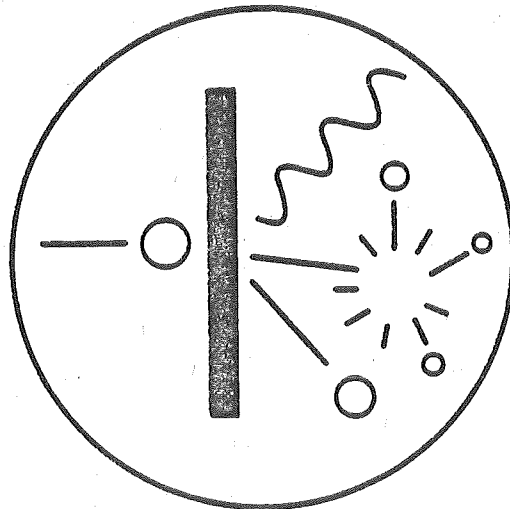


**INTERNATIONAL NUCLEAR
TARGET DEVELOPMENT SOCIETY**

NEWSLETTER



AUGUST 1985

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The INTDS Newsletter is an informal source of information for and from the Membership.

The INTDS assumes no responsibility for the statements and opinions advanced by the contributions.

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E D I T O R I A L

Dear Colleagues,

It's almost 1 year ago that most of us met at the INTDS Conference in Antwerpen. The proceedings of this conference have been published 1 June 1985 in Nuclear Instruments and Methods in Physics Research, Vol. A 236 N°3 and the members received meanwhile their copy. This quick publication was only possible thanks to the efforts of all contributors and the hard work of the reviewers.

Joe Gallant together with Peter Dmytrenko have started the organisation of the 13th Conference at the Chalk River Nuclear Laboratories, Canada on 7 - 9 September 1986 as you will read in this Newsletter.

We are sure that this Conference will be a succes and I hope to see all of you at Chalk River.

J. VAN AUDENHOVE



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Chalk River
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AWARD TO J.L.GALLANT

IT IS MY PLEASURE TO ANNOUNCE THAT JOE GALLANT IS A RECIPIENT OF A NEWLY FORMED 'DISCOVERY AWARD' FOR THE INNOVATIVE DEVELOPMENT OF TRITIUM SOURCES FOR DOCUMENT IMAGING. THE AWARDS ARE GIVEN TO AECL EMPLOYEES WHO DEVELOP IDEAS THAT HAVE COMMERCIAL POTENTIAL. THE AWARD CONSISTS OF A COMMEMORATIVE PLAQUE AND A CASH AWARD.

THE ROYAL CANADIAN MOUNTED POLICE(RCMP) HAVE CONTRACTED AND RECEIVED TRITIUM SOURCES THAT ARE CURRENTLY UNDERGOING FIELD TESTS. THE PRELIMINARY DATA OBTAINED FROM RCMP TESTS HAS AROUSED CONSIDERABLE INTEREST FROM OTHER POLICE FORCES THAT EMPLOY FORENSIC TECHNIQUES. THE TRITIUM SOURCES PROVIDE AN ECONOMICAL AND HIGHLY PORTABLE METHOD FOR EXAMINING DOCUMENTS AND CURRENCY FOR FORGERIES AND ALTERATIONS.

SHOWN IN THE ENCLOSED PHOTOGRAPH IS JOE RECEIVING HIS AWARD FROM BOB HART, EXECUTIVE VICE-PRESIDENT OF AECL.



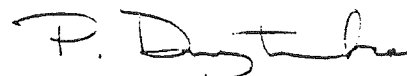
J.L. GALLANT RETIREMENT

EFFECTIVE AUGUST 15, 1985 JOE GALLANT WILL RETIRE FROM HIS POSITION OF BEING IN CHARGE OF THE TARGET LABORATORY. HIS DECISION TO LEAVE AT THIS TIME WAS A RESULT OF AN EARLY RETIREMENT PROGRAM IMPLEMENTED BECAUSE OF BUDGET CUTS AT CHALK RIVER. IT IS HOPED THAT HE WILL BE AVAILABLE ON A CONTRACTUAL BASIS TO PRODUCE TARGETS AT CHALK RIVER IF DEMANDS EXCEED THE CAPABILITIES OF A SINGLE TARGET-MAKER. IN ANY EVENT JOE HAS AGREED TO MAKE HIMSELF AVAILABLE TO HELP IN ORGANIZING THE 1986 INTERNATIONAL INTDS CONFERENCE AT CHALK RIVER.

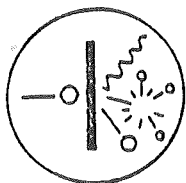
THREAT TO CHALK RIVER NUCLEAR LABORATORIES

IN ITS RECENT BUDGET, THE CANADIAN FEDERAL GOVERNMENT HAS ANNOUNCED A REDUCTION IN FUNDING FOR AECL'S RESEARCH AND DEVELOPMENT PROGRAM FROM THE PRESENT LEVEL OF 200 MILLION DOLLARS PER YEAR TO 100 MILLION DOLLARS PER YEAR BY 1990. SUCH A DRASTIC CUT IS ALMOST CERTAIN TO HAVE A SERIOUS EFFECT ON THE CHALK RIVER NUCLEAR LABORATORIES, WHERE IT IS LIKELY TO MEAN A REDUCTION OF 40% IN THE TOTAL BUDGET.

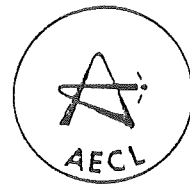
YOURS SINCERELY



PETER DMYTRENKO



13th WORLD CONFERENCE OF THE
INTERNATIONAL NUCLEAR TARGET DEVELOPMENT SOCIETY
SPONSORED BY THE CHALK RIVER NUCLEAR LABORATORIES
CHALK RIVER, ONTARIO, CANADA KOJ 1JO
SEPTEMBER 17-19, 1986



TELEX: 053-34555
TELEPHONE: 613-584-3311

CO-CHAIRMEN: J. L. GALLANT
P. DMYTRENKO

JULY 12, 1985

DEAR MEMBERS

IT IS A PLEASURE TO ANNOUNCE THAT THE 13TH WORLD CONFERENCE OF THE INTERNATIONAL NUCLEAR TARGET DEVELOPMENT SOCIETY WILL BE HELD AT THE CHALK RIVER NUCLEAR LABORATORIES, CHALK RIVER, ONTARIO, CANADA ON SEPTEMBER 7-9, 1986.

WE ARE LOOKING FORWARD TO YOUR PRESENCE AND PARTICIPATION. A FIRST DETAILED ANNOUNCEMENT WILL BE FORWARDED SHORTLY.

PLEASE ADDRESS ALL CORRESPONDENCE TO THE CONFERENCE SECRETARY:

MRS. JUNE ELLIOTT
NUCLEAR PHYSICS
CHALK RIVER NUCLEAR LABORATORIES
CHALK RIVER, ONTARIO, CANADA KOJ 1JO

A.H.F. Muggleton,
Department of Nuclear Physics,
Research School of Physical Sciences,
The Australian National University,
P.O. Box 4, Canberra, ACT 2601,
Australia.

Activity Report

Targets produced during the past year varied in thickness from a few $\mu\text{g}/\text{cm}^2$ to several mg/cm^2 and were as follows:

Ag, Au, BaCl_2 , $^{134}\text{BaCl}_2$, $^{135}\text{BaCl}_2$, Bi_2O_3 , C, ^{48}Ca , ^{164}Dy , Er, ^{166}Er , ^{170}Er ,
Gd, ^{156}Gd , ^{157}Gd , Ho, In, La, Lu, ^{24}Mg , ^{142}Nd , ^{150}Nd , Os, ^{208}Pb , ^{208}Pbs ,
 ^{141}Pr , ^{194}Pt , ^{196}Pt , ^{198}Pt , S, Ag_2 ^{32}S , Ag_2 ^{34}S , Ag_2 ^{36}S , ^{144}Sm , ^{147}Sm , ^{150}Sm , ^{152}Sm ,
 ^{154}Sm , ^{116}Sn , ^{118}Sn , ^{120}Sn , ^{122}Sn , ^{124}Sn , Ta, ^{130}Te , Th, ^{180}Tm , $^{182}\text{Wo}_3$.

Approximately 800 carbon stripper foils, 3-5 $\mu\text{g}/\text{cm}^2$ thick, were produced as well as routine carbon foils for target backings. Several self-supporting carbon foils, 1 $\mu\text{g}/\text{cm}^2$ thick, were fabricated for charge distribution experiments.

^{36}S Targets

A number of very neutron rich nuclei may be produced in nuclear reactions between heavy ions and a ^{36}S target, but until recently there was no commercial source of ^{36}S isotope with sufficient enrichment to make such experiments feasible. However, sulphur enriched to 82% in ^{36}S is now available at a cost of \sim \$200 per mg. Hence a method is required for making sulphur targets which will withstand heavy-ion bombardment. Such a method, which is extremely economical in its use of the expensive isotope, was described by Peter Maier-Komor at the 11th Conference of the INTDS, Seattle, 1982. Apparatus similar to that used by Maier-Komor

has been designed and built at ANU. Briefly, targets of Ag_2S are produced by first evaporating a silver layer onto a mounted carbon backing, and then placing the resulting targets in a small vacuum vessel with $<1\text{mg}$ of the enriched elemental sulphur. The vessel is evacuated and placed in an oven at $\sim 120^\circ\text{C}$ when the silver is completely converted to silver sulphide. Targets of ^{32}S , ^{34}S and ^{36}S have been successfully produced using this technique with thicknesses of the Ag_2S layer ranging from 175 to $300\ \mu\text{g}/\text{cm}^2$.

Thin Osmium Targets.

Thin isotopic osmium targets, ($<1\text{mg}/\text{cm}^2$), were produced by the electroplating technique described by Stuchbery (Stuchbery A.E., Nucl.Inst. and Methods. 211 (1983). 293-295).

A small quantity ($\sim 12\text{mg}$) of isotopically enriched osmium powder was placed in a pyrex glass bulb and heated in an airstream to convert it to OsO_4 gas. The gas was collected in $\sim 10\ \text{ml}$ of an aqueous solution containing equal quantities of NaOH and $\text{H}_2\text{HPO}_4 \cdot \text{H}_2\text{O}$ (each $3\text{g}/80\ \text{ml}$). This procedure was carried out in a fume-cupboard as OsO_4 is highly toxic. After collection the solution was normally yellowish-brown in colour. Approximately 116mg of sulphamic acid ($\text{H}_2\text{N}_2\text{SO}_3 \cdot \text{H}$) was added to the solution followed by the same weight of potassium hydroxide (KOH).

The plating solution was transferred to a small plating cell consisting of a glass tube with an aperture the diameter of the target required (10mm diameter). A copper foil $125\ \mu\text{m}$ thick was used as the target backing and a small piece of platinum used as the anode.

Plating current was applied to the plating cell immediately the solution was admitted. The plating solution was maintained at $70\text{-}80^\circ\text{C}$ throughout the plating cycle. Plating parameters were: heater watts 24w , plating current 20mA , area of deposit 2.7cm^2 , plating time $1\ \text{hr}$. In all cases a black powdery deposit formed on the plated surface. To increase the film thickness the powdery deposit was washed

off with distilled water and the plating process repeated.

Finally the copper backed foils were thoroughly rinsed in distilled water and a $10\mu\text{g}/\text{cm}^2$ carbon film evaporated onto the plated surface. The copper backing was removed using a standard trichloroacetic solvent and the carbon backed osmium films mounted on target frames in the normal way.

Joint Research Centre, CBNM, B - 2440 Geel, Belgium
Sample Preparation

THE PREPARATION OF A HIGH QUALITY TARGET FOR THE STUDY OF THE CHARACTERISTICS
OF ^{242}Pu (s.f.) FRAGMENTS

In 1981 a study of the energy- and mass characteristics of the ^{242}Pu (s.f.) fragments was carried out at CBNM (1). The results of this study have now been improved significantly, as both the energy - and mass resolution have been ameliorated by more than a factor of 2. Thus the influence of the neutron shells could be demonstrated and explained in a more precise and convincing way (2). The pre-neutron emission fission fragment mass distributions for ^{242}Pu (s.f.) as determined in the 1981 study (1) and in the 1985 study (2) are shown in Fig. 1. In the latter, fine structure due to neutron shells with 82 and 88 neutrons can be clearly observed. Also the improvement in mass resolution is clearly illustrated by this Figure.

These results were obtained after significant improvement of the quality of the $^{239-242}\text{Pu}$ target used, which was made possible by :

1. The realisation of exceptionally thin polyimide foils ($16 \mu\text{g}/\text{cm}^2$) by a factor of 2 below the limits suggested by the rupture constant of polyimides (3). Due to this improvement, the energy loss in each substrate foil could be reduced from 2.4 to 1.2 MeV for 70 MeV ^{141}Pr , and from 2.2 to 1.0 MeV for 98 MeV ^{98}Tc .
2. The development of the dry hydrofluorination of plutonium (4), which made it possible to prepare plutonium deposits on such thin plastic foils using vacuum deposition, as PuF_3 evaporates at ca. 1000°C lower temperature as PuO_2
3. The use of the sandwich technique for the production of mixed targets, as described by Mast (5).

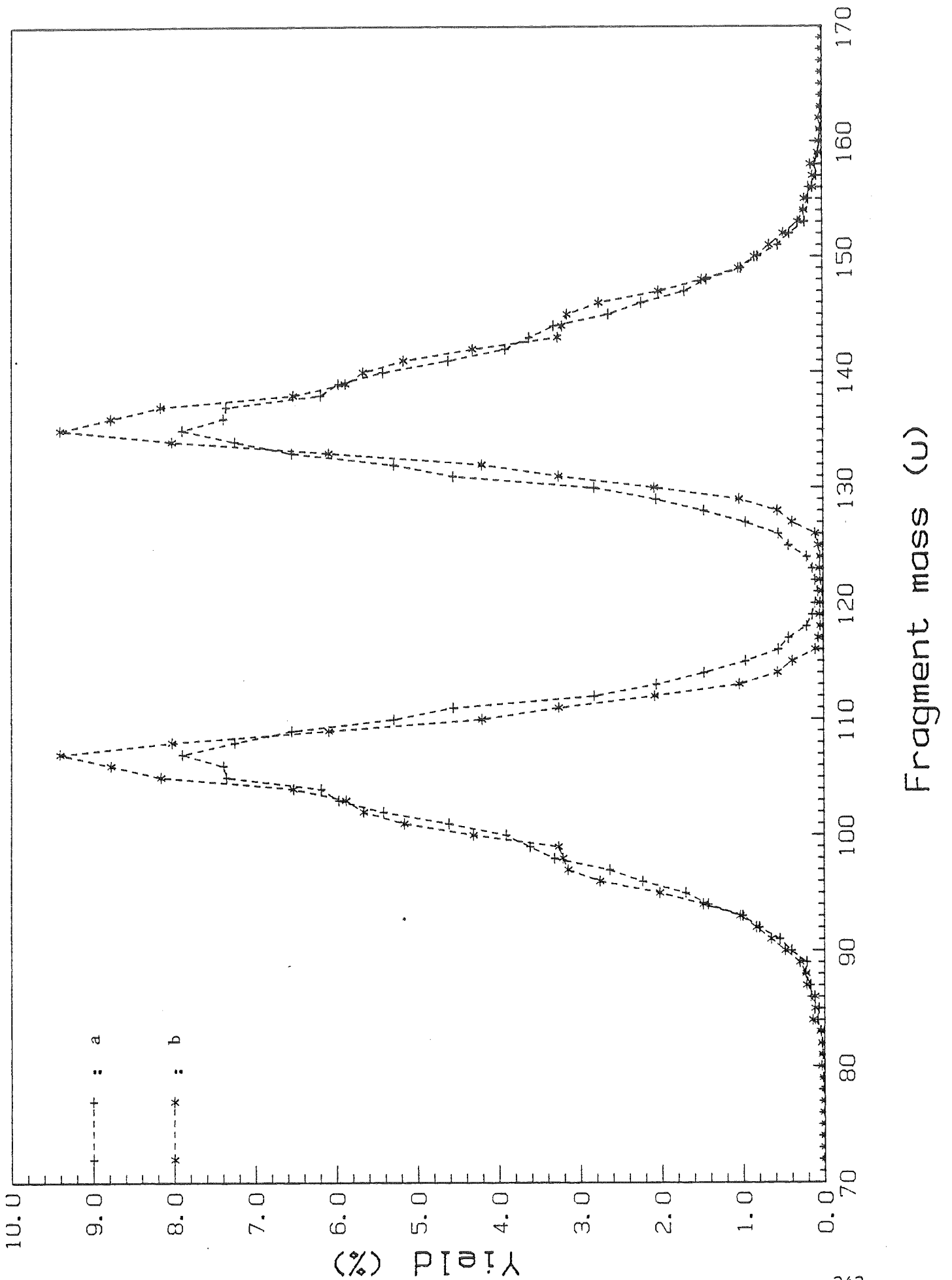


Fig. 1 : Pre-neutron emission fission fragment mass-distributions for ^{242}Pu (s.f.) as determined (a) by Allaert e.a. (1) and (b) by Schillebeeckx e.a. (2).

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- (1) E. Allaert, C. Wagemans, G. Wegener-Penning, A.J. Deruytter and R. Barthélémy.
Nuclear Physics, A380 (1982) 61
- (2) P. Schillebeeckx, C. Wagemans, A.J. Deruytter and R. Barthélémy
Proc. 4th Int. Symp. on Neutron Induced Reactions, Smolenice, June 1985
- (3) J. Van Gestel, J. Pauwels and J. Van Audenhove
Preparation of Nuclear Targets for Particle Accelerators,
Plenum Press, New-York and London (1981), 117
- (4) R. Eykens, J. Pauwels and J. Van Audenhove
Nucl. Instr. and Methods, A 236, (1985) - 499
- (5) H. Mast and J. Pauwels
Newsletter of the INDTS, June 1982, 10

Preparations of Heavy-Ion Targets and Developments of Preparation Procedures*

H. Folger, W. Hartmann, J. Klemm and W. Thalheimer, GSI Darmstadt
B. Goertz, Institut für Kernchemie, Universität Mainz

100 different enriched isotopes, elements, and chemical compounds were taken as basis materials in the fabrication of 13,135 heavy-ion targets, absorbers, degraders, or stripper foils in 1984. The thicknesses ranged from 0.002 to 200 mg/cm^2 , requiring various preparation and measuring methods. A comparison of target production rates at GSI in fig. 1 shows significant increases in the demands for carbon films and evaporated layers from 1980 to 1984, mainly due to the increasing need for large target areas on rotating wheels in high-intensity heavy-ion bombardments at the UNILAC. (Targetnumbers normalized to areas of 20x20mm.)

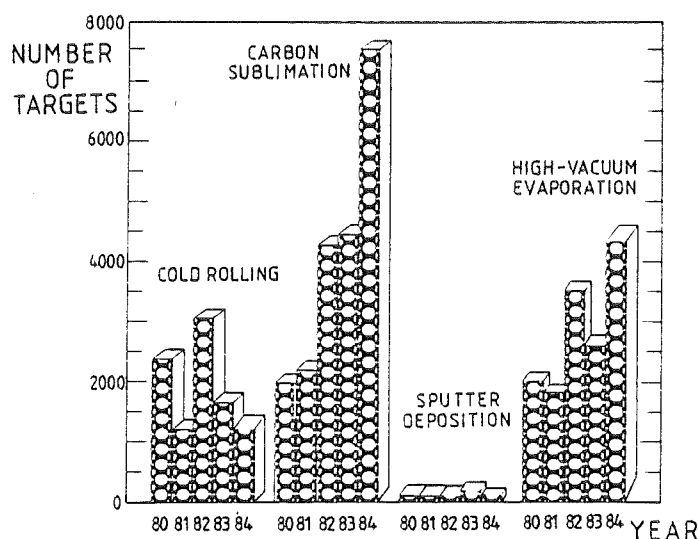


Fig. 1: Target production rates from 4 different preparation techniques from 1980 to 1984.

Cold rolling between 0.5mm thick stainless steel is a very effective way to prepare ductile targets of area-densities of more than 0.5 mg/cm^2 ; moreover, the procedure became important in the fabrication of numerous 0.5 μm thick Ti backings. The use of one rolling mill in a glove-box system allows to handle sandwich targets like Ti/U/Ti as well as milligram amounts of oxygen-sensitive isotopic substances as obtained from high-vacuum reduction-distillations. The list of rolled targets contains 45 different materials in 1984:

Ag, ¹⁰⁹Ag, Al, Au, Ca, ^{40,48}Ca, Cu, ¹⁶⁴Dy, ¹⁷⁰Er, Fe, Gd, ^{160,162}Gd, Mg, ^{24,25,26}Mg, Mo, ^{94,98}Mo, Nd, ¹⁴²Nd, ¹⁴⁴Nd, Ni, ^{58,64}Ni, Pb, ¹⁹⁸Pt, Re, Rh, ^{148,152,154}Sm, Sn, Ta, Th, Ti, ⁴⁸Ti, Tm, Ti/U/Ti, V, W, Y, and Zr.

By high-vacuum carbon sublimation-condensations large amounts (see fig. 1) of target backings (mostly 0.04 mg/cm^2), protective layers, stripper foils (0.04 mg/cm^2), or post-stripper foils (0.1–0.4 mg/cm^2) are prepared. All foils of 0.002 to 0.1 mg/cm^2 are obtained from resistive heating of spectrographite rods. Layers of 0.1 to 1 mg/cm^2 are sublimed and condensed during electron bombardments of carbon rods at 10 kV from a 6 kW e-gun.

Focused heavy-ion beam sputter depositions are performed in a high-vacuum chamber using an extracted Ar⁺-beam of 1 mA at 10 kV. Recent and former GSI data show (see fig. 2) that the sputtering yield is relatively high, with a decreasing tendency towards higher target mass numbers. Especially small

* This contribution was taken from GSI, Scientific Report 1984, GSI 85-1, ISSN 0174-0814, p. 269. The typeset for the INTDS-NEWSLETTER was performed by M. Feyerabend, GSI Rechenzentrum, using T_EX.

amounts of high-melting target materials can efficiently be converted into thin target layers. The sputtered targets were in 1984:

$^{109}\text{Ag}/\text{C}$, $^{100}\text{Mo}/\text{C}$, Nb/C , $^{108}\text{Pd}/\text{C}$, $^{110}\text{Pd}/\text{C}$, Rh/C , ^{96}Ru , $^{104}\text{Ru}/\text{C}$, Si/Ta , $^{28}\text{SiO}_2/\text{C}$, Ta/C , Th/C , $^{186}\text{W}/\text{Be}$, Zr/C , $^{90}\text{Zr}/\text{Au}$, $^{90}\text{Zr}/^{208}\text{Pb}$, $^{92}\text{ZrO}_2/\text{Au}$, and $^{96}\text{Zr}/\text{C}$.

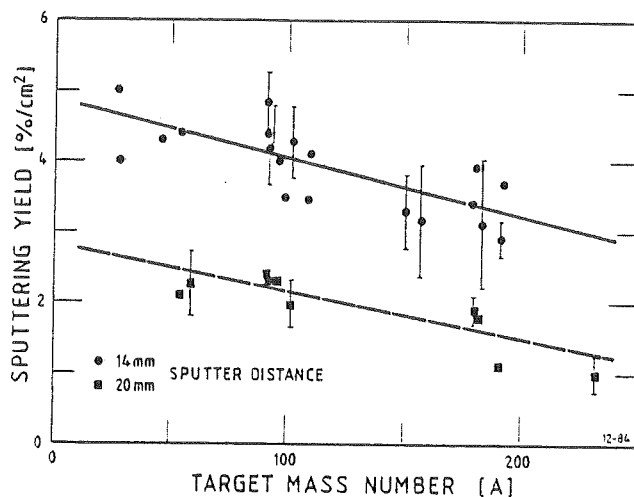


Fig. 2: Sputtering yield as a function of the target mass number measured from sputter depositions.

In a variety of high-vacuum evaporation-condensations self-supported films or targets on backings were prepared in area densities of 0.002 up to 200 mg/cm^2 . In 1984 the list of 78 evaporated target materials includes poisonous elements like ^{205}Tl , chemical compounds like Eu_2O_3 , enriched isotopes as Na^{37}Cl , and radioactive U:

Ag , ^{109}Ag , Al , Au , Au/C , $\text{Au}/\text{CR39}$, Au/Zr , B/Au , B/C , B/Ta , $^{138}\text{BaF}_2/\text{C}$, Bi , $\text{Bi}/\text{CR39}$, $\text{C}/\text{Bi}/\text{C}$, CaF_2/C , ^{114}Cd , $^{116}\text{Cd}/\text{C}$, ^{50}Cr , Cu , $\text{Eu}_2\text{O}_2/\text{Al}$, $\text{Eu}_2\text{O}_2/\text{C}$, Fe , Fe/C , ^{74}Ge , Ge/C , Gd/C , GdF_3/C , $\text{Gd}_2\text{O}_3/\text{C}$, KCl/C , KF/C , $^{39}\text{KF}/\text{C}$, LiCl/C , LiF/C , MgO/C , $\text{Na}^{37}\text{Cl}/\text{C}$, NaF/C , $^{143}\text{Nd}/\text{C}$, Ni , Ni/C , Pb , ^{206}Pb , $^{207,208}\text{Pb}$, $^{208}\text{Pb}/\text{C}$, $\text{C}/^{208}\text{Pb}/\text{C}$, $\text{C}/^{208}\text{Pb}/\text{Ti}$, Sc/C , $^{28}\text{Si}/\text{Au}$, $^{29}\text{Si}/\text{Au}$, Sm/C , $^{144,148,152}\text{Sm}/\text{C}$, $^{116,118,120,122,124}\text{Sn}$, $^{118,120}\text{Sn}/\text{C}$, $^{88}\text{SrF}_2/\text{C}$, $\text{C}/^{130}\text{Te}/\text{C}$, Ti , Ti/C , $\text{Ti}/\text{C}/\text{Ti}$, $\text{C}/\text{Ti}/\text{C}$, $\text{C}/^{205}\text{Tl}/\text{C}$, $\text{C}/\text{U}/\text{C}$, $\text{GLASS}/\text{Ni}/\text{U}/\text{Ni}$, UF_4/C , W , W/C , Y , Yb/C , $\text{Yb}_2\text{O}_3/\text{C}$, Zr , Zr/C , and ZnS/C .

Progress has been achieved in the preparation of large areas of very homogenous targets on rotating wheels, obtaining high deposition yields as can be seen from table 1.

Table 1
Results from target preparations using rotating wheels.

| ISOTOPE | RUNS | EVAPOR. MATERIAL (mg) | S-T-S † (mm) | TARGET AREA (cm²) | TARGET LAYER (μ/cm²) | DEPOSITION YIELD (%) |
|-------------------|------|-----------------------|--------------|-------------------|----------------------|----------------------|
| ^{130}Te | 3 | 97 ± 3 | 12 | 93.3 | 313 ± 29 | 30.2 ± 1.4 ‡ |
| ^{205}Tl | 2 | 103 ± 6 | 12 | 119.3 | 472 ± 39 | 50.4 ± 0.9 |
| ^{207}Pb | 4 | 93 ± 5 | 14 | 120.4 | 412 ± 22 | 55.2 ± 2.2 |
| ^{208}Pb | 16 | 99 ± 6 | 12 | 119.3 | 408 ± 14 | 46.4 ± 2.1 |

† (Source-to-substrate distance) ‡ (Smaller frames)

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PEOPLE'S REPUBLIC OF CHINA

The Nuclear Target Society of China
Sun Shu-Hua Xu Guo-ji
Institute of Atomic Energy
Beijing, China

Since more than thirty years nuclear targets for nuclear physics experiments are prepared in China. In the early years (1955 - 1970) targets were made by physicists themselves. To meet increasing requirements in a variety of targets for nuclear physics research some target laboratories were created at the Institute of Atomic Energy Beijing, the Shanghai Institute of Nuclear Research, Shanghai, and the Institute of Modern Physics, Lanzhou, in the 1970's.

To exchange information and technology, the first national symposium on nuclear targets was held in Beijing in 1981. 54 papers were presented and 41 persons from 13 units attended the meeting. The information exchange concerned evaporation (joule heating, electron gun), electro-deposition, sputtering, measurement of target thickness, etc.....

The second national symposium on nuclear targets was held in Yangzhou city, province Jiangsu, 1984. the meeting was attended by 41 participants from 10 laboratories and 40 papers were presented. The scope of the meeting was concerned with preparation techniques of isotope targets, focussed heavy ion sputtering, rolling, strip-per foils etc. . . .

At that meeting the Nuclear Target Society of China was established and a board of directors was elected. The president is Dr. Sun Shu-hua, Institute of Atomic Energy, Beijing, the vice presidents are Dr. Wei Yang-qing, Shanghai Institute of nuclear research, Shanghai, and Mrs. Wang Xiu-ying, Institute of Modern Physics, Lanzhou. Now Society membership spans 14 laboratories and includes 50 persons.

The third national symposium of nuclear target will be held in 1987.

Thin Self-supporting Zirkonium targets
produced by heavy ion sputtering.

J.A. Sørensen and G. Sletten
Niels Bohr Institute, Denmark.

We have prepared self-supporting zirkonium targets in the thickness range from 100 to 500 micrograms per cm² by heavy ion sputtering. Before sputtering about 100 mg of zirkonium powder was pelletized to a 8 mm diameter disc and put into a 8 mm diameter recess in the beam-stop of the NBI sputtering apparatus¹. Zirkonium metal powder with natural isotope composition was used during the test preparations described in this article.

The zirkonium deposits were collected on object glass slides coated with thin layers NaCl or BaCl₂. The parting agents were put onto the slides by vacuum evaporations in a separate vacuum system. The beam stop to substrate distance was 2 - 3 cm and the entire holder assembly was rotated about the beam stop at 30 rpm.

We kept the argon beam intensively at about 200 µA and collected the films over a period of 7 hours. The process is extremely stable and needs no attention during deposition. For details of the sputtering apparatus we refer to ref. 1. and 2.

The zirkonium foils were floated off in water and mounted on target frames as single foils but also as double foils folded over the target frame. The success rate is close to 100% with NaCl.

The ready mounted target foils were assayed by proton induced X-ray fluorescence and single foils from 190 to 450 $\mu\text{g}/\text{cm}^2$ were mounted as well as double foils from about 350 to 500 μcm^2 . An estimate of the efficiency of the preparation can be done by computing the sputtering ratio from the amount of Zr consumed and the integrated ion current. This yields a ratio of 10. A more interesting figure for a target maker is the total film area which was available for mounting. We got an integral of 5 mg of zirkonium film from 20 mg consumed material.

The foils contain less than 0.2% chlorine and sodium or barium can not be detected. Impurities of iron and the chemical homologue hafnium were detected but could both be traced to the zirkonium stock material. An oxygen content of 10% by weight was found in all of the films. Since no precautions with respect to oxidation was taken we assume that the films oxidized to a certain depth after preparation. The targets have not yet been tested in beam, but they will be exposed to zirkonium beams in the near future. The outcome of these tests will perhaps be the subject of a forthcoming newsletter.

References:

- G. Sletten, Nucl. Instr. and Meth. 200, (1982), 21.
G. Sletten and P. Knudsen, *ibid.* 102, (1972) 459.

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MEENS, Andrée

Activity Report 1984

Some 130 requests for different types of targets for 22 physicists were satisfied during the year.

The majority of these targets (self-supported and otherwise) were made by evaporation and by rolling. The rolled targets were usually isotopically separated metals, with a thickness around 1 mg/cm^2 (^{64}Zn and ^{66}Zn have been rolled down to $450 \text{ }\mu\text{g/cm}^2$). Isotopes in the form of the oxide had first to be reduced to the metal. This was the case for Sm, Gd, Fe, Sn and Cd.

Magnetic targets of Fe(nat.), Gd(nat.) and $^{158}, ^{160}\text{Gd}$, with good adherence to an Ag backing were prepared with the following thicknesses : 0.5 to 2 mg of Fe and 1 to 4 mg of Gd on 1 to 8 mg Ag backings.

Targets of ^{80}Se were prepared by evaporation onto Au and covered by a $100 \text{ }\mu\text{g/cm}^2$ layer of Au. These targets were stable under the Se accelerator beam.

^{204}Hg has been transformed to HgS, evaporated onto a C backing and then covered with a C layer for stability in the beam. Two mg of HgS was used to prepare a $600 \text{ }\mu\text{g/cm}^2$ target on $40 \text{ }\mu\text{g/cm}^2$ C.

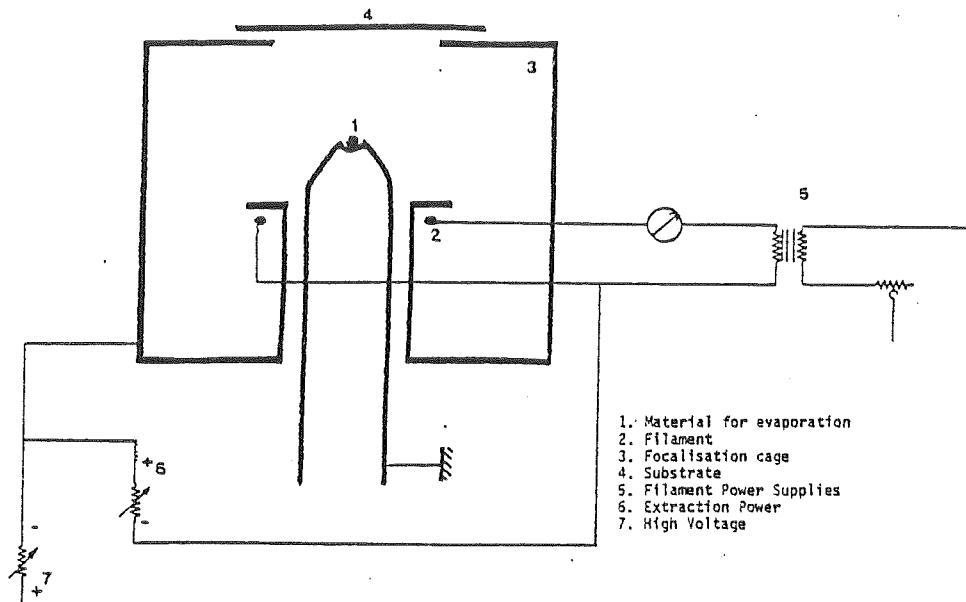
When possible, all of our target thicknesses were checked by α -particle energy loss measurements.

One mg/cm^2 self-supported Ca targets, protected with collodion were obtained by evaporation on glass slides polished with Teepol. Their thicknesses were determined by a prior calibration with titriplex III on other Ca targets made in the same conditions.

About 400 carbon stripper foils have been prepared by cracking, half of them slackened.

Fe_3C^{13} , FeS^{34} , CdSe^{80} , CdSe^{82} , Cd^{82} and Ni^{58} sputter targets for the MP source have been prepared.

Finally, inspired by the communication in Antwerp of J.M. and J.S. Heagney, we have built an electron gun. The design was similar to theirs, except that our crucible can be easily removed for cleaning. We also put an extraction voltage between the filament and focalisation cage (see the figure). This brings better efficiency and stability to the whole system.



At the present time only a few self-supporting ^{74}Ge targets of $250 \mu\text{g}/\text{cm}^2$ to $1 \text{ mg}/\text{cm}^2$ have been prepared, but we hope that in the near future we will be able to give some more results.

This text is a translation of my contribution to our 1984 Annual Report.

K.O. Zell

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Activity Report

Plastic foil helps making thin tablets

Tablets of 2-5 mg/cm² MoO, Te, and rare earth oxides of 2 cm diameter were pressed. A difficulty usually is that thin tablets tend to stick to the pressing device and break when removed. This can be avoided if one puts a thin plastic foil at the bottom and on top of the powder and presses it together with the foil. The foil does not stick to the pressing device and can also easily be removed from the tablet.

Molybdenum plunger targets

¹⁰⁰Mo has been rolled for plunger targets to a thickness of 0.5 mg/cm² starting from powder which was molten by an electron gun. To avoid pinholes it seemed to be important to roll at a very low speed and to increase the pressure only slowly.

Bismuth as hot melting glue

We had the problem to roll 0.5 mg/cm² molybdenum onto 10 mg/cm² gold which cannot be done without any glue. As only a very heavy material was allowed we evaporated 1 mg/cm² bismuth onto the gold and rolled this between steel plates to the molybdenum. Molybdenum does not stick to the bismuth and gold foil when rolled cold, therefore the pack was heated before on a gas flame to a temperature at which the water vapour from the flame does not condense any more on the steel plates and rolled hot.

Thin iron, chromium, and nickel targets

Selfsupporting iron, chromium, and nickel targets of 0.2-0.3 mg/cm² were made by evaporating the material in 5 cm distance from a tungsten tube onto a 10 µm copper foil which was heated by a quartz lamp to 300 °C (Fe,Ni) or 350 °C (Cr). The copper was etched with trichloroacetic acid and NH₃. While we obtained targets without any stress from iron and chromium, nickel tended to form small rolls after the etching.

BaO targets

50 $\mu\text{g}/\text{cm}^2$ BaO targets were made by heating BaO in a carbon tube and evaporating the decomposed barium on a 10 $\mu\text{g}/\text{cm}^2$ carbon foil which was rotated in such a way that the barium went to both sides of it thus avoiding any stress (especially that from oxidation when the vacuum is broken). As the BaO likes to jump out of the tube we melted it by heating the carbon tube on a gas flame before putting it into the vacuum. This trick works only with natural material as isotopically enriched BaO does not melt.

Negligible Coulomb excitation from bismuth backings in heavy ion beams

In our report on bismuth backings on last years target conference we gave only spectra obtained with the (α, n) -reaction showing that there is no disturbing γ -line from the Coulomb excitation of bismuth. There was some concern that this could change with more heavy ions. We now have used bismuth backings also with such heavy ions as ^{22}Ne , ^{30}Si , and ^{36}S and no bismuth line could be seen in the spectra of bismuth backed tin and calcium targets.

Other targets

(e) made by evaporation, (r) made by rolling

Pd 1 mg/cm^2 (r) + Bi 60 mg/cm^2 (r) + In 1 mg/cm^2 (e) + Cu 250 mg/cm^2 (r),
 ^{118}Sn 1.8 mg/cm^2 (r) + Bi 52 mg/cm^2 (r) + In 1 mg/cm^2 (e) + Cu 100 mg/cm^2 ,
 ^{64}Zn 2 mg/cm^2 (r) + Bi 60 mg/cm^2 (r) + In 1 mg/cm^2 (e) + Cu 100 mg/cm^2 ,
 ^{117}Sn 2.7 mg/cm^2 (r),
 ^{117}Sn 1.3 mg/cm^2 (r) + Pb 80 mg/cm^2 + In 1 mg/cm^2 + Cu 100 mg/cm^2 (r),
 ^{70}Ge 1.5 mg/cm^2 (e) + Bi 100 mg/cm^2 (r) + epoxy glue + Cu 100 mg/cm^2 ,
Bi 0.1 mg/cm^2 (e) + ^{40}Ca 0.74 mg/cm^2 (e) + Bi 60 mg/cm^2 + In 1 mg/cm^2 +
Cu 100 mg/cm^2 ,
 ^{40}Ca 2 mg/cm^2 (e) + Au 0.3 mg/cm^2 (e)
 ^{120}Sn 80 $\mu\text{g}/\text{cm}^2$ (e)
 ^{208}Pb 0.2 mg/cm^2 (e)

CBNM PROGRAMME
REFERENCE MATERIALS FOR REACTOR NEUTRON DOSIMETRY

STATUS REPORT 31/7/1985

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Since the last status report (1/6/1984) a joint communication was made by A. Fudge to the 5th ASTM-EURATOM SYMPOSIUM ON REACTOR DOSIMETRY, GKSS Geesthacht, 24-28 September 1984 and by J. VAN AUDENHOVE to the 12th WORLD CONFERENCE of the INTERNATIONAL NUCLEAR TARGET DEVELOPMENT SOCIETY, Antwerpen, 25-28 September 1984, a second meeting of "Consultants and Collaborators" was held in Geel on 12 March 1985 to discuss the 1984 and 1985 certification campaign and a meeting of the Monitor Materials Subgroup of the Euratom Working Group on Reactor Dosimetry (EWGRD) was held in Petten on 22 April 1985 to review the different projects.

A provisional timetable (See Figure 1) was drawn up and accepted on the understanding that material would be released prior to the completion of the official EC-certificate. The material should be made available for sale as soon as it had been converted into its final form and its quality assured achieved with sufficient analytical evidence.

Progress made for the different RM-projects is summarized below.

1. ALUMINIUM

Aluminium in metallic form is used in neutron dosimetry :

(a) as a fast neutron monitor, > 6 MeV [$^{27}\text{Al} (n, \alpha)^{24}\text{Na}$]. Traces of ^{23}Na interfere when they produce ^{24}Na by capture with thermal neutrons present in the same spectrum.

(b) as a dilution element for alloying with other dosimeter materials.

For application (a) a low and well known sodium content is required, whereas for (1) the purity requirements are less stringent.

Figure 1 : Tentative timing

| | 1986 | 1987 | 1988 | 1989 | 1990 | 1991 |
|--|-----------------------------|-------------|-------------------------|--------------------|------------------|------------------|
| <u>MATERIALS SUBJECT TO CERTIFICATION ANALYSES IN 1984</u> | | | | | | |
| Al | ANAL - DRAFT | | <u>CERTIFIC.</u> | | | |
| Ni | DRAFT | | <u>CERTIFIC.</u> | | | |
| U 238 | ANAL - DRAFT | | <u>CERTIFIC.</u> | | | |
| <u>MATERIALS SUBJECT TO CERTIFICATION ANALYSES IN 1985</u> | | | | | | |
| Cu | ANAL | DISC.-DRAFT | <u>CERTIFIC.</u> | | | |
| Fe | ANAL | DISC.-DRAFT | <u>CERTIFIC.</u> | | | |
| <u>MATERIALS IN PREPARATION</u> | | | | | | |
| Nb | SAMPL. ANALYSES | | DISC.-DRAFT | <u>CERTIFIC.</u> | | |
| AlCo | PREP. & TRANSF. | | ANALYSES | DISC.-DRAFT | <u>CERTIFIC.</u> | |
| Np 237 | TRANSFORMATION | | ANALYSES | DISC.-DRAFT | <u>CERTIFIC.</u> | |
| V | SAMPL. ANALYSES | | DISC. | <u>PREP.ENCAPS</u> | | |
| <u>OTHER MATERIALS</u> | | | | | | |
| Th 232 | | | PREP.&TRANSF ANALYSES - | DISC.-DRAFT | <u>CERTIFIC.</u> | |
| In | | | PREP.&TRANSF ANALYSES - | DISC.-DRAFT | <u>CERTIFIC.</u> | |
| V-Co | | | PREP.&TRANSF-ANALYSES | | DISC.-DRAFT | <u>CERTIFIC.</u> |
| V-U 235 | | | PREP.&TRANSF-ANALYSES | | DISC.-DRAFT | <u>CERTIFIC.</u> |
| V-Pu 239 | | | PREP.&TRANSF-ANALYSES | | DISC.-DRAFT | <u>CERTIFIC.</u> |
| Rh | ACQ. BASE MAT. TRANSFORM. - | | ANALYSES - | DISC.-DRAFT | <u>CERTIFIC.</u> | |
| Ti | | | | | | |

Four types of aluminium samples have been subjected to certification analyses:

- RM foil of 0.1 mm thick ;
 - RM foil of 1 mm thick ;
 - RM wire of 1 mm diameter ;
 - 1 mm thick foil of aluminium purified by high frequency levitation melting. These analyses were carried out by AERE Harwell, CBNM Geel, CNRS Orléans, INW Gent and SCK/CEN Mol. From their provisional results it could be concluded that :
- the bulk of the material is very pure (at least 99.99 %, but probably 99.999 %) and contains very little sodium ($< 0.1 \mu\text{g/g}$) ;
 - as most of the sodium impurity is found as contamination in the oxide surface layer, the material is best used by etching between irradiation and counting ;
 - if this is not possible, the material should be finally cleaned before use. This can be done by etching in purified concentrated hydrochloric acid containing less than $3 \mu\text{g/l}$ of sodium and rinsing with freshly distilled water. In this way, the sodium content can be kept below an equivalent of $0.5 \mu\text{g/g}$.

Further work on reducing the impurity level of sodium and to find optimum methods of utilisation of this material are in progress.

This RM will be offered for sale, either in bulk form (unit size : 100 cm^2 or 1 m) or in the form of individually packed dosimeters.

2. NICKEL

The nuclear requirement of nickel for use in neutron dosimetry is that the ^{60}Co produced by the $^{59}\text{Co}(n,\gamma)^{60}\text{Co}$ reaction should not exceed 10 % of the count rate of ^{58}Co produced by the $^{58}\text{Ni}(n,p)^{58}\text{Co}$ reaction. For the monitoring of spectra with a high thermal/fast neutron energy ratio this requires less than 1 part per million of cobalt in nickel.

Two types of samples have been subjected to certification analyses :

- RM foil of 0.1 mm thick ;
- RM wire of 0.5 mm diameter.

These analyses were carried out by AERE Harwell, BAM Berlin, CBNM Geel and INW Gent. From these analyses it can be concluded that both foil and wire are of an overall purity better than 99.95 %. Their cobalt content is lower than 0.10 µg/g as shown in Table 1.

Table 1 : Certification results for Co in Ni

| <u>METHOD</u> | <u>RESULTS (µg/g)</u> |
|---------------|-----------------------|
| AAS | < 0.05 (n=6) |
| | < 0.10 (n=8) |
| ICP | < 0.10 (n=8) |
| NAA | < 0.10 (n=8) |
| | < 0.02 (n=8) |

On the basis of these results a draft report and certificate will be prepared. In the meantime, a number of complementary pieces of information on impurities will be collected and an isotopic analysis will be carried out to verify the natural composition of the material.

The material will as soon as possible be made available for sale either in bulk form (unit size : 100 cm² or 1 m) or in the form of individually packed dosimeters.

3. URANIUM 238

²³⁸U has been used extensively in neutron dosimetry as a fissionable material with a threshold energy response ($E_T = 1.5$ MeV). In order to reduce the interference from thermal fission due to residual ²³⁵U and neutron capture and subsequent fission of ²³⁹Pu, it is surrounded by a thermal neutron absorbing material such as Cd, or Gd while it is being irradiated. This interference is also reduced by using material with as low as a ²³⁵U content as possible, ideally at less than 15 µg/g ²³⁵U (2).

Uranium-238 oxide of high isotopic (99.999 %) and chemical (99.98 %) purity purchased from the Oak Ridge National Laboratory, USA has been transformed into 0.5 and 1.0 mm spheres by sol-gel precipitation at AERE Harwell, UK (3). The spheres have been analysed for possible contamination by alpha spectrometry and thermal ionisation mass spectrometry (See Table 2). Homogeneity has been checked by gamma counting 25 of the 1 mm spheres. The observed reproducibility was not significantly different from the one expected from the counting statistics (1 s = 1.3 %).

Table 2 : Impurities determined in $^{238}\text{UO}_2$ gel spheres (in g/g)

| <u>NUCLIDE</u> | <u>ISOTOPIIC ANALYSIS</u> (M.S.) | <u>ALPHA SPECTROMETRY</u> | |
|----------------|-------------------------------------|---------------------------|-------------------|
| | | <u>MEASURED</u> | <u>UPPER UNIT</u> |
| U-232 | - | 5 10^{-13} | < 10^{-11} |
| U-233 | - | 0.09 10^{-6} | < 0.13 10^{-6} |
| U-234 | < 0.2 10^{-6} | 0.14 10^{-6} | < 0.20 10^{-6} |
| U-235 | 10.5 10^{-6} | 5 10^{-6} | < 60 10^{-6} |
| Pu-239 | - | 0.4 10^{-9} | < 3 10^{-9} |
| Pu-240 | - | 0.1 10^{-9} | < 0.8 10^{-9} |
| Pu-238 | - | 0.02 10^{-9} | < 0.04 10^{-9} |
| Am-241 | - | 0.07 10^{-9} | < 0.2 10^{-9} |

Measurements of the stoichiometric composition of the uranium dioxide spheres were carried out on both 0.5 and 1.0 mm spheres by isotope dilution mass spectrometry (IDMS), potentiometry and gravimetry (Table 3). A two-way analysis of variance carried out on all 36 results reveals that no significant difference can be observed between 0.5 and 1.0 mm spheres. The difference between the methods is statistically significant, but anyhow small enough to be acceptable.

All measurements were carried out at CBNM by the analytical science, radionuclide and mass spectrometry groups.

Table 3 : Certification results for the U/UO₂ mass fraction

| <u>METHOD</u> | <u>RESULTS IN g/g</u> |
|--------------------------|-----------------------------|
| | <u>MEAN ± 95 % C.L. (n)</u> |
| IDMS | 0.878 74 ± 0.000 85 (12) |
| Potentiometry | 0.878 38 ± 0.001 62 (12) |
| Gravimetry | 0.880 88 ± 0.000 16 (12) |
| Proposed Certified Value | 0.879 3 ± 0.003 4 (36) |

Other homogeneity checks using larger numbers of spheres and X-ray fluorescence failed to provide any improved data. On the 0.5 mm spheres, a standard deviation of 3 % was obtained.

On the basis of the above results a draft report and certificate will be prepared. The R.M. will be made free for sale as soon as possible (units of 50 mg or 100 mg). Sale of encapsulated spheres will be a matter of direct negotiation between customers and CBNM.

4. COPPER

The nuclear reaction of copper used for neutron dosimetry is, $^{63}\text{Cu}(n,\alpha)^{60}\text{Co}$. Thus the impurity element of greatest importance is cobalt since interference is produced by the thermal neutron reaction $^{59}\text{Co}(n,\gamma)^{60}\text{Co}$. However, traces of other impurities commonly found in copper such as silver, can increase the uncertainty of the measurements if present in significant quantities.

Copper 99.999 % was purchased, but - after NAA control carried out at SCK/CEN Mol - was found to be heterogeneous (Table 4). After homogeneisation at CBNM by induction melting and casting in argon atmosphere, the homogeneity was rechecked under identical conditions and found to be appropriate. The material was then transformed into 0.1 and 1 mm thick foil and 0.5 and 1 mm diameter wire.

Table 4 : NAA results for Co, Sb and Ag in Cu, before and after homogeneisation

| ELEMENT | RESULTS IN $\mu\text{g/g}$ (N) | |
|---------|--------------------------------|---------------------------------|
| | <u>BEFORE</u> | <u>AFTER</u> homogeneisation |
| Co | < 0.01 to 0.3 (5) | 0.012 to 0.019 (10) |
| Sb | 0.3 to 3 (5) | 1.0 to 1.2 (10) |
| Ag | 0.2 to 2 (5) | 0.76 to 0.87 (10) |

Certification analyses on all 4 forms are planned for 1985. They will include a verification of the natural isotopic composition, a verification of the overall purity by SSMS and NAA, and the determination of cobalt (probably < 0.1 $\mu\text{g/g}$) and silver using different methods (Table 5). The analyses will be carried out by AERE Harwell, BAM Berlin, CBNM Geel, CNRS Orléans, INW Gent and SCK/CEN Mol.

Table 5 : Methods to be used for the certification of Co and Ag in Cu and Co and Mn in Fe

| METHOD | COPPER | | IRON | |
|---------|-----------|-----------|-----------|-----------|
| | <u>Co</u> | <u>Ag</u> | <u>Co</u> | <u>Mn</u> |
| NAA | + | + | + | + |
| AAS | + | + | + | + |
| ICP-MS | + | + | + | - |
| ICP-AES | - | - | - | + |

5. IRON

Iron is used in neutron dosimetry via the reactions $^{54}\text{Fe}(n,p)^{54}\text{Mn}$ and $^{58}\text{Fe}(n,\gamma)^{59}\text{Fe}$. The impurity elements that result in interference in the counting techniques are manganese and cobalt via the reactions $^{55}\text{Mn}(n,\gamma)^{56}\text{Mn}$ and $^{59}\text{Co}(n,\gamma)^{60}\text{Co}$.

Iron 99.99 % was transformed at CBNM into foil 0.1 mm thick and wire 0.5 mm diameter. The reception analyses carried out at SCK/CEN Mol using NAA, showed that the overall purity of the material is very good, and in particular its Co and Mn content is $< 0.1 \mu\text{g/g}$.

On both foil and wire certification analyses are planned for 1985. They will include a verification of the natural isotopic composition, a verification of the overall purity by SSMS and NAA, and the determination of cobalt and manganese using different methods (Table 5). The analyses will be carried out by AERE Harwell, BAM Berlin, CBNM Geel, CNRS Orléans, INW Gent, Risø National Laboratory Roskilde and SCK/CEN Mol.

The present best value of the ^{54}Fe abundance of natural iron is only known at 2 % relative (5.8 ± 0.1). It is not yet clear whether a verification of the natural isotopic composition would be sufficient or if a major effort would be justified to improve the present knowledge via the preparation of synthetic isotopic mixtures. This will depend on whether or not the cross-section of the reaction $^{54}\text{Fe}(n,p)^{54}\text{Mn}$ was determined using natural iron (in which case there would be a correlation between σ and θ) or using enriched ^{54}Fe (in which case there would be no correlation). In the first case it is important to know which abundance value was used.

6. OTHER MATERIALS

One kg (16 monocrystals) niobium 99.999 % containing less than $0.4 \mu\text{g/g}$ Ta has been prepared by Max-Planck-Institut für Metallforschung, Stuttgart (4). This has to be transformed into foil (th. = $20 \mu\text{m}$ and 0.1 mm) and wire (diameter = 0.5 mm).

Furthermore, a second quality material ($\leq 60 \mu\text{g/g}$ Ta) is in preparation at Gesellschaft für Elektrometallurgie, Nürnberg. This material will be transformed into foil and wire before the other one, in order to contaminate the tools with niobium before start.

Several preparation methods to obtain sufficiently homogeneous aluminium-cobalt alloys containing 0.1 and 1 % Co respectively have been investigated at CBNM. These include

(a) induction melting in a crucible of 1.5 kg amounts of alloy, followed by casting of 20 mm thick plates in water cooled graphite moulds.

(b) high frequency levitation melting of 20 g amounts of alloy followed by casting of 3 mm thin plates in a water cooled copper mould, resulting in a ca. 1 second solidification. In both cases the required homogeneity (≤ 1 % at 20 mg sample size) could not be achieved.

As the resulting less homogeneous material (± 2 % at the 20 mg level) was not accepted, efforts will now be concentrated on the production of vanadium-cobalt alloys as soon as V with less than 10 $\mu\text{g/g}$ of Ta becomes available. For V-1 % Co and V - 0.1 % Co, the required homogeneity is however < 1 % at the 5 mg scale. They are however single phase substitutional alloys whereas the Al-Co alloys are double phase ones with a fine dendritic network of Al_9Co_2 (containing 32 % Co) surrounding pure Al grains of about 10 μm . Consequently, these are more likely to be produced with the required homogeneity.

Vanadium with < 10 $\mu\text{g/g}$ Ta will be prepared by Gesellschaft für Elektrometallurgie, Nürnberg (5). As well as its use as matrix for cobalt alloys, this material will also be used to prepare uranium 235 (0.2 and 5 % U) and plutonium 239 (3 % Pu) alloys and as canning material for dosimeters.

Neptunium-237 oxide of 99.99 % isotopic purity has been purchased from ORNL and sent to AERE Harwell for transformation into 0.5 and 0.8 mm gel spheres. This transformation is expected to be terminated before october 1986.

Thorium-232 oxide and indium oxide (mixed with Al_2O_3 or SiO_2) have been suggested to be transformed into gel spheres, but it has not yet been decided whether or not to transform them.

Rhodium foil containing less than 15 $\mu\text{g/g}$ Pt and Ir probably does not exist, but a 300 g batch of Rh powder containing ca. 25 $\mu\text{g/g}$ Pt and 20 $\mu\text{g/g}$ Ir has been located. It is foreseen to investigate the transformation of this material in 50 μm foil at CBNM.

Titanium containing less than 0.1 $\mu\text{g/g}$ Sc has not yet been located.

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Situation wanted

Nuclear Industrial Engineer, option electronics, 23 years old, with one year experience in nuclear target preparation at the Central Bureau for Nuclear Measurements, Geel, Belgium, is seeking for an engineer position with challenge.

Salary and location open.

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