with neutron resonances.
20th Intern. Symp. Nucl. Phys. - Nucl. React. Mechanisms, Gaussig, November 12-16, 1990

ROHR, G.
Gaussian distribution for spacings of simple neutron resources.
7th Intern. Symp. Conf. Gamma-Ray Spectrosc. and Rel. Topics, Asilomar, October 14-19, 1990

SWIETLICKI, E.
PIXE and its capabilities per source apposition.
2nd European Aerosol Conf., Zürich, October 1st-5, 1990

SWIETICKI, E., LÖVESTAM, G., WÄTJEN, U.
Proton microprobe single particle analysis.
2nd European Aerosol Conf., Zürich, October 1st-5, 1990

TAGZIRIA, H., PAUWELS, J., VERDONCK, J., VAN GESTEL, J., EYKENS, R., GILLIAM, D.M., SCOTT, R.D., BYRNE, J., DAWBER, P.G.
Problems related to the determination of mass densities of evaporated reference deposits.
15th World Conf. Intern. Nucl. Target Dev. Soc., Santa Fe, September 10-14, 1990

WAGEMANS, C., SCHILLEBEECKX, P., DERUYTTER, A.J., BARTHÉLÉMY, R.
The subthermal neutron induced fission cross sections of the common fissile isotopes and their impact on the Westcott g_f-factor.
Intern. Conf. Phys. of Reactors: Operation, Design and Computation, Marseille, April 23-26, 1990

WAGEMANS, C., DRUYTS, S., WEIGMANN, H., BARTHÉLÉMY, R., SCHILLEBEECKX, P., GELTENBORT, P.
Study of (n,p) and (n,α) reactions related to astrophysics.
General Meeting Belgian Phys. Soc., Louvain-la-Neuve, May 30-June 1st, 1990

WÄTJEN, U., BAX, H., RIETVELD, P.
Evaporated and implanted reference layers for calibration in ion beam analysis.
11th Intern. Conf. Appl. Accelerator in Res. and Industry, Denton, November 5-8, 1990

WEIGMANN, H., KECK, B., WARTENA, J.A., GELTENBORT, P., SCHRECKENBACH, K.
Measurement of eta of ^{235}U for subthermal neutron energies.
Intern. Conf. Phys. of Reactors: Operation, Design and Computation, Marseille, April 23-26, 1990

SCIENTIFIC OR TECHNICAL ARTICLES

ALTZITZOGLOU, T.
The half-life of ^{125}I.
Appl. Radiat. Isot., Int. J. Radiat. Appl. Instrum., Part A (to be published)

BÖCKHOFF, K.H., CARLSON, A.D., WASSON, O.A., HARVEY, J.A., LARSON, D.C.
Electron linear accelerators for fast neutron data measurements in support of fusion energy applications.
Nucl. Sci. Eng. 106 (1990) 192

BORTELS, G., MOUCHEL, D., EYKENS, R., GARCÍA-TORAÑO, E., ACEÑA, M.L., WILTSHIRE, R.A.P., KING, M., FUDGE, A.J.
Alpha-particle emission probabilities in the decay of ^{237}Np.
Nucl. Instr. Methods, Phys. Res. A295 (1990) 199

BORTELS, G., MOUCHEL, D., GARCÍA-TORAÑO, E., ACEÑA, M.L.
Synthetic doublets from ^{234}U alpha-particle spectra for assessing in peak fitting.
Nucl. Instr. Methods, Phys. Res. A286 (1990) 429

BYRNE, J., DAWBER, P.G., SPAIN, J.A., WILLIAMS, A.P., DEWEY, M.S., GILLIAM, D.M., GREENE, G.L., LAMAZE, G.P., SCOTT, R.D., PAUWELS, J., EYKENS, R., LAMBERTY, A.
Measurements of the neutron lifetime by counting trapped protons.
Phys. Rev. Lett. 65 (1990) 289

CORVI, F., FIONI, G., GASPERINI, F., SMITH, P.B.
The weighting function of a neutron capture detection system.
Nucl. Sci. Eng. 107 (1991) 272

COURSEY, B.M., VANINBROUKX, R., REHER, D., HUTCHINSON, J.M.R., MULLEN, P.A., DEBERTIN, K., GEHRKE, R.J., ROGERS, J.W., KOEPPEN, L.D., SMITH, D.
The standardization of ^{93m}Nb for use as reference material for reactor neutron dosimetry.
Nucl. Instr. Methods, Phys. Res. A290 (1990) 537

DE BIÈVRE, P.
Isotope dilution mass spectrometry; what can it contribute to accuracy in trace analysis ?
Fresenius' J. Anal. Chem. 337 (1990) 766

DENECKE, B., BAMBYNEK, W., GROSSE, G., WÄTJEN, U., BALLAUX, C.
A set of X-ray fluorenscence reference sources for the intrinsic efficiency calibration of Si(Li) detectors down to 1 keV.
Nucl. Instr. Methods, Phys. Res. B49 (1990) 152

DENECKE, B., GROSSE, G., WÄTJEN, U., BAMBYNEK, W., BALLAUX, C.
Efficiency calibration of Si(Li) detectors with X-ray reference sources of energies between 1 and 5 keV. Nucl. Instr. Methods, Phys. Res. A286 (1990) 474

HELLIN, H., PAULSEN, A., LISKIEN, H., DECATTE, G., WAMBERSIE, A., LEONARD, A., BAUGNET-MAHIEU, L.
Chromosome aberrations induced in vitro in human lymphocytes by monoenergetic 2.5 MeV neutrons and ^{60}Co gamma rays.
Strahlentherapie und Onkologie (to be published)

KNITTER, H.H.
Fission fragment angular distribution.
in: The Nuclear Fission Process, CRC Press, Boca Raton, Florida, USA

KNITTER, H.H., BROSA, U., BUDTZ-JØRGENSEN, C.
Neutron and gamma emission in fission.
in: The Nuclear Fission Process, CRC Press, Boca Raton, Florida, USA

KNITTER, H.-H., HAMBSCH, F.-J., BUDTZ-JØRGENSEN, C.
Nuclear mass and charge distributions in the cold region of the spontaneous fission of ^{252}Cf.
Nucl. Phys. (to be published)

LAMBERTY, A., PAUWELS, J.
How to correct for blanks in isotope dilution mass spectrometry.
Intern. J. Mass Spectrom. Ion Proc. 104 (1991) 45

LAMBERTY, A., EYKENS, R., TAGZIRIA, H., PAUWELS, J., DE BIÈVRE, P., SCOTT, R.D., BYRNE, J., DAWBER, P.G., GILLIAM, D.M., GREENE, G.L.
Recalibration of CBNM reference boron deposits used in the measurement of the neutron lifetime.
Nucl. Instr. Methods, Phys. Res. A294 (1990) 393

LE DUIGOU, Y.
Certification of a plutonium dioxide reference material for chemical analyses.
EUR 12893 EN (1990)

LEIDERT, W.
Accurate coulometric determination of the uranium content of uranium reference materials.
EUR 12880 EN (1990)

LIEVENS, F., PAUWELS, J., INGELBRECHT, C.
Certification of an aluminium metal reference material for neutron dosimetry (EC-NRM 523).EUR 12895 EN

LIEVENS, F., INGELBRECHT, C., PAUWELS, J., SCHULZE, K.
Certification of a niobium metal reference material for neutron dosimetry (EC-NRM 526).EUR 12894 EN

LIEVENS, F., INGELBRECHT, C., PAUWELS, J.
Certification of a niobium metal reference material for neutron dosimetry (EC-NRM 525).EUR 12892 EN

LISKIEN, H., WÖLFLE, R., WIDERA, R., QAIM, S.M.
Excitation functions of (n,α) and (n,x) reactions on molybdenum isotopes.
Appl. Rad. Isot., Int. J. Radiat. Appl. Instrum., A 41 (1990) 83

MARTIN, P.W., BICHARD, J.W., BUDTZ-JØRGENSEN, C.
Studies of graphite intercalation compounds using time-differential perturbed angular distributions.
J. Chem. Phys. 93 (1990) 6092

PAUWELS, J., DE ANGELIS, L., PEETERMANS, F., INGELBRECHT, C.
Determination of traces of silver in copper by direct Zeeman graphite furnace atomic absorption spectrometry.
Fresenius' J. Anal. Chem. 337 (1990) 290

PAUWELS, J., INGELBRECHT, C.
Samples and targets for nuclear measurements delivered by CBNM during 1990.
INTDS Newsletter (to be published)

REHER, D.F.G., MERLO, P.
A quality control exercise of radionuclide calibrators among Belgian hospitals.
Eur. J. Nucl. Med. 17 (1990) 103

STÜBER, W.
Eine einfache Anordnung zur Eichung von Hochfrequenzoszillatoren mit Hilfe des Senders DCF77.
Elektronik 2 (1990) 78

WÄTJEN, U., DANNECKER, W., KRIEWS, M.
On the status of preparing an aerosol filter reference material for elemental analysis.
Nucl. Instr. Methods, Phys. Res. B (to be published)

WEIGMANN, H.
Fission barriers.
in: The Nuclear Fission Process, CRC Press, Boca Raton, Florida, USA

WEIGMANN, H.
Neutron induced fission cross sections.
in: The Nuclear Fission Process, CRC Press, Boca Raton, Florida, USA

WÖLFLE, R., QAIM, S.M., LISKIEN, H., WIDERA, R.
Systematic studies of excitation functions of (n,t) reactions on medium and heavy mass nuclei.
Radiochimica Acta 50 (1990) 5

SPECIAL PUBLICATIONS

HANSEN, H.H. (ed.)
Annual Progress Report on Nuclear Measurements and Reference Materials, January - December 1989, EUR 12615 EN (1990)

HANSEN, H.H. (ed.)
Annual Progress Report on Nuclear Data 1989.
NEANDC (E) 312 "U", Vol. III Euratom ; INDC (EUR) 024/G (1990)
EUR 12822 EN (1990)

GLOSSARY

AERE	Atomic Energy Research Establishment, Harwell (GB)
ALS	Accélérateur Linéaire de Saclay, Saclay (F)
ANL	Argonne National Laboratory, Argonne (USA)
BAL	Broncho-Alveolar Lavages
BIPM	Bureau International des Poids et Mesures, Sèvres (F)
CBNM	Central Bureau for Nuclear Measurements (JRC-Geel), Geel (B)
CEA	Commissariat à l'Energie Atomique, Paris (F)
CEC	Commission of the European Communities
CERN	Centre Européen pour la Recherche Nucléaire
CIEMAT	Centro de Investigación Energética, Medio Ambiental y Tecnología
CRNS	Centre National de la Recherche Scientifique
CRP	Coordinated Research Programme
DFN	Deutsches Forschungsnetz
DG	Direction Générale
DPh N/ STAS	Département de Physique Nucléaire / Services des Techniques d'Accélération Supraconductrice, Saclay (F)
DPSV	Differential Pulse Stripping Voltametry
EC	European Community
ECN	Energieonderzoek Centrum Nederland, Petten (NL)
ECSAM	European Commission's Safeguards Analytical Measurements
EFF	European Fusion File
ENDF	Evaluated Nuclear Data File
ENEA	Comitato Nazionale : Energia Nucleare e Energia Alternative
ESARDA	European Safeguards Research and Development Association
ETAAS	Electrothermal Atomic Absorption Spectrometry
ETL	Electrotechnical Laboratory, Ibaraki (Japan)
EWGRD	European Working Group on Reactor Dosimetry
FWHM	Full Width at Half Maximum
GELINA	Geel Electron Linear Accelerator
IAEA	International Atomic Energy Agency, Vienna (A)
ICP	Inductive Coupled Plasma
ICRM	International Committee for Radionuclide Metrology
IDMS	Isotope Dilution Mass Spectrometry
ILL	Institut Laue-Langevin, Grenoble (F)
INDC	International Nuclear Data Committee
IPN	Institut de Physique Nucléaire, Lyon (F)
IRK	Institut für Radiumforschung und Kernphysik, Wien (A)
IRM	Isotope Reference Material
JEF	Joint European File

JENDL	Japanese Evaluated Data Library
JRC	Joint Research Centre
KFA	Kernforschungsanlage, Jülich (D)
KFK	Kernforschungszentrum Karlsruhe, Karlsruhe (D)
KU	Katholieke Universiteit, Leuven (B)
LEU	Low Enriched Uranium
LSAI	Laboratoire de Spectrométrie Atomique et Ionique
LURE	Laboratoire pour l'Utilisation du Rayonnement Electromagnétique
MOX	Mixed Oxide
NBS	National Bureau of Standards, Gaithersburg (USA)
NCG	Nuclear Certification Group
NEA	Nuclear Energy Agency, Paris (F)
NEANDC	Nuclear Energy Agency's Nuclear Data Committee
NIRAS/ ONDRAF	Nationale Instelling voor Radioactief Afval en Splijtstoffen, / Organisme National des Déchets Radioactifs et des Matières Fissiles, Brussels (B)
NIST	National Institute of Standards and Technology, Gaithersburg (USA)
NPL	National Physical Laboratory, Teddington (GB)
NRM	Nuclear Reference Material
NTI	Negative Thermal Ionization
PHYSOR	International Conference on the Physics of Reactors, 1990, Marseille (F)
PIXE	Particle Induced X-Ray Emission
PTB	Physikalisch-Technische Bundesanstalt, Braunschweig (FRG)
RBS	Rutherford Backscattering
REIMEP	Regular European Interlaboratory Measurement Evaluation Programme
RIVM	Rijksuniversiteit voor Volksgezondheid en Milieuhygiëne, Bilthoven (NL)
RM	Reference Material
RUG	Rijksuniversiteit Gent, Ghent (B)
SCK/CEN	Studiecentrum voor Kernenergie/ Centre d'Etudes Nucléaires, Mol (B)
SIR	Système International de Référence
SURRC	Scottish Universities Research and Reactor Centre (UK)
TBT	tributyltin
TH	Technische Hochschule
TOF	Time of Flight
TR	Transition Radiation
WRENDA	World Request List for Neutron Data Measurements

European Communities - Commission

EUR 13456 - ANNUAL REPORT 90 of the
Central Bureau for Nuclear Measurements

H.H. Hansen (ed.)

Luxembourg: Office for Official Publications of the European Communities

1991 - pag. 88 - 21.0 x 29.7 cm

EN