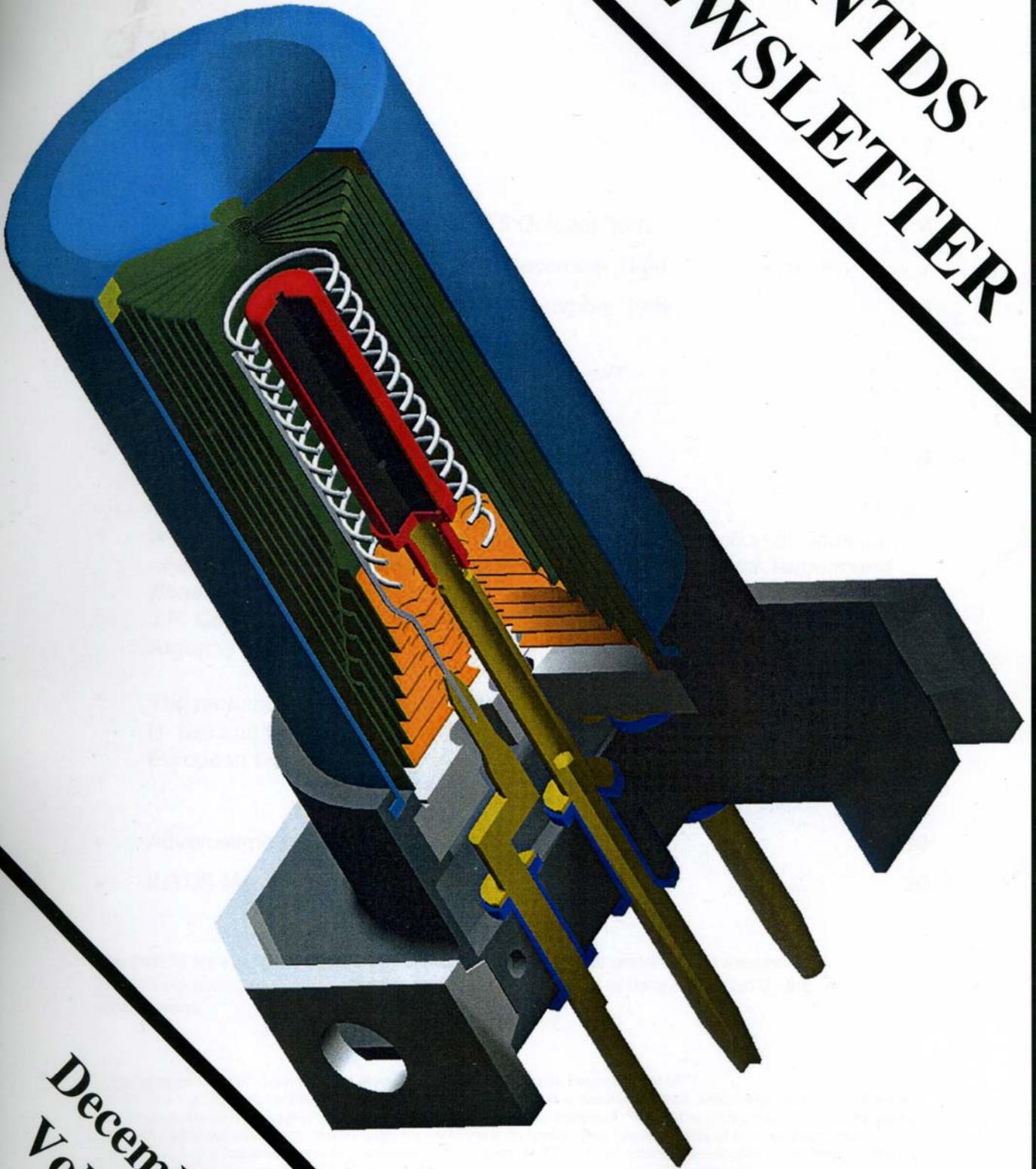


**INTIDS
NEWSLETTER**



**December 1999
Vol. 26, No. 2**

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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 contributors.

Cover picture: Ion Source for the Munich Accelerator for Fission Fragments (MAFF)

For the new high flux reactor FRM II, a fission fragment accelerator is under construction, which will supply intense, mass separated radioactive ion beams of neutron-rich nuclei, produced by fission of ^{235}U [1]. The picture shows an artists view of the integrated target-ion source, featuring the fission target in its centre. This target consists of 99% enriched $^{235}\text{UC}_2$, dispersed in a graphite matrix, which is encapsulated in a protective Re sheath having a small aperture for beam extraction at the front. Find more information in the Proceedings of the 19th World Conference of the INTDS [2]

[1] D. Habs et al. (eds.), MAFF – Physics Case and Technical Description, Oct. 1998,
<http://www.ha.physik.uni-muenchen.de>

[2] H.J. Maier et al., Target Conception for the Munich Accelerator for Fission Fragments, NIM A, to be published

Editor's Note

Thank you to the regular contributors to the Newsletter. Your articles are much appreciated and, we hope, an inspiration to other members, who we seldom hear from, to make laboratory reports or more ambitious contributions.

The millennium INTDS Conference will be held on October 2 – 6 in Antwerp, hosted by IRMM, as previously announced. It is the intention of the organisers to attract a truly international and representative audience and some funds will be available to support the attendance of young target makers or those who may not otherwise be able to travel. "Deserving cases" are invited to contact me.

Good cheer for the New Year and I hope to see many of you in 2000.

Chris Ingelbrecht
INTDS Newsletter Editor
ingelbrecht@irmm.jrc.be

Retirement announcement

Joe G. Tracy
Oak Ridge National Laboratory
December 17, 1999

After more than 40 years of dedicated service, Joe Tracy has decided to retire from the Oak Ridge National Laboratory. Joe came to Oak Ridge in 1959 after serving in the US Navy and completing his degree in Mathematics and Physics from Eastern Kentucky State University. Joe helped start the Isotopes Program in Oak Ridge, beginning in the calutron operations group. He advanced to become the Manager of the ORNL Isotope Enrichment Facility (Calutrons) in 1981 and is widely recognized as an expert in the electromagnetic enrichment of stable and actinide isotopes. Over these 40 years, he has been a key contributor to the success of the Isotope Program that has made invaluable contributions to research, medicine, and industry around the world.

Along the way, Joe received a patent for a novel method of separating silicon isotopes, several Department of Energy and ORL commendations and awards, and has authored approximately 30 publications.

As a member of the International Nuclear Target Development Society and the International Isotope Society, as well as in his roles within the ORNL Isotope Program, Joe has made many good friends around the world. While the ORNL staff is very sad to see him leave the Calutrons, they are very happy that he can now enjoy a well-deserved retirement. Joe's wife, Billye, two young grandsons, and many area golf courses should help keep his retirement days busy and entertaining.

W. Scott Aaron

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Heaviest Element News

Helmut Folger *

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Nearly all accelerator experiments are bound to the interaction between energetic charged particles and our targets which have to be prepared carefully of chemical elements, chemical compounds or out of the great number of stable isotopes. In the GSI target laboratory, for instance, heavy-ion targets were fabricated from nearly all chemical elements and from a large number of isotopes up to the heaviest naturally occurring species, U, as well as of isotopes of the actinoids Pu and Cm. No samples were made of Tc, Po, At, Fr, Ra, Ac, Pa and the rare gases. The best survey of all chemical elements for targets is the Periodic Table of the Elements shown in the adjacent figure.

The heaviest elements of the table cannot be regarded as target material; they are rather the outcome of interactions of an accelerator-beam with a target; they are short-lived and produced in 'sub-target' quantities only. For a long time there was a discussion about the names of the heavy actinoids (elements **101-103**) and of the known transactinoids (elements **104-112**). The IUPAC has published in the meantime their recommended names for the elements **101-109** to be as follows:

The heavy actinoids:	101Md (<i>Mendelevium</i>),	102No (<i>Nobelium</i>),	103Lr (<i>Lawrencium</i>).
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The transactinoids:	104Ru (<i>Rutherfordium</i>),	105Db (<i>Dubnium</i>),	106Sg (<i>Seaborgium</i>),
	107Bh (<i>Bohrium</i>),	108Hs (<i>Hassium</i>),	109Mt (<i>Meitnerium</i>).

The names for the heaviest elements **110-112** being produced at GSI have been proposed and submitted. The target relevant aspects for the productions of the elements **107-112** were explained at INTDS World Conferences each time, and a review of several development stages was presented by *H. Folger et al., NIM A334 (1993) 69*.

Recently, new heaviest elements were observed in Berkeley, USA and in Dubna, Russia:

a) A fusion reaction was investigated at the LBL, Berkeley, USA, with $C/^{208}\text{Pb}/C$ targets of the GSI type and a ^{86}Kr beam in the reaction: $^{86}_{36}\text{Kr} + ^{208}_{82}\text{Pb} \rightarrow ^{293}\mathbf{118} + ^1_0\text{n}$. The evaporation residue $^{293}\mathbf{118}$ is the heaviest element detected to date. It decays by subsequent emission of ∇ -particles into the nuclei $^{289}\mathbf{116}$, $^{285}\mathbf{114}$, $^{281}\mathbf{112}$, $^{277}\mathbf{110}$ and $^{273}\mathbf{108}$. Three decay chains have been reported with the respective half-lives and reaction cross-section. This means that in the one experiment the three transactinoid elements **114**, **116** and **118** were observed by *V. Ninov et al., Phys. Rev. Lett. 83,Nr.6 (1999) 1104*.

b) At the JINR, Dubna, Russia, element **114** was seen in two fusion reactions with ^{48}Ca beams using ^{242}Pu and ^{244}Pu targets, i.e.: $^{48}_{20}\text{Ca} + ^{242}_{94}\text{Pu} \rightarrow ^{287}\mathbf{114} + 3\ ^1_0\text{n}$, and $^{48}_{20}\text{Ca} + ^{244}_{94}\text{Pu} \rightarrow ^{289}\mathbf{114} + 3\ ^1_0\text{n}$. The evaporation residue $^{287}\mathbf{114}$ decays with a half-life of 1.3 s by ∇ -emission into a spontaneous fissioning $^{283}\mathbf{112}$, as published by *Yu.Ts. Oganessian et al., Nature 400 (1999) 242*. The heavier evaporation residue $^{289}\mathbf{114}$ decays with a half-life

of 30 s by ∇ -particle emission into $^{285}\text{112}$ and further by subsequent ∇ -decay into $^{281}\text{110}$ and $^{277}\text{108}$ following *Yu.TS. Oganessian et al., Phys. Rev.Lett., 83 (1999) 3154.*

Chemically the new heaviest transactinoids belong to the 6d transition elements as proposed already by *B. Fricke and W. Greiner, Phys. Lett. B30 (1969) 317*. As a result of the short half-lives and the low reaction cross-section these elements are difficult to investigate using chemical procedures. In 1997 it was possible to isolate and investigate radio-chemically the element ^{106}Sg from the ^{22}Ne -bombardment of a ^{248}Cm target at GSI Darmstadt. The produced $^{265,266}\text{Sg}$ was found to be a homologous of ^{74}W , as described by *M. Schädel et al., Nature 388 (1997) 55*. This year a ^{249}Bk target was successfully bombarded with ^{22}Ne at the Paul Scherrer Institute, Villigen, Switzerland; the data are now analyzed with respect to the chemical behavior of ^{107}Bh by an international collaboration. For experiments next year preparations are made to look into the chemistry of ^{108}Hs using a beam of ^{26}Mg and targets of the isotope ^{248}Cm . An international collaboration plans also to investigate the chemistry of element 112 in the interaction of a ^{48}Ca beam with ^{238}U target layers in Dubna (*Private communication, M. Schädel, GSI Darmstadt*).

The outcome of all the interesting experiments will provide information about the structures, decay properties, reaction cross-section and chemical properties of the elements and their isotopes in the region of the island of stability around the super-heavy element 114. The heavy-ion fusion experiments will provide us with more exciting Heaviest Element News in the future.

Periodic Table of the Elements																		^1H	^2He		
^3Li	^4Be															^5B	^6C	^7N	^8O	^9F	^{10}Ne
^{11}Na	^{12}Mg															^{13}Al	^{14}Si	^{15}P	^{16}S	^{17}Cl	^{18}Ar
^{19}K	^{20}Ca	^{21}Sc	^{22}Ti	^{23}V	^{24}Cr	^{25}Mn	^{26}Fe	^{27}Co	^{28}Ni	^{29}Cu	^{30}Zn	^{31}Ga	^{32}Ge	^{33}As	^{34}Se	^{35}Br	^{36}Kr				
^{37}Rb	^{38}Sr	^{38}Y	^{40}Zr	^{41}Nb	^{42}Mo	^{43}Tc	^{44}Ru	^{45}Rh	^{46}Pd	^{47}Ag	^{48}Cd	^{49}In	^{50}Sn	^{51}Sb	^{52}Te	^{53}I	^{54}Xe				
^{55}Cs	^{56}Ba	^{57}La	^{72}Hf	^{73}Ta	^{74}W	^{75}Re	^{76}Os	^{77}Ir	^{78}Pt	^{79}Au	^{80}Hg	^{81}Tl	^{82}Pb	^{83}Bi	^{84}Po	^{85}At	^{86}Ra				
^{87}Fr	^{88}Ra	^{89}Ac	^{104}Rf	^{105}Db	^{106}Sg	^{107}Bh	^{108}Hs	^{109}Mt	110	111	112	113	114	115	116	117	118				
Lanthanoids		^{58}Ce	^{59}Pr	^{60}Nd	^{61}Pm	^{62}Sm	^{63}Eu	^{64}Gd	^{65}Tb	^{66}Dy	^{67}Ho	^{68}Er	^{69}Tm	^{70}Yb	^{71}Lu						
Actinoids		^{90}Th	^{91}Pa	^{92}U	^{93}Np	^{94}Pu	^{95}Am	^{96}Cm	^{97}Bk	^{98}Cf	^{99}Es	^{100}Fm	^{101}Md	^{102}Nb	^{103}Lr						
Transactinoids:		104-118			GSI-TL targets:						Heaviest elements produced at GSI:			107-112							

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A PROCEDURE FOR IMPROVED YIELDS IN THE ELECTROLYTIC REDUCTION OF CADMIUM AND ZINC OXIDES TO BE USED IN THE PREPARATION OF ACCELERATOR TARGETS AND BEAMS

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Abstract

A procedure is presented for improved yields in the electrolytic reduction of the oxides of cadmium and zinc to the metallic form and subsequent use in the production of accelerator targets and beams. A description of the apparatus used is given along with a discussion of some of the preparation techniques used for these elements.

1. Introduction

For the preparation of isotopic targets of cadmium and zinc metals, the enriched oxide obtained must first be reduced to the elemental form. A reduction/distillation procedure employing carbon as the reducing agent exists [1], along with more recent work by Marcus and Zevenbergen [2], for producing high quality isotopic zinc metal starting from the oxide. We chose, instead, the more efficient method of electrodeposition. This method has the advantage of high yields using small quantities of precious isotope and directly produces the cadmium and zinc metal which is then fashioned into targets by any of the known target preparation techniques.

2. Description of the Electroplating Apparatus

Our experimental apparatus is based on the original set-up of Heagney and Heagney [3]. The electrolysis is created through a simple circuit that includes a DC power supply, a platform elevator, and a stainless steel sheet to support a 10-ml Pt crucible. The stainless steel plate/crucible sits on the platform and is electrically isolated by a nylon block. It is important to note that a hardened tool steel rod be used for the collection electrode. Figure 1 shows a schematic of the electrolytic reduction apparatus.

3. Electrolytic Reduction of Zinc and Cadmium Oxide to Metal

3.1 Procedure

The procedure for zinc electroplating starts with the preparation of the electrolyte solution. The ZnO powder is first ground using a mortar and pestle and dissolved into 5% acetic acid using a hot plate to expedite the dissolution. The end of the hardened tool steel rod used as the electrode to collect the zinc is first polished using a diamond wheel. The rod enters the solution by raising the platform, with the bottom of the rod just touching the surface of the solution. The current is then slowly raised to approximately 40mA. A photograph of the experimental equipment used is shown in Figure 2.

Every few minutes the system is checked to make sure it is functioning properly, meaning that the current is relatively constant and zinc is collecting on the electrode. The formation of small bubbles may, at times, occur which impedes the deposition. When this condition

appears, the current needs to be turned off, the bubbles dispersed from the rod by slight stirring, and the deposition restarted.

The first deposition attempt lasts anywhere from an hour to 100 minutes in duration. Successive collections of zinc material from the same solution each will take less time and have to be watched carefully. The second deposition then averaged just over 30 minutes while the expected deposition times for the rest of the trials are no more than 15 minutes each as shown in Table 1. Once the total yield approaches 90%, the remaining depositions last around 5 minutes each. Also given in Table 1 is the amount of zinc recovered for each trial.

After the rod deposited with Zn is withdrawn from the solution, a small petri dish is placed as a cover over the crucible to prevent any Zn metal which does not adhere to the electrode from falling back into the solution. If any Zn falls back into the solution while trying to withdraw the electrode, it is allowed either to redissolve back into solution, or it may be retrieved using tweezers and placed on weighing paper under a heat lamp to dry.

For all trials, an average of one hour and 10 minutes was needed to dry the metal without the use of a heat lamp. The best method found to dry the reduced zinc was first to rinse the metal with distilled water and then flow Argon over it until dry, as this minimizes re-oxidation.

3.2 *Reduction Efficiencies*

For the first solution, 170 mg of the oxide was dissolved in 10 ml of 5 % acetic acid and left on the hot plate overnight, resulting in 7.5 ml of solution the next morning. Using 5 ml of this solution, filling the crucible half-way, should yield approximately 91.5 mg zinc metal. By performing many trial runs over shorter and shorter time duration, the total amount of Zn metal collected approached 100% (see Table 1.). For solutions #2 and #3, 170 mg of ZnO was dissolved in 10 ml of the acetic acid with 5 ml again used in the crucible for electrodeposition. The amount of zinc metal reduced was expected to be 68.3 mg. The reduction efficiencies for these runs were 96.5% and 99.9%, respectively. It is not known whether these high efficiencies were due to the incomplete reduction of the oxide, although the collected material has the appearance of powdered zinc. Further analysis needs to be performed on the reduced material.

We concentrated here mainly on zinc, as it was desirable to obtain reduced metal for the production of a ^{70}Zn accelerator beam, (^{70}Zn has a natural abundance of 0.62%) where the enriched stable isotope is furnished as zinc oxide. For this application, the steel electrode with the material deposited on the tip could be directly introduced into the ion source for sputtering [4]. Alternatively, the collected reduced powdered zinc could be loaded into a miniature high-temperature oven for introduction into the ion source.

4. Preparation of Metal Targets and Foils

4.1 *Resistive Evaporations of metallic Zinc*

Due to the low vapor pressure of metallic zinc, the method of choice for producing thin targets was evaporation by resistive heating from a tantalum boat [5]. The first attempt was

to deposit about $300 \mu\text{g}/\text{cm}^2$ Zn onto $40 \mu\text{g}/\text{cm}^2$ C backings previously mounted on standard ATLAS frames ($2.54 \text{ cm} \times 2.54 \text{ cm}$ with a 12.5 mm aperture) and situated on the fixture 6 cm above the source. A ME6A .015 Ta open boat (R.D. Mathis Co., Long Beach, CA, USA, 90809) was used for this evaporation. Approximately 50 mg of the collected powdered zinc was weighed out and placed carefully in the boat. During the deposition it was observed that little zinc had collected on the carbon targets. The zinc was not "sticking" to the carbon backings even though the deposition was measured to be $200 \mu\text{g}/\text{cm}^2$ using a quartz crystal monitor. Also, it appeared that the fixture which held the carbon targets was coated with zinc.

To investigate alternative backings, $300 \mu\text{g}/\text{cm}^2$ gold foils, mounted on target frames were next placed in the apparatus and an evaporation performed. After the deposition, the gold targets were also observed to have collected little zinc. It was thought that perhaps the zinc was not pure, that possibly there was something from the electrolytic reduction contaminating the material. In order to investigate this possibility, pure zinc shot (99.99%) was evaporated onto the gold backings to check whether or not the zinc would adhere to the gold. This high purity material did adhere to the gold backings, confirming the idea that the electroplated zinc must somehow be contaminated.

For the next deposition, 36 mg from a new batch of reduced zinc was pressed into a pellet and placed into the Ta source boat. A $40 \mu\text{g}/\text{cm}^2$ C slide and a $200 \mu\text{g}/\text{cm}^2$ Au slide were each placed on either side of the thickness monitor for this evaporation. At 70 A current applied to the source boat, deposition occurred and some zinc was collected on the monitor. The slides were checked and were found to contain some deposited film, but most of the zinc remained as a pellet in the boat. The slides were replaced with fresh ones and the deposition continued. When completed, it was found that zinc was evaporated on the gold slide, but it was observed that little zinc had collected on the carbon slide.

In order to further investigate the "sticking" probability of the zinc on gold vs. carbon, another deposition was performed with one carbon and one gold slide, using the pure zinc shot as before. Results from this evaporation were similar to that done using the electrolytically reduced material, with the zinc deposited primarily on the gold slide. When attempting to float this film, a problem was encountered in that the zinc became very flaky and, after mounting it on a target frame, cracked while drying. This prompted a return to mounted gold target backings instead of using slides.

Two mounted $200 \mu\text{g}/\text{cm}^2$ Au target backings were placed in the evaporator along with one gold coated microscope slide of the same thickness. A ME6A .005 W boat (R.D. Mathis Co., Long Beach, CA, USA, 90809) was employed for this deposition, charged with 25 mg of reduced zinc pressed into a pellet. The goal was to collect $100 \mu\text{g}/\text{cm}^2$ Zn onto the gold backings. This worked, however the zinc deposit on the gold coated slide still would not float successfully.

Finally, a deposition was performed on $40 \mu\text{g}/\text{cm}^2$ C mounted target backings, having first evaporated $2\text{-}4 \mu\text{g}/\text{cm}^2$ Au onto them, and then attempting to collect $100 \mu\text{g}/\text{cm}^2$ Zn. At 100 A of applied current, the W boat was glowing slightly and deposition began. The current was slowly increased to 150 A, where the pellet was observed to dance around the boat and so the current was reduced to 100 A, where deposition occurred at a steady rate with little movement of the pellet. The resulting target had a thickness of $109 \mu\text{g}/\text{cm}^2$ Zn.

4.2 *Mechanical Rolling of Zn and Cd*

Thick targets of isotopic Cd and Zn have been obtained from electroplated foils and subsequently thinned to a few mg/cm² by Lozowski [6]. Using the method described above, both cadmium and zinc oxide powder samples were reduced to metal, collected and consolidated for rolling. The resulting metal was compressed in a compaction die and rolled directly in a stainless steel pack [7]. Foils of cadmium and zinc with thicknesses of 1 mg/cm² have been produced rather easily by this method for use as accelerator targets.

5. Conclusion

In conclusion, we have shown by using this modified procedure for the electrolytic reduction of cadmium and zinc reduction efficiencies well above 90% can be achieved. The metals produced can be used to prepare heavy-ion beams or directly fashioned into targets by evaporation onto backings or by mechanical rolling for use in nuclear physics experiments.

Acknowledgments

The authors would like to thank Dr. Walter Henning, the Physics Division Director, and Dr. Irshad Ahmad, the Target Facility Group Leader, for their continuing support of these efforts. We also wish to thank two students, Jennifer Nemanich and Randall Gasa for their contributions, and Dave Kurth for his help in preparing the figures and photograph. This work is supported by the U.S. Department of Energy, Nuclear Physics Division, under Contract No. W-31-109-ENG-38.

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- [7] John P. Greene and George E. Thomas, Physics Division, Argonne National Laboratory, 9700 S. Cass Avenue, Argonne, IL 60439, USA, **PHY-8557-HI-96**, June, 1997

Table 1. Zinc Electroplating Trials

Solution Number	Trial Number	Time (Minutes)	Zn Recovered (mg)	Total Zn Recovered (mg)	Percent Recovered (%)
#1 (91.5 mg Zn measured)					
	1	101	50	50.0	54.6
	2	38	9.3	59.3	64.8
	3	16	12.9	72.2	79.0
	4	14	3.6	75.8	82.8
	5	10	9.4	85.2	93.1
	6	6	4.0	89.2	97.5
Weight of Zn after drying				85.4	97.5
	7	7	3.9	89.3	97.5
	8	5	2.0	91.3	99.8
#2 (68.25 mg Zn measured)					
	1	89	32.7	32.7	48.0
	2	34	18.4	51.1	74.8
	3	12	6.2	57.3	84.0
	4	10	6.0	63.3	92.7
	5	5	2.6	65.9	96.5
#3 (68.25 mg Zn measured)					
	1	92	33.5	33.5	49.1
	2	39	22.3	55.8	81.7
	3	11	6.9	62.7	91.9
	4	5	2.8	65.5	95.9
	5	6	2.0	67.5	99.0
	6	6	0.7	68.2	99.9

ELECTROPLATING APPARATUS

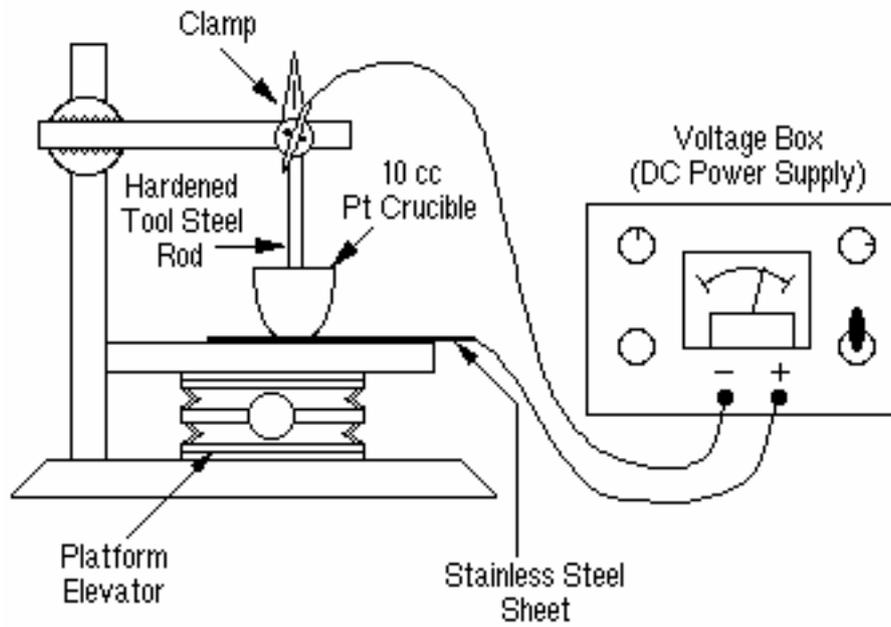


Figure 1. Schematic drawing of the electrolytic reduction set-up based on the method of Heagney and Heagney [1].

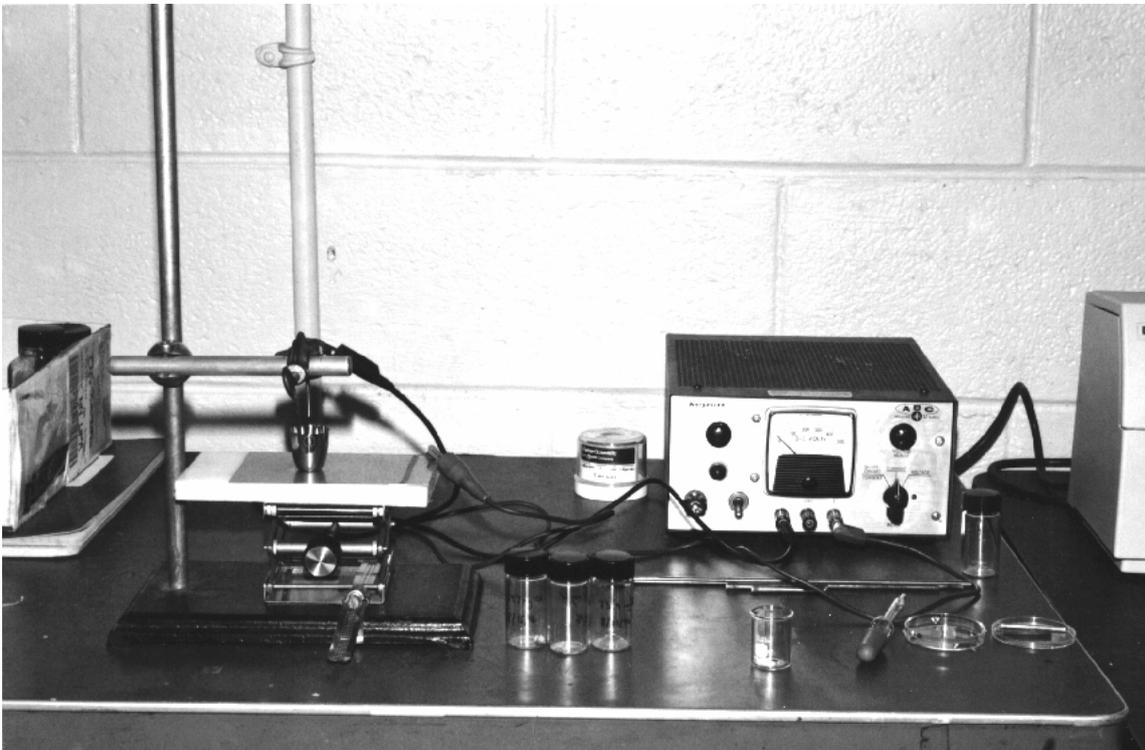


Figure 2. Photograph of the experimental equipment.

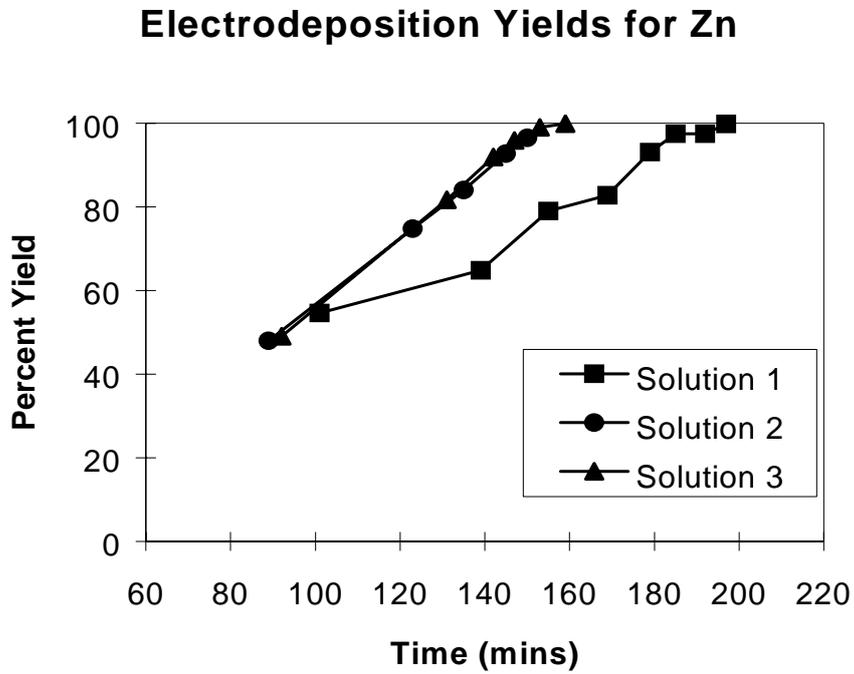


Figure 3. Plot of experimental yield for successive trials of zinc depositions

The preparation of ^{183}W targets by electrodeposition for (n, α) studies

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Introduction

Rectangular samples consisting of a thin, homogeneous ^{183}W layer on an aluminium backing were requested for the use in a (n, α) reaction study. Electrodeposition from organic media appeared to be the most appropriate method. From the literature, this method gives the best results in terms of yield and target quality, and layer uniformity in particular. Because of the limited amount of enriched isotopic material available, experimental dissolution and deposition conditions were investigated by using material of natural isotopic composition.

Target Preparation

A tungsten mother solution of 40 g/l W was prepared by dissolving natural WO_3 in 3.6 M KOH at approximately 65 °C. Addition of 25 – 200 μl of this mother solution to 25 – 50 ml of alcohol (methanol, ethanol, 2-propanol) made up the electrolyte.

Preliminary deposition experiments were performed on etched 40 mm diameter aluminium discs (thickness 0.25 mm), using a polyacetal electrolysis cell with a rotating platinum cathode (6 rpm). The best results were obtained using methanol as a solvent, while precipitation prior to the actual experiment occurred in the electrolytic solutions using ethanol or 2-propanol. Especially in the case of 2-propanol, visually inhomogeneous deposits were produced. Semi-quantitative EPMA-SEM measurements indicated that substantial amounts of tungsten were present in the coatings, despite the imperfect nature of some of them. The presence of potassium appeared to be the main interference.

Based on these results, scaled up deposition experiments were carried out on 20 μm thick aluminium foils, mounted on rectangular brass frames. A PVC electrolysis cell was constructed meeting the requirements of size and shape of the targets. White rectangular deposits with a spot size of 50 x 60 mm^2 were produced, having a thickness between 100 and 200 $\mu\text{g}/\text{cm}^2$. Optimum experimental deposition conditions were as follows:

- three successive deposition cycles (50 μl WO_3/KOH mother solution in 50 ml methanol)
- low current density (0.5 - 1 mA/cm^2)
- 15 min reaction time
- calcination at 120 °C

It must be stressed that the proposed experimental conditions probably do not lead to the highest deposition yield possible, but so far the emphasis in this work was on deposition homogeneity rather than yield.

Contrary to the results with the aluminium discs, adherence problems between the top layer and the aluminium foil were noticed. Although expected, calcination up to 400 °C did not improve this property. According to previous similar studies, treating the aluminium backing with a 2500 grit SiC paper prior to the reaction might be a possible solution.

Future Work

In order to get a more complete picture of the deposited layer, Neutron Activation Analysis (NAA), $^{\text{X-ray}}$ diffraction (XRD) and infrared analysis (IR) will be performed.