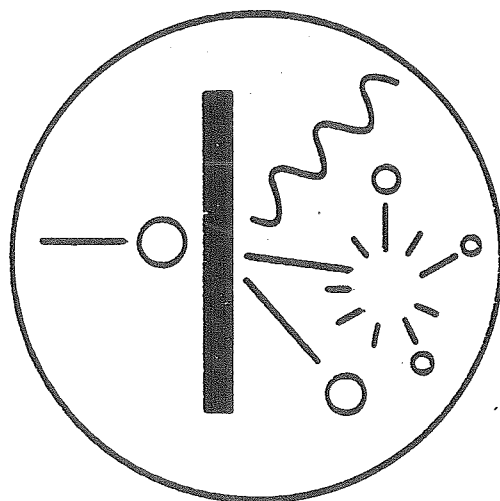


**INTERNATIONAL NUCLEAR
TARGET DEVELOPMENT SOCIETY**

NEWSLETTER



December 1988

Vol. 15 - N° 2 - December 1988

Published two times a year by :

International Nuclear Target Development Society

c/o Mrs. Joanne M. Heagney
P.O. Box 123
123 Madrona Lane
Deer Harbor, WA 98243
USA
Tel. : (206) 376 - 4007

Editor : Jan Van Audenhove
Joint Research Centre - CBNM
Commission of the European Communities
Steenweg op Retie
B - 2400 Geel - Belgium

Tel. : 32-14-571211 or 571314
Telex : 33586 EURAT B
Telefax : 32-14-584273

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.

CONTENTS

	<u>Page</u>
● Word from our President	1
● Minutes of the INTDS Business Meeting, Sept. 7, 1988	2
● Treasurer's report July 1, 1987 - June 30, 1988	5
● Guidelines for INTDS Awards	6
● Preparation of $^{10}\text{B}_2\text{O}_3$ vacuum evaporated targets William R. Lozowski and Jeffrey D. Hudson Indiana University Cyclotron facility, 2401 Milo B. Sampson Lane Bloomington, IN 47405, USA	8
● Activity Report A. Cecchi Istituto Nazionale di Fisica Nucleare Largo E. Fermi, 2, 50125 Firenze, Italy	10
● Development of Carbon Stripper Foils by Arc Evaporation N. Ueta Departamento de Fisica Nuclear, Instituto de Fisica da Universidade de São Paulo, C.P. 20156, 01498 São Paulo SP, Brasil	12
● Activity Report from ISN Grenoble R. Blanc, M. Bouriant, J.P. Richaud Institut Sciences Nucléaires, 53, Avenue des Martyrs, 38026 Grenoble, France	16
● Samples and Targets for Nuclear measurements delivered by CBNM during 1988 J. Pauwels, C. Ingelbrecht, R. Eykens CEC, JRC, CBNM, Geel Establishment, 2440 Geel, Belgium	20
● Information on literature	25
● INTDS Membership list of October, 1988	26


FOURTEENTH INTDS CONFERENCE A TREMENDOUS SUCCESS

The Fourteenth INTDS Conference held at GSI, Darmstadt, Federal Republic of Germany, from September 5-9, 1988, was one of the most successful conferences held since the formation of INTDS. It was a truly international conference in which some 104 attendees from 19 countries participated.

The Fourteenth INTDS Conference was organized in cooperation with the International Atomic Energy Agency (IAEA) - International Nuclear Data Committee (INDC). Sixty-four papers were presented in 16 sessions that included contributions discussing the following topics:

1. target problematics and applications;
2. heavy ion target users' comments;
3. fissioning targets;
4. cross-section measurements;
5. tritium processing and tritium target problematics;
6. stripper foils;
7. electrodepositions;
- 8,9. various target preparations (two sessions);
10. activity reports of target laboratories;
11. oxide reductions;
12. characterizing targets and target damage;
13. rolling and milling of targets;
14. isotope separations;
15. medical isotope production; and
16. electron, gas, and plasma targets.

On behalf of INTDS, I convey our thanks to our host, GSI (Drs. Paul Kienie and Helmut Folger) and Dr. Koichi Okamoto from IAEA-INDC for their efforts in making the meeting a success.


H. L. Adair
President, INTDS

Minutes of the INTDS Business Meeting
September 7, 1988
GSI, Darmstadt, FDR

The meeting was called to order at 1730 hours by the President, Harold Adair.

Joanne Heagney regretfully informed the group of the death of Darrell Erikson in an automobile accident in February, 1988. A moment of silence was observed to express the deep sense of loss felt by all.

Agenda, Actions and Resolutions

1. **Comments on the 14th INTDS Conference at GSI**
Harold Adair acknowledged the fine work of Helmut Folger and GSI which produced the highly rewarding conference in progress. In recognition and appreciation, Harold presented an INTDS lapel pin to Helmut on behalf of the members.
2. **Minutes of the 1986 Business Meeting**
There were no comments from the floor concerning the minutes of the meeting in Deep River.
3. **Secretary/Treasurers' Report**
Corresponding Secretary/Treasurer, Joanne Heagney presented a financial report and stated that it will be published in the December issue of the Newsletter. She also reported the number of members to be seventy-seven as of September 7, 1988: thirty-four from Europe, thirty-three from North America, and ten from other locations.
4. **Revision of the By-laws**
Harold Adair announced that all members responding to the mail vote on the proposed changes to the By-laws were in favor of them. The By-laws are thereby revised as proposed. Thus, the Society now has biennial dues and an election technique for board members in which the entire membership will participate; not only those who can attend the world conferences. Harold reviewed transition measures to be implemented concerning the application of the revised By-laws. These measures had been submitted for approval along with the proposed by-law revisions.
Accordingly, Harold explained: four members would be elected at the current meeting to serve four year terms of office on the Board. One additional elected member would serve the two remaining years of Darrell Erikson's term.
5. **Nominating committee for the 1990 elections**
The Nominating Committee chosen by the Board was announced to be: Jean Pauwels, Peter Maier-Komor, and Joanne Heagney.

6. **Guidelines for Hosts of INTDS Conferences**
The Guidelines recently approved by the Board were discussed with the members. Harold Adair read the "Summary" section which states "The intent of these guidelines is not to restrict hosts for biennial INTDS conferences to follow a rigid format without flexibility; however, to the extent possible, the overall objectives of INTDS should be the major consideration in all phases of conference organization..." Members may request a copy of the Guidelines from Joanne Heagney.
7. **Awards**
The members were asked to submit to the Board the name of any member who should be considered for an achievement award which could be presented at the 1990 conference in Santa Fe.
8. **Committee on the Future Directions of the INTDS**
Co-chairmen Harold Adair and Hans Maier will reconvene with the other committee members (Jan Van Audenhove, Helmut Folger, Gierr Sletten, and Bill Lozowski) to reexamine the subject at the 1990 Santa Fe Conference. In the interim, Harold and Hans agreed to work together in an effort to present some possibilities for consideration at that board meeting. Suggestions concerning future directions were also solicited from the members.
9. **Committee on the Use of INTDS Funds**
The recording secretary read the following statement "A committee to study appropriate uses for INTDS funds was formed. The initial constitution of this committee is: Hans Maier (Chair), Helmut Folger, Joanne Heagney, and Bill Lozowski. INTDS members are encouraged to approach the members of this committee with specific or general proposals which would benefit the Society. Examples of proposals might be: people to be considered for travel scholarships, Society advertisements or publications, etc. The committee will report to the full Board."
Geirr Sletten made the observation that some funds may be required for publication of the Newsletter when the CBNM and Jan Van Audenhove can no longer provide this service.
10. **1990 INTDS Conference in Santa Fe**
Peter Gabbie gave an entertaining preview of the beautiful Santa Fe area providing narration to accompany a bevy of photographic transparencies. He also said that Carlsbad Caverns was close enough for an all day trip (not organized by Los Alamos) and that the date of the conference had yet to be decided.
11. **Special Training of INTDS members**
The members were told of the IAEA's (K. Okamoto's) desire that the INTDS find laboratories where training in target making could be provided for individuals. Hans Maier stated that his lab has trained people in target making

techniques on an exchange basis, with each lab providing the financial support for their person. K. Okamoto added that there might be funds available for this purpose. Harold Adair said that the Board could act as a clearing house for requests to the INTDS for individualized technical training. It was agreed that specific proposals could be posted in the Newsletter.

12. Board Member Election

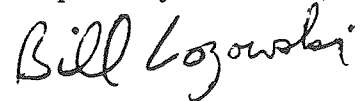
The election was held, the ballots were counted, and the results were announced. Those elected were: Helmut Folger, Peter Maier-Komor, Jean Pauwels, Geirr Sletten, and George Thomas. Geirr Sletten will serve for two years.

13. Appraisal of the Joint Conference

The Board and Membership were in agreement that the joint INTDS/IAEA-INDC meeting was beneficial to the INTDS and that future joint meetings between the two groups would be encouraged.

The meeting adjourned at 1815 hours.

Respectfully submitted,



Bill Lozowski
Recording Secretary

GUIDELINES FOR INTDS AWARDS

1. Award presentation(s) should be made during biennial conferences. Although the most logical time of the award would be at the banquet or business meeting, the specific time will be left to the discretion of the President.
2. Under normal circumstances, the maximum number of awards per biennial conference should be limited to one; however, under unusual circumstances, it may be necessary to increase the number of awards to two. Under no circumstances should there be more than two awards per award period.
3. The selection of individuals for awards will be made by the INTDS Board of Directors during nonconference years. Candidates for awards can be nominated by any INTDS member in good standing. The nomination will be in written form and should contain detailed information justifying why the nominee should receive the award.

The names and justifications for awards for all nominees will be presented to the Board of Directors by the President. Votes will be taken until one of the nominees receives at least two-thirds of the votes of the eligible Board Members. If a nominee is a Board member, then he/she is not eligible to vote for themselves; however, they can vote for other nominees. In those years where it is decided that two awards should be given, this decision should be supported by two-thirds vote of the Board of Directors.

4. Nominees for awards should be restricted to those INTDS members who have played an important role in the life of the Society and/or have realized several important developments during their career. Candidates for awards should have a minimum of ten years membership in INTDS.

**Awards of Recognition to INTDS Members
(as of August 1988)**

Dan Riel:	1982, Seattle
Ed Kobisk:	1983, Willowbrook
George Thomas:	1984, Antwerp
Jan Van Audenhove:	1986, Deep River
Joe Gallant:	1986, Deep River
Katherine Glover:	1986, Deep River
Judy Gursky:	1986, Deep River
Arthur Peck:	1986, Deep River
Don Ramsey: (post humously)	1986, Deep River
Jack Stinson:	1986, Deep River

Preparation of $^{10}\text{B}_2\text{O}_3$ Vacuum Evaporated Targets¹
William R. Lozowski and Jeffrey D. Hudson
Indiana University Cyclotron Facility

We have recently prepared (p,p') oxygen targets of 3.5 mg/cm^2 $^{10}\text{B}_2\text{O}_3$ evaporated onto $260 \text{ } \mu\text{g/cm}^2$ mounted Au foils.

Previous experience at IUCF with oxygen targets of 5 mg/cm^2 pressed disks of $\text{H}_3^{10}\text{BO}_3$ had revealed them to be thermally unstable in a proton beam. The rate of this decomposition was acceptably decreased when the disks were jacketed in $200 \mu\text{g/cm}^2$ Au leaf; however, the hydrogen present in the boric acid masked the structure of interest at some scattering angles and necessitated its removal. For this reason, the acid was thermally decomposed to $^{10}\text{B}_2\text{O}_3$, and evaporated with an electron beam gun using the following technique.

Boric acid enriched to 98.36 atom% ^{10}B was obtained as a stock item from Eagle Picher[1] and carefully decomposed as described in reference [2]. The method produces "practically anhydrous" B_2O_3 if the boric acid is ground to a fine powder, slowly heated to $260^\circ\text{-}270^\circ \text{ C}$ at $133\text{-}266 \text{ Pa}$ (1-2 Torr), and maintained at this temperature for six hours. If the pressure is above 5.3kPa (40 Torr) or the heating is too rapid, sintering takes place and complete dehydration is prevented [2].

With these guidelines, we constructed a tube furnace using a glass tube (1 mm wall x 1 cm dia. x 15 cm long) wound with resistance wire. For the decomposition, the furnace was used inside a high vacuum evaporator. A Ni combustion boat (0.45 mm thick x 5 cm long x 5 mm tall) was constructed to contain $\sim 800 \text{ mg}$ of $\text{H}_3^{10}\text{BO}_3$. The temperature was monitored using a temperature probe placed inside the furnace near the combustion boat. After evacuating the evaporator to $1.3 \times 10^{-4} \text{ Pa}$ (1×10^{-6} Torr) and reaching 265°C in ~ 2 hours, the decomposition was continued overnight. The measured weight loss revealed the $^{10}\text{B}_2\text{O}_3$ to be anhydrous. Since the compound is hygroscopic, air exposure was kept to a minimum.

Surprisingly, it was not necessary to press the powder into a pellet for evaporation from a 2 kW Varian e-gun (180° bent beam). The evaporation was accomplished by loading $\sim 300 \text{ mg}$ of $^{10}\text{B}_2\text{O}_3$ into the water-cooled hearth of the e-gun, fusing the powder by moving the beam around at low power, and then increasing the power until evaporation took place at $30\text{-}40 \text{ } \text{Å}/\text{sec}$.

The 3.5 mg/cm^2 thickness was accomplished in three evaporations of

approximately 1.2 mg/cm². Before the second and third evaporations, the Au backing was flipped front-to-back to help balance the evaporation-induced mechanical stress in the ¹⁰B₂O₃. Gold leaf backings were found to withstand this stress much better than comparably-thick evaporated films of Au.

Floating the 260 μg/cm² leaf in water and lifting it vertically onto a clean frame produced a reasonably taut, self adherent backing. The frames had an open-sided 2 cm dia. hole (i.e. "C-shape") necessitated by the energy-dispersed beam in the IUCF K600 spectrometer. A 0.5 mm thick Al mask with a 1.59 cm dia. chamfered hole defined the area exposed to the evaporation. It was spaced ~ 0.5 mm in front of the frame. The frame and mask were linked to a rotating feedthrough which allowed the assembly to be positioned between the source (e-gun) and a crystal thickness monitor during the evaporation. A source-to-substrate distance of 3.6 cm was found to provide an acceptable evaporation efficiency, target thickness uniformity and heat-load to the backing. The amount of hydrogen in the target was not quantified but was reported to be very small.

Summary

While it is easy and fun to produce thin films of B₂O₃ by decomposing boric acid in air within a nickel crucible and blowing it as one does with other glasses, the quantity of boric acid required for this technique precludes its use with isotopically enriched material. If a backing of some kind can be tolerated, straight-forward evaporations of enriched B₂O₃ are possible with an electron beam gun and a water-cooled hearth.

Address and Reference

- [1] Eagle-Picher Industries, Inc., Specialty Materials Division, Miami, OK 74355.
- [2] Kirk-Othmer, Encyclopedia of Chemical Technology, second ed., vol. 3, (John Wiley and Sons, NY, 1963) p. 610.

Activity report

Istituto Nazionale di Fisica Nucleare

Largo E. Fermi, 2

50125 Firenze - Italy

A. Cecchi

During 1988, the primary activity of our laboratory was the preparation of self-supporting ruthenium targets.

Numerous experiments by the nuclear spectroscopy group, operating in our Department, required the use of ruthenium targets which varied in shape and ranged in thickness from $.5 - 2 \text{ mg}\cdot\text{cm}^{-2}$.

Targets of self-supporting isotopes of ruthenium, ^{96}Ru , ^{100}Ru and ^{102}Ru , were obtained by electroplating^(*) ruthenium salt. The salt had been obtained by means of alkaline fusion of the isotope and subsequent solubilization in hydrochloric acid. A lucite cell with spiral shaped platinum anode and a flat platinum cathode, covered by a copper film, was used. Once the ruthenium had been deposited on the copper backing, a solution containing trichloroacetic acid and ammonium hydroxide was used to dissolve the copper. During this process the low current density and temperature allowed for the obtaining of strong, uniform and stable self-supporting targets, which were also highly resistant to heavy ion bombardment.

Nevertheless, analysis performed by means PIXE method revealed the presence of copper in the ruthenium targets; the weight ratio was $\approx .2$. The high copper contamination was ascribed to the slight solubility of the copper backing in the acid solution during the electroplating.

In order to eliminate this problem, the electrodeposition process was repeated using all the same parameters except one: a silver backing was used as the cathode instead of a copper one.

At the end of this procedure, the cell was washed repeatedly in bidistilled water and the silver with the ruthenium deposit was floated onto the surface of a 30% nitric acid solution. The targets obtained appeared to have satisfactory strength. The silver to ruthenium weight ratio was estimated to be \approx .07.

It appears that there is a suitable alternative in the preparation of self-supporting ruthenium targets using electroplating.

(*) A. Cecchi and P. Sona: submitted to N.I.M., section A.

DEVELOPMENT OF CARBON STRIPPER FOILS BY ARC EVAPORATION

N. UETA

Departamento de Física Nuclear, Instituto de Física da Universidade de São Paulo, C.P. 20516, 01498 São Paulo SP, BRASIL

Most stripper foils used in the Pelletron Accelerator of the University of São Paulo are made by usual vacuum evaporation of pure carbon rods. Many of the foils are slackened in an apparatus similar to those used in München¹⁾. Almost all foils are chosen examining them through a microscope to avoid foils with pinholes or with big imperfections.

The stripper foils are mounted on a standard NEC foil exchanger with 88 frames. Usually the whole batch is changed each four or five months which is not a bad performance, for the moment. Nevertheless, if heavier projectiles are to be used frequently, longer lifetimes are preferable.

The comparison between the mean lifetime of the strippers obtained in our laboratory (mostly ¹⁶O beam) with the published values for heavy beams as Cl, Ar and S, really shows the need of further investigations in the productions of long lived foils.

Many international laboratories showed good performances using stripper foils obtained by cracking of ethylene^{2,3,4)}. This technique was tried also in our laboratory in the early eighties. The average lifetime was much better than those of the usual evaporated strippers; but the reproducibility of the best conditions is very difficult, for some not yet identified reason. It was also seen that

the root mean square error in the lifetime was about 100% of the mean lifetime, in contrast to about 30% for the evaporated foils. (It should be mentioned that the lifetimes are calculated using a notebook written by the users of the accelerator, not always carefully filled!). A mixture of ethylene and argon was used to get the plasma, with commercially available gases (both ethylene and argon were provided by Oxigênio do Brasil).

The carbon arc method is an old technique but still used in many laboratories with success^{5,6}). As pure carbon rods are available, it is possible that good strippers can be obtained in a reproducible way. Carbon rods were cut from a hard and pure graphite block used in nuclear reactors (given by Instituto de Pesquisas Energéticas e Nucleares, São Paulo). We also used carbon from Balzers, 3 mm diameter.

A schematic view of the apparatus can be seen in figure 1.

One batch of strippers which are within the accelerator shows that the carbon foils evaporated from a hard graphite have longer lifetimes. We are now producing stripper foils exactly in the same way as those, in order to test within a statistically reasonable number of foils.

Although sophisticated optical facilities are not available in our laboratory, the glass substrates with carbon are being examined through a microscope.

REFERENCES:

1. P. Maier-Komor, E. Ranzinger and H. Münzer
Proceedings of the 3rd International Conference on Electrostatic Accelerator Technology, page 163.
2. B. Huck, E. Jaeschke, W. Kratschmer, R. Repnow and H. Wirth
Nuclear Instruments & Methods 184 (1981) 215-221.
3. N.R.S. Tait, D.W.L. Tolfree, B.H. Armitage and D.S. Whitmell
Nuclear Instruments & Methods 167 (1979) 21-24.
4. J.L. Gallant and P. Dmytrenko
Nuclear Instruments & Methods 200 (1982) 1-3.
5. S. Takeuchi and S. Kanazawa
Nuclear Instruments & Methods 206 (1983) 331-333.
6. I. Sugai, T. Fujino, K. Yamazaki, T. Hattori & M. Ogawa
Nuclear Instruments & Methods A236 (1985) 576-589.

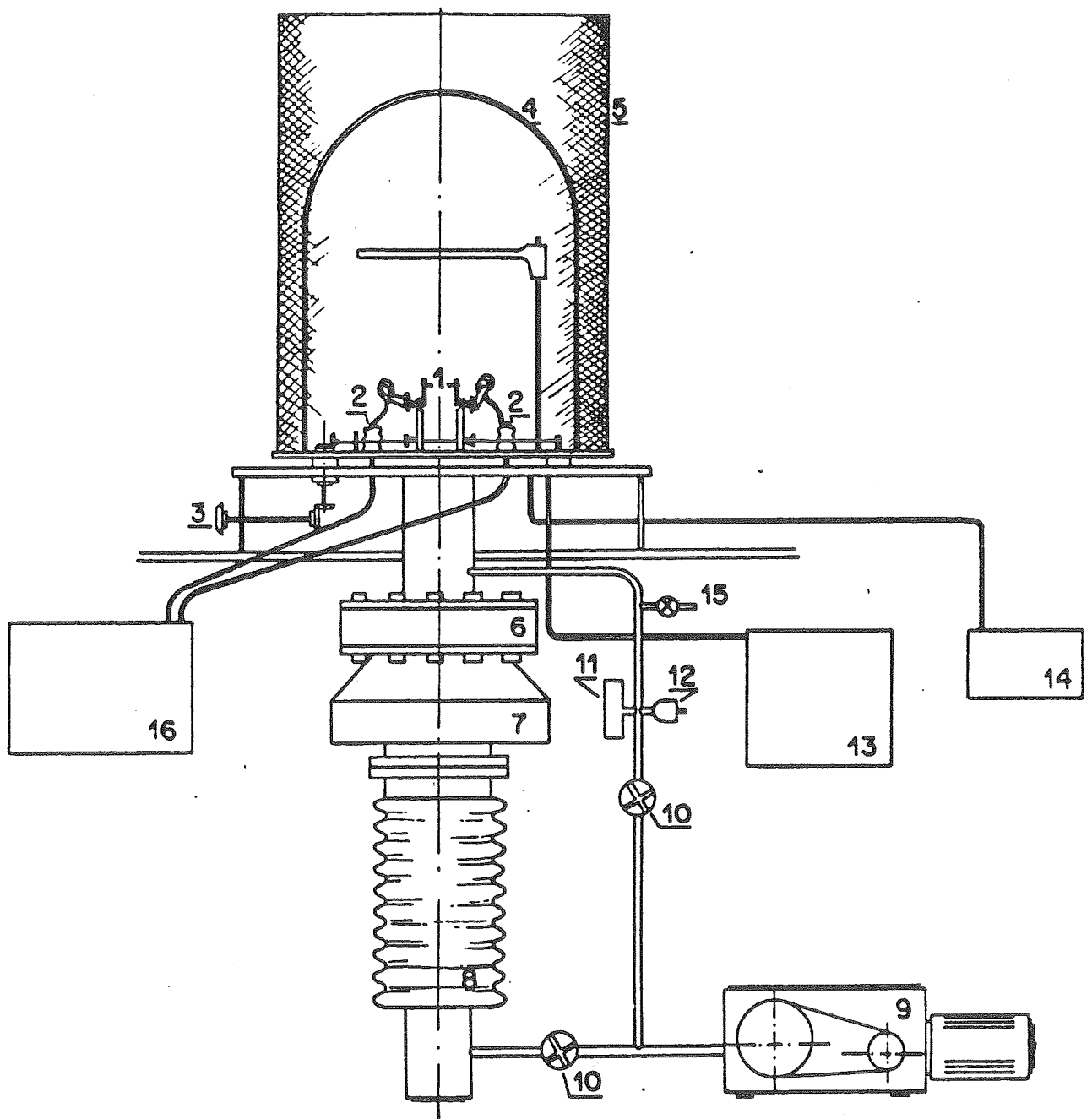


Figure 1. 1-carbon rod; 2-feed through; 3-mechanical coupling; 4-bell jar; 5-implosion guard; 6-valve; 7-liquid nitrogen trap; 8-diffusion-pump; 9-mechanical pump; 10-valves; 11-bad vacuum protection; 12-thermo couple; 13-substrate heater; 14-high vacuum meter; 15-air or gas inlet; 16-current supply.

Activity report at ISN Grenoble

R. BLANC, M. BOURIANT, J.P. RICHAUD.

INSTITUT SCIENCES NUCLEAIRES

53, Av. des Martyrs 38026 Grenoble.

During this last period, the activity of our group was mainly devoted to preparation of thin targets used in heavy ion nuclear reaction experiments. Most of the 350 targets prepared are reported in table I, with specificities, methods and results.

In addition, new techniques have been developed and improved for the various applications mentioned below.

1) Carbon strippers

Properties of thin carbon foils used under heavy ion beams at SARA and GANIL accelerators have been systematically studied for more than 250 samples. (50 - 100 - 150 - 200 $\mu\text{g}/\text{cm}$ thicknesses).

A comparison with commercial products is in progress.

2) B_2O_3 targets

Experiments based upon (d, n) reactions on B_2O_3 targets have been developed at ISN in nuclear medicine.

As the $^{11}\text{CO}_2$ produced is used to synthesize organic acids primary B_2O_3 samples have to be free of CO_2 . The method used involves a fusion under vacuum followed by a fusion under helium.

By use of a new tubular, horizontal oven heating up to 1400°C , series of 15 targets have been prepared and 170 samples have been obtained.

3) ^{44}Ca target

A self supporting ^{44}Ca target, $780 \mu\text{g}/\text{cm}^2$ thick and 20 mm of diameter has been prepared in our group from 150 mg of enriched carbonate. The method was based upon the $^{44}\text{CaCO}_3 \xrightarrow{\ominus^\circ} ^{44}\text{CaO} + \text{CO}_2$ reaction followed by $^{44}\text{CaO} + \text{Zr} \xrightarrow{\ominus^\circ} ^{44}\text{Ca} + \frac{1}{2} \text{Zr O}_2$.

7,8 % of efficiency have been reached and the target was free of oxygen and of additional impurity connected to the method.

4) Metallic samples for the ECR ion-source

New metallic heavy ion beams are in progress near the SARA accelerator. Starting from powders several roasted rods of various compounds such as. LiF , Li_2O , Li_2ZrO_3 , CaO , Ca F_2 , CuO have been made. Pressure of 8 tons/ cm^2 were applied to powder samples placed inside a 4 mm diameters cylindrical frame.

Heating under vacuum gives the mechanical rigidity. Up to now lengths of 40 mm have been reached.

In addition pure metallic Ca samples, fused under the atmosphere have been obtained with this method, all other elements having a mass $A = 40$ are eliminated and cannot pollute. The futur heavy ion beam 40 to 60 mm long, parallelepipedic samples, few mm^2 section, have been tooled under inert atmosphere. Those samples are currently used in the ECR ion-source at SARA 800 n Ac of $^{40}\text{Ca}^{11+}$ beams have been already extracted for the physic.

5) Optical "rubber-like" discs

Optical rubber like discs have been produced for a new multidetector involving 148 elements. Several materials have been tested to ensure both transparency and flexibility. Due to the number of discs, a special method has been used. The two component silicone elastomere retained has been spread on a scaled glass frame and polymerized at 35°C . Discs of 68 mm of diameter have been extracted from 4 mm thick plate with a special cutter home made. This technique has been improved on more than 180 samples.

Annisal report

Table I

Starting material form	Target chemical mg/cm ²	Target thickness	Method	Comments
Wire	Al	0,54 à 1	2	
Foil	"	10 à 200	1	
Wire	Ag	0,5 à 1	2	
Foil	"	20 à 200	1	
Wire	Au	0,5 à 1	2	
Foil	"	5 à 200	1	
Powder	¹³⁸ BaCO ₃	2 et 5	3	(c)
Ingot	ACd	1,4 à 2,5	1	(a) ou (b)
"	Ca	1 à 4	2	
Powder	⁴⁴ Ca	0,7	2	(d)
Rod	C	0,01 à 1	2	
Rod or "Papyex"	"	1 à 10	1 ou 2	
Rod	"	2 à 200	4	
Foil	Co	1	1	
Rod	Cu	0,4 à 1	2	
Foil	"	20 à 200	1	
Ingot	⁵⁴ Cr	1	2	
Foil	⁵⁴ Fe	9,5	1	
"	Hf	5 à 11	1	
"	Ho	2	1	(e)
Powder	kCl	1	2	
Foil	AMo	0,6 à 2	1	
"	^{58,60} Ni	1	1	
"	Nb	1,7	1	
Powder	¹²⁴ Os ₂ O ₃	7	3	(c)
Foil	¹¹⁰ Pd	1,6	1	
Ingot	²⁰⁸ Pb	1	2	
Rod	Pb	5 à 100	1	
Foil	Sc	1	1	
Ingot	ASn	0,4 à 1	2	
"	"	3 à 4	1	
Foil	Ta	1	1	
Powder	¹³⁰ Te	2 à 5	3	
Foil	Tb	20	1	(e)
"	ATi	0,9 à 10	1	
Oxide powder	^{173,174} Yb	1 à 5	2	(d) ou (c) et (e)
Ingot	Y	1,5	2	
"	⁶⁴ Zn	1,5	1	
Foil	^{90,94} Zr	0,55 à 3,4	1	

- 1 : Cold rolling between stainless steel plates.
- 2 : Vacuum evaporation.
- 3 : Powder centrifugation.
- 4 : Slicing and lapping.

- (a) : $25\mu\text{m}$ Pb backing.
- (b) : 30 mg/cm^2 Au backing.
- (c) : $3\mu\text{m}$ mylar backing.
- (d) : vacuum reduction and vacuum evaporation.
- (e) : Cold rolling under Argon.

SAMPLES AND TARGETS FOR NUCLEAR MEASUREMENTS

DELIVERED BY CBNM DURING 1988

J. Pauwels, C. Ingelbrecht, R. Eykens

Commission of the European Communities

Joint Research Establishment - Geel Establishment

1. SAMPLES AND TARGETS ON REQUEST

During the first 11 months of 1988 245 samples corresponding to 58 orders from 14 customers have been supplied. These are:

- (1) thin deposits prepared by vacuum deposition using electron beam or resistance heating, by air-brush spray painting or by electro spraying of solutions or suspensions. Cathodic sputtering was also used to provide protective or conductive layers on substrates. The α -emitting deposits were characterised by low-geometry α counting and the thickness of non-radioactive layers was measured by spectrophotometry, by oscillating quartz crystal or by weighing;
- (2) thin foils of polyimide used as substrates or supplied directly to interested customers. These were prepared by mixing the liquid ingredients and polymerisation on a rotating disc. The foils were then floated off in water and collected on aluminium rings or frames;
- (3) bulk samples of metals, alloys or oxide powders. Alloys were prepared by induction melting either in a crucible or directly on a water-cooled pedestal without a crucible. An alloy of composition ^{238}U -20% ^{235}U -3% ^{239}Pu was requested as a candidate spike material for uranium and plutonium IDMS measurements. A 15 g button was prepared by crucible-less induction melting and then cut to produce individual solid spikes of mass about 120 mg. The plutonium homogeneity was subsequently measured by the MS group and found to be $\pm 0.2\%$ (1s) for individual solid spikes. This offers the possibility of more accurate plutonium determinations in feed solutions.

An intermetallic compound of HfTe_5 was prepared by blending of the elemental powders and heat treatment inside evacuated quartz ampoules. Lattice parameters were later confirmed by X-ray diffraction (Analytical Sciences group).

Studies of the determinations of metal in metal matrices by solid sampling Zeeman atomic absorption have been continued. In particular,

measurements of silver, cobalt, iron and cadmium in copper have been carried out. This technique has the advantage of rapid sampling on small sample sizes and will be particularly useful for homogeneity determinations of metals and dilute alloys for nuclear and non-nuclear applications.

The SP group is collaborating with Sussex University, NIST and ILL in a new measurement of the neutron lifetime. Layers of ^{10}B and ^6Li have been prepared for use in these experiments and characterisation of these layers is continuing.

Table 1 . Supply of thin deposits, thin films and bulk material

Preparations	Number of Orders	Number of Samples	Preparation Methods ¹⁾
<u>Thin deposits</u>			
^{10}B on silicon	2	4	VD
^6Li on silicon	2	4	VD
Platinum on stainless steel	1	4	CS
Lithium on tantalum	1	1	VD
Aluminium on proportional counters	1	6	VD
^{243}Am on polyimide or Al	2	2	SO-EL
^{241}Am on titanium	1	6	SP
^{241}Pu on polyimide	1	1	SO
^{239}Pu on Ti or stainless steel	7	29	VD,SP,RC
^{233}U on Ti or molybdenum	4	19	SP, SO
^{235}U on Ti, Ta, polyimide, Al or stainless steel	9	33	VD,SP,SU,SO
^{236}U on aluminium	1	1	SU
^{238}U on tantalum	1	6	SU
^{232}Th on aluminium	1	2	SU
^{237}Np on nickel	1	2	VD
^{26}Mg on tantalum	1	1	CAN
Al or Al_2O_3 on zirconia	1	3	CS
Gold on polyimide	1	1	VD
CaF_2 on tantalum	1	1	VD
<u>Thin foils</u>			
Polyimide	2	6	CE
Polyimide on glass	2	39	CE

Samples	Number of Orders	Number of Samples	Preparation Methods ²⁾
<u>Bulk samples</u>			
Tantalum	1	3	MA
Copper	1	6	WD
HfTe ₅	1	5	HT
UO ₂	1	17	PR - CAN
²⁴³ Am acetate	1	1	CH
¹⁵⁷ Gd ₂ O ₃	1	1	CAN
²³⁹ PuO ₂	1	1	PR - CAN
Cadmium	1	1	M
Fe ₂ O ₃	1	1	PR - CAN
Ni-Cr	1	2	R - MA
Aluminium	1	6	R - MA
⁶ Li	1	1	R - MA - CAN
²³⁵ U- ²³⁸ U- ²³⁹ Pu	1	20	M - MA
Au-Cr	1	9	M
Au-Fe	1	1	M

1) List of preparation methods

- CAN canning
- CE centrifuging a polymer solution
- CH chemical
- CS cathodic sputtering
- EL electrolysis
- H heat treatment
- M melting
- MA machining
- PR pressing
- R rolling
- RC counting/calibration
- SO solution spraying
- SP spraypainting
- SU suspension spraying
- VD vacuum deposition
- WD wire drawing
- VD vacuum deposition
- WD wire drawing

2. REFERENCE MATERIALS FOR REACTOR NEUTRON DOSIMETRY

CBNM is collaborating with the Euratom Working Group on Reactor Dosimetry to produce a series of reference materials for neutron metrology for reactor surveillance and other purposes. These are activation dosimeters of alloys or high purity metals or fission dosimeters of uranium or neptunium oxide. The preparation of the metallic reference materials is being carried out by the Sample Preparation group who are also co-ordinating and participating in the characterisation programme. New materials are made available for sale as soon as sufficient analysis results have been received to allow a provisional certificate to be issued. During 1988 up to December 57 units corresponding to 4 orders have been supplied.

Table 2 Reactor neutron dosimetry Reference Materials available for sale

Material	Form	Diameter or Thickness [mm]	Status
²³⁸ U oxide (NRM 501)	Spheres	0.5 1.0	Certification report redrafted
²³⁷ Np oxide (NRM 502)	Spheres	0.5 1.0	Certification analyses ongoing
Ni (EC-NRM 521)	Foil Wire	0.1 0.5	EC certificate and report available
Cu (NRM 522)	Foil Wire	0.1 1.0 0.5 1.0	Analyses complete Provisional certificate available
Al (NRM-523)	Foil Wire	1.0 0.1 1.0	Analyses complete. Provisional certificate available
Fe (NRM 524)	Foil Wire	0.1 0.5	Analyses complete Provisional certificate available
Niobium (NRM 525) (NRM 526)	Foil Wire	0.1 0.02 0.5	Analyses complete Provisional certificate available
Al-0.1 Co (NRM 527)	Wire	0.5	CBNM certificate available
Al-1.0 Co (NRM 528)	Wire	0.5	CBNM certificate available

Two new dosimetry reference materials NRM 527 (Al-0.1Co) and NRM 528 (Al-1.0Co) have been produced during 1988. Only a relatively small quantity of material has been certified in each case in response to urgent requests that these alloys be made available. It is not intended that these reference materials be submitted for full EC certification, but CBNM certificates covering the cobalt content in each alloy have been issued. It has also been shown that reference materials 527 and 528 meet a demanding requirement for cobalt homogeneity of 0.5 % relative standard deviation with sample masses of 20 mg. This approximately represents the dosimeter sample size that will be used. The participating laboratories were SCK/CEN Mol (neutron activation analysis) and CBNM Sample Preparation (spectrophotometry). The preparation of a larger batch of Al-Co alloy for full EC-Certification is planned.

High purity rhodium powder (300 g) containing about $22 \text{ mg}\cdot\text{kg}^{-1}$ iridium and $8 \text{ mg}\cdot\text{kg}^{-1}$ platinum has been transformed into foil. The powder was compacted into pellets, each of 100 g, by "semi-levitation melting" i.e. induction melting with the sample supported on a pedestal, but without a crucible. The buttons were then individually sealed into stainless steel envelopes and rolled at 900°C . The samples were resealed in new envelopes after every one or two passes. The resulting thick foil was then cold rolled with annealing at 1000°C under an inert atmosphere initially after every pass. The final foil thickness was $50 \mu\text{m}$. The rolling of the complete 300 g batch is complete and the characterisation of the material is being planned.

The certification report for NRM 501 ($^{238}\text{UO}_2$) has been redrafted to include additional data on the homogeneity of the uranium content between individual spheres (CBNM Mass Spectrometry). The revised report will be distributed shortly for approval by the Nuclear Certification Group.

The 53rd meeting of the EWGRD took place at JRC Ispra in April. The Monitor Materials Subgroup concluded that demand for present and future EC certified dosimetry reference materials should be stimulated by distribution of a technical advertising brochure. This is necessary because of a general slow-down in reactor building programmes, and because of a tendency to rely on old stocks of dosimetry reference materials. The brochure is being prepared at CBNM.

Source : CBNM Programme Progress Report 1988

Information on literature

- Deposition techniques for the preparation of thin nuclear targets
Vacuum ; Volume 37 ; numbers 11/12 ; pages 785 - 817 / 1987
A.H.F. Muggleton
Dep. of Nuclear Physics, Research School of Physical Sciences ;
The Australian National University, GPO Box 4, Canberra, Act 2601,
Australia

- The influence of target and sample properties on nuclear data measurements
Summary report INDC (NDS)-220/G, MY
IAEA Advisory Group / INTDS
CBNM, Geel : 21 - 24 September 1987
Edited by : K. Okamoto (January 1988)

- The influence of target and sample properties on nuclear data measurements
Summary report INDC (NDS)-213/G + , M3
IAEA Advisory Group / INTDS
Darmstadt, Federal Republic of Germany : 5 - 9 September 1988
Edited by : K. Okamoto (October 1988)
IAEA, Nuclear Data Section
Wagramstrasse 5
A - 1400 Vienna