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TARGET PREPARATIONS AND THICKNESS MEASUREMENTS

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ABSTRACT

A wide variety of isotope target preparative methods have been used, including rolling of metals, vapor deposition, electrodeposition, chemical vapor deposition, and sputtering, to obtain thin and thick films of most elements or compounds of elements in the Periodic Table. Most thin films prepared for use in self-supported form as well as those deposited on substrates require thickness measurement (atom count and distribution) and/or thickness uniformity determination before being used in nuclear research. In this paper, some preparative methods will be described together with thickness and uniformity determination procedures applicable to samples being prepared (*in situ*) and to completed samples. Only nondestructive methods are considered applicable to target samples prepared by the ORNL Solid State Division, Isotope Research Materials Laboratory. Thickness or areal density measurements of sufficient sophistication to yield errors of <±1% have been achieved with regularity. A statistical analysis procedure is applied which avoids error caused by balance zero-point drift in direct weight measurement methods.

I. INTRODUCTION

Targets of both stable and radioactive materials are required by experimentors for various areas of nuclear or atomic research. Many thin self-supporting targets are used by physicists to obtain more information

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about nuclear structure by bombarding the target nuclei with charged particles and examining the nuclear states formed. Other targets may be used in precision cross-section measurements. In most instances, the methods used in preparing and characterizing the targets is dictated by what the experimentor can tolerate with respect to impurities and thickness variations.

The Isotope Research Materials Laboratory (IRML) at the Oak Ridge National Laboratory was established to provide targets of any material which cannot be obtained commercially. Methods used by IRML to determine the thickness, uniformity, and impurities in the samples will be described. A brief summary of the target preparation techniques is given.

II. TARGET PREPARATION METHODS

In many instances the techniques used by IRML to prepare targets of the various materials are similar to standard techniques described in the literature. However, since most of the samples prepared by IRML consist of very expensive isotopic materials, many of the standard techniques must be modified or new methods established to minimize the amount of material consumed in the process.[1]

Rolling

Normally, separated isotopic materials are inventoried as a compound. Before rolling, the material is reduced to its elemental form and then melted or sintered into a bead. The consolidated mass is flattened by using a hydraulic press and then rolled to the desired thickness. Normally the metal is placed in a stainless steel sandwich for rolling which prevents the material from adhering to the rolls of the mill and enables a much thinner foil to be prepared. A list of the various samples and thickness ranges of materials prepared by the rolling process is included in Table 1.

Vacuum Evaporation

Rolling is by far the most conservative process with regard to material loss in preparing thin targets. However, to obtain ultrathin targets of isotopes in their elemental form and to obtain both thin and thick targets of materials in compound form, vacuum evaporation is frequently used. The vacuum evaporation process is very inefficient and frequently

TABLE 1. Target Preparations[*]

Legend:
a -- evaporation
b -- rolling
c -- electrolytic deposition
d -- casting or pressing
f -- miscellaneous

1 -- self-supporting
2 -- metal backing
3 -- thin carbon

Element	Range of Thickness, $\mu g/cm^2$	Method of Preparation	Backing	Form of Deposit
Aluminum	5 to 12,000	a	2	Al
	150 up	a	1	Al
		d	1	Al
Americium	1 up	a	2	Am or Oxide
	1000 up	b	1	Am
Antimony	500 to 3000	a	1	Sb
Arsenic	10 to 200	a	2	As
Barium	10 to 1000	a	2	Salt
		d	1	Salt
	2000 up	a,b	1,2	Ba
Beryllium	Undetermined	a	1,2,3	Be
Bismuth	500 to 15,000	a	1	Bi
	10 to Undetermined	a	2	Bi
Boron	20 to 250	a	1	B
	10 to 1000	a	2	B
Bromine	10 to 1000	a	2	Bromide
	5000 up	d	1	Bromide
Cadmium	10 to 200	a	2	Cd
	1000 up	b	1	Cd
		d	1	Cd
Calcium	10 to 1000	a	2	Ca
	700 up	b	1	Ca
		d	1	Ca or Carbonate

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[*] Target size, price and delivery schedule are available upon request. Rolled foils are usually 1 x 1 in.^{2}.

Element	Range of Thickness, $\mu g/cm^2$	Method of Preparation	Backing	Form of Deposit
Californium	0.001 to 1.0	c	2	Oxide
		f	2,3	Oxide
Carbon				
Normal	1.5 to 200	a	1	C
Isotopic	10 to 200	f	2	C
Cerium	10 to Undetermined	a	2	Ce Oxide
Cesium	10 to 1000	a	2	Salt
Chlorine	10 to 1000	a	2	Salt
	5000 up	d	1	Salt
Chromium	300 up	c	1	Cr
	10 to Undetermined	a	2	Cr
	100 to 300	d	1	Cr
		a	1	Cr
Cobalt	500 up	b	1	Co
	10 up	a	2	Co
	100 to 1000	a	1	Co
Copper	500 up	b	1	Cu
	10 to 1000	c	2	Cu
	200 up	a	1	Cu
		d	1	Cu
Curium	10 up	a	2,3	Oxide
Dysprosium	1000 up	b	1	Dy
	10 up	a	2	Dy
Erbium	10 to Undetermined	a	2	Er
	1000 up	b	1	Er
Europium	10 to Undetermined	a	2	Eu
	1000 up	b	1	Eu
Fluorine	25 to 500	a	2	Fluoride
Gadolinium	1000 up	b	1	Gd
	10 up	a	2	Gd
Gallium	10 to Undetermined	a	2	Ga
	10 to 200	a	3	Ga_2O_3
		d	1	Ga_2O_3
Germanium	10 up	a	2	Ge
	20 to Undetermined	a	1	Ge

Element	Range of Thickness, $\mu g/cm^2$	Method of Preparation	Backing	Form of Deposit
Gold	10 up	a	2	Au
	Undetermined	a	1	Au
		d	1	Au
Hafnium	Undetermined	b	1	Hf
^{3}H Adsorbed on Zr, Y, Ti or Er				
^{2}H	100 up	f	2	Tritide
Indium	5000 up	b	1	In
	10 up	a	2	In
		d	1	In
Iridium	10 to 100	a	2	Ir
	10 to Undetermined	f	2	Ir
Iron	500 up	b	1	Fe
	10 up	a	2	Fe
		d	1	Fe
Lanthanum	10 to Undetermined	a	2	La
	1000 up	b	1	La
		d	1	La
Lead	7000 up	b	1	Pb
	500 to 8000	a	1	Pb
	10 to 1000	a	2	Pb
		d		Pb
Lithium	4000 to any cast size	b,d	1	Li
	10 to 500	a	3	LiH
	10 to 500	a	2	Li
Lutetium	10 to Undetermined	a	2	Lu
Magnesium	150 up	b	1	Mg
	Up to 20,000	a	1	Mg
		d	1	Mg
Manganese	300 to 1600	a	1	Mn
	10 to Undetermined	a	2	Mn
Mercury	1000 to 4000	c	on Au	Hg
	Undetermined	a	2	Salt
Molybdenum	10 to 1000	a	2	Mo
	500 up	b	1	Mo
Neodymium	10 to Undetermined	a	2	Nd
	1000 up	b	1	Nd

Element	Range of Thickness, $\mu g/cm^2$	Method of Preparation	Backing	Form of Deposit
Neptunium	10 up	a	2,3	Oxide
Nickel	500 up	b	1	Ni
	10 up	a	2	Ni
		d	1	Ni
Niobium	10 up	a	2	Nb
	1000 up	b	1	Nb
Palladium	1000 up	b	1	Pd
	10 up	a	2	Pd
		d	1	Pd
Platinum	1000 up	b	1	Pt
	10 up	a	2	Pt
		d	1	Pt
Plutonium	1 up	a	2	Oxide
	5000 up	b	1	Pu (1% Al)
Potassium	7500 to any cast size	b,d	1	K
Praseodymium	1000 up	b	1	Pr
	10 up	a	2	Pr
Promethium	10 up	a	2	Pm Oxide
		a	2	Pm
	Undetermined	b	1	Pm
Samarium	1000 up	b	1	Sm
	10 up	a	2	Sm
Scandium	10 up	a	2	Sc
	1000 up	b	1	Sc
Selenium	300 to 5000	a	1	Se
	10 to 5000	a	2	Se
		d	1	Se
Silver	1000 up	b	1	Ag
	100 to 5000	a	1	Ag
	10 to 1000	a	2	Ag
		d	1	Ag
Silicon	10 to 1000	a	2	Si
	50 to 400	a	1	Si
	10 to 150	a	2,3	SiO_2
	25 to 150	a	1	SiO_2
Sodium	7500 to any cast size	b,d	1	Na

Element	Range of Thickness, $\mu g/cm^2$	Method of Preparation	Backing	Form of Deposit
Strontium	2000 up	b	1	Sr
Sulfur	5000 up	d	1	S
	10 to 100	a	2	S
Tantalum	1000 up	b	1	Ta
	10 to 1000	a	2	Ta
Technetium	10 to 1000	a	2	Tc
	10 up	c	2	Tc
Tellurium	10 to 1000	a	2,3	Te
		d	1	Te
Terbium	1000 up	b	1	Tb
	10 up	a	2	Tb
Thallium	8000 up	b	1	Tl
	10 up	a	2	Tl
Thorium	4000 up	b	1	Th
	10 up	a	3	Th
Thulium	1000 up	b	1	Tm
	10 up	a	2	Tm
Tin	3000 up	b	1	Sn
	10 to 1000	a	2	Sn
	100 to 4000	a	1	Sn
Titanium	500 up	b	1	Ti
	10 up	a	2	Ti
Tungsten	In Cu matrix	b	2	W
	>2000	d	1	W
Uranium	10 up	a	2	Oxide
	2000 up	b	1	U
Vanadium	1000 up	b	1	V
	10 up	a	2	V
Ytterbium	10 to Undetermined	a	2	Yb
	1000 up	b	1	Yb
Yttrium	10 to Undetermined	a	2	Y
	700 up	b	1	Y

Element	Range of Thickness, $\mu g/cm^2$	Method of Preparation	Backing	Form of Deposit
Zinc	1000 up	b	1	Zn
	10 up	a	2	Zn
Zirconium	500 up	b	1	Zr
	10 to 5000	a	2	Zr

evaporation efficiencies of only 1% are obtained. In general, resistance, electron bombardment, or radiofrequency heating are used by IRML to evaporate the materials which are subsequently condensed on many different substrate materials.

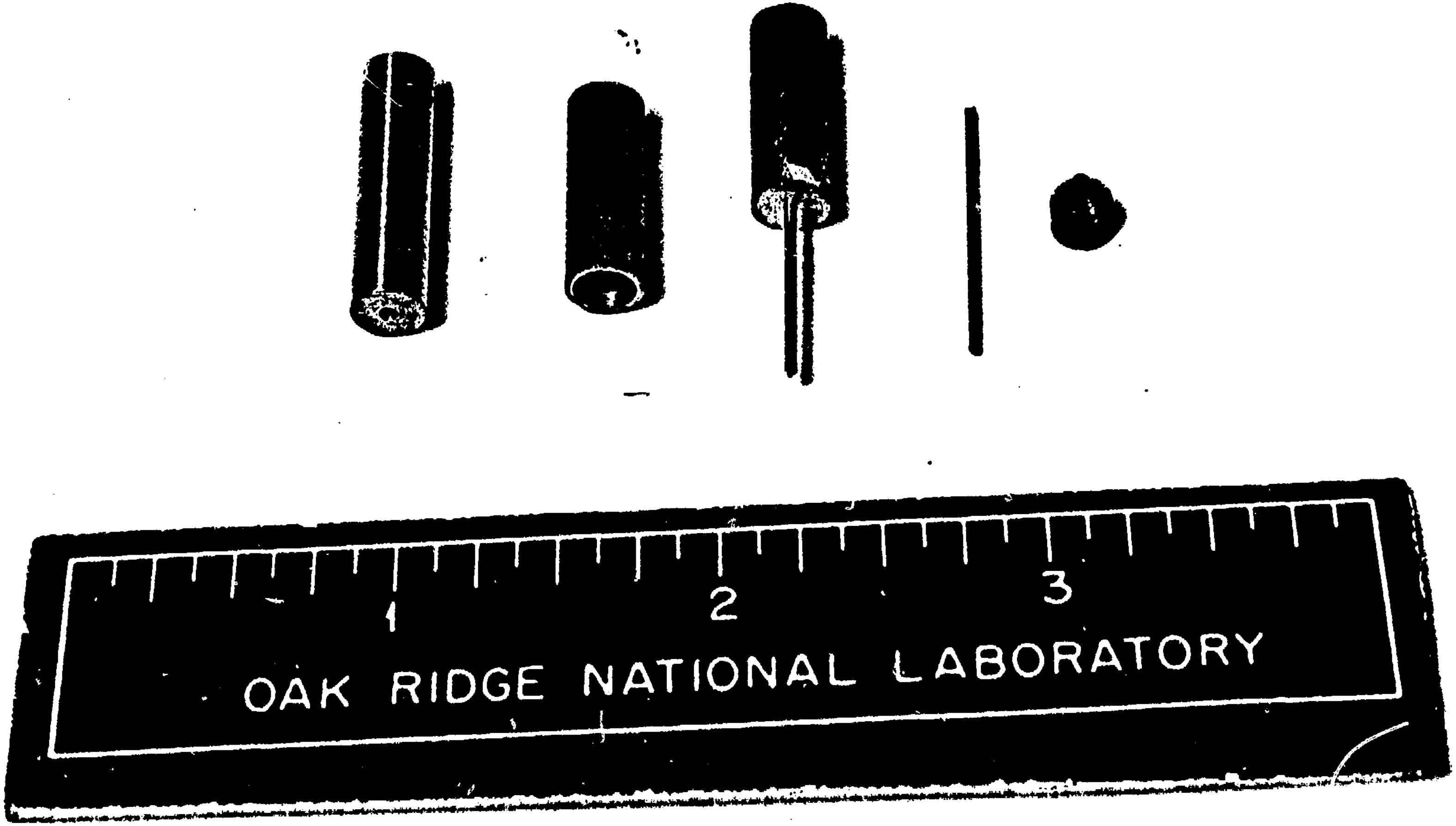
In addition to evaporation efficiency, crucible compatibility with the element or compound must be considered when evaporating the various materials. Normally, expensive isotopic materials are evaporated from tubular carbon, molybdenum, tungsten, or tantalum crucibles similar to those shown in Fig. 1. For normal assay or less costly isotopic materials, commercial electron bombardment guns (Fig. 2) are used to vaporize the material. All actinide materials are evaporated by using an electron bombardment gun or by using radiofrequency heating and tantalum or tantalum carbide crucibles. The vacuum reduction-distillation technique is frequently used to prepare elemental targets or high purity metals directly from oxides.[2-4] A schematic of the system used for the preparation of Cm, Am, and Pu metals is shown in Fig. 3.

Electroplating

Electroplating is used by IRML to prepare targets that are not readily formed by other processes. In particular targets of uranium and ^{252}Cf are prepared by this process. The exact procedures are described in the literature and will not be described here.[5,6]

Sputtering

Sputtering is another technique which is used to prepare targets of various materials such as Al_2O_3 which are difficult or impossible to prepare by other techniques. An inherent problem in this process is the preparation of sputtering targets which are used as the bulk source of



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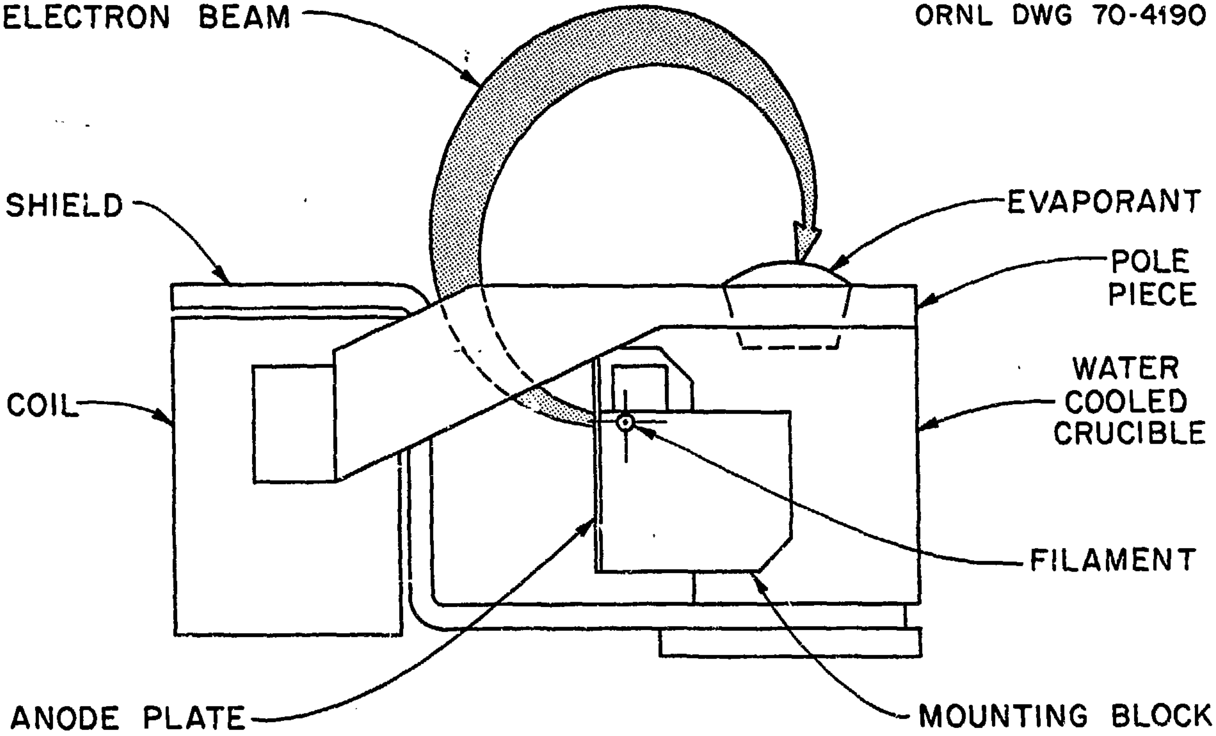
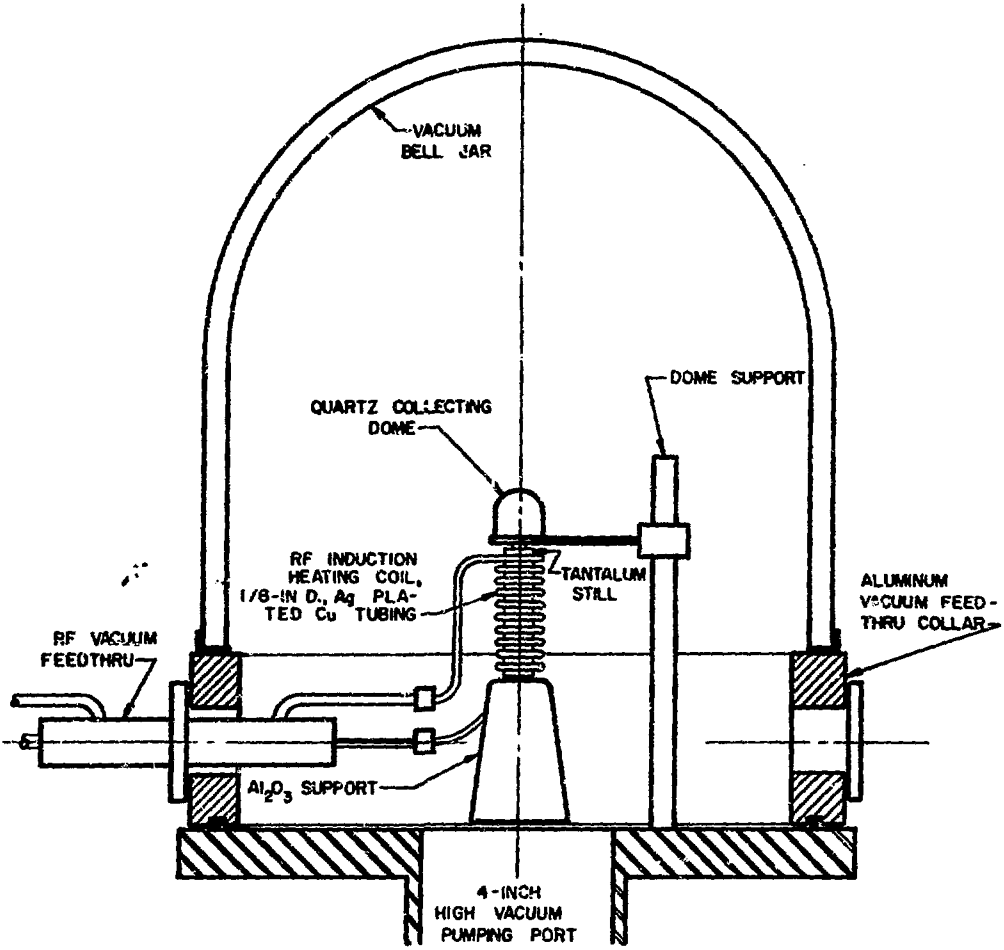
2
3
OAK RIDGE NATIONAL LABORATORY


ELECTRON BEAM
ORNL DWG 70-4190
SHIELD
EVAPORANT
POLE PIECE
COIL
WATER COOLED CRUCIBLE
FILAMENT
ANODE PLATE
MOUNTING BLOCK

ORNL-DWG 69-1090



material to be deposited by the sputtering process. A large number of sputtering targets can be obtained commercially, but IRML prepares isotopic sputtering targets since these are unavailable. The target electrode of a commercial sputtering system has been modified to permit sputtering targets of 6.4 cm diam instead of the 12.7 cm diam targets for which the system was originally designed. Rolled isotopic foils are used as sputtering targets from which material is sputtered and collected on various substrates; this method has proved to be a very reproducible process.

Levitation Melting and Arc Melting

Frequently high purity metal beads of alloys of various elements and/or isotopes are required and are prepared by either arc melting or levitation melting. Figure 4 shows a 100-mg sample of molybdenum being levitated. Levitation melting has the advantage of producing a more homogeneous alloy because of stirring effects (eddy currents) induced by the opposing magnetic fields.[3]

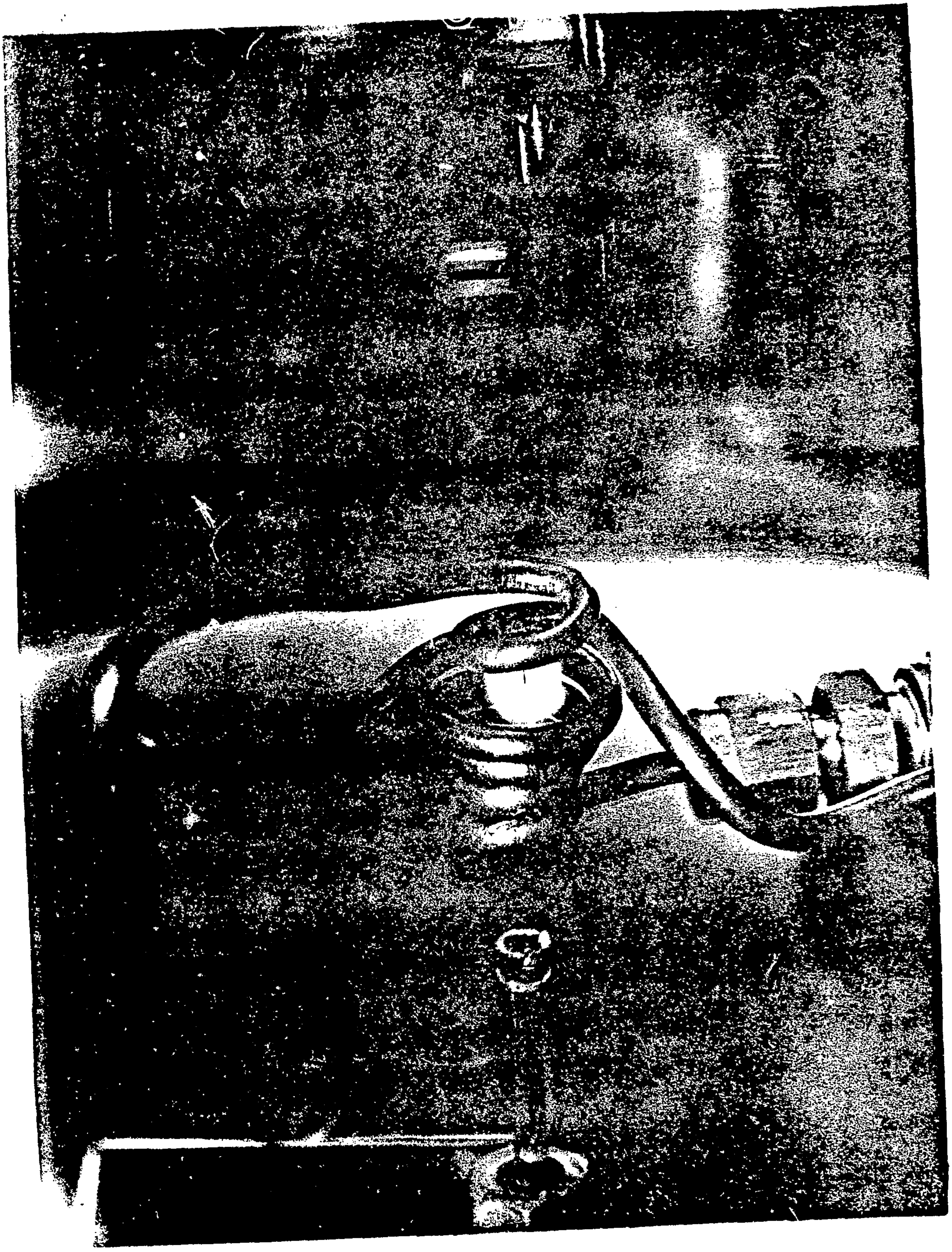
Chemical Vapor Deposition

Chemical vapor deposition of metals from their respective hydrides and halides is used to form films and bulk coatings of isotopic materials such as silicon and zirconium.[7] These elements are very reactive at elevated temperatures and films or high purity samples are difficult to prepare by other methods.

III. TARGET CHARACTERIZATION BY THICKNESS MEASUREMENTS

After preparing the desired target in a specified configuration, it becomes necessary to characterize the sample with respect to thickness, uniformity, and impurities.

Accelerator targets are most frequently described by their weight per unit area or "areal density" for significant reasons. Use of the areal density concept inherently avoids a linear measurement of thickness which may be impossible to perform nondestructively. Linear measurements can lead to grossly erroneous values of atom content by virtue of included microscopic voids or a mixture of various crystal phases. Linear thickness is translated into the number of nuclei in the target by using a value of theoretical bulk density of the target material. A weight measurement,



however, is directly proportional to the number of nuclei in the target provided the stoichiometry of the target is known. Weight measurements can be made during and/or after target preparation by a variety of methods with an accuracy of ≤±0.5% in some cases.

Weighing

The most frequently used method for determining areal density of a target is by weighing the sample directly on either an analytical or microbalance. A number of laboratories have constructed a special weighing room wherein temperature, humidity, and air currents are regulated so that reproducible and accurate weight determinations can be made. In the normal laboratory environment, convective currents, vibration, and myriad other perturbations make weight measurements difficult. However, for samples which contain ≥10 mg of material, weight measurements in a laboratory environment can be made to ≤0.5%. Frequently experimentors require samples, having a total weight of <10 mg, to be weight-determined to an accuracy of ≤0.5%; this normally creates only a small problem for samples >1 mg. However, for samples <1 mg or samples which require weighing in a glove-box environment, special precautions must be taken to achieve the desired accuracy. One method which has proven highly useful is accomplished by using a weighing scheme developed by the National Bureau of Standards.[3] In this scheme the sample(s) and standard weights, of approximately the same weight as the sample, are weighed in a particular sequence. At the end of the sequence, the sensitivity of the balance can be determined; normal zero-point drift of the balance does not effect the final weight determination. Sequence weighing is easily programmable to give an output of final weights for each sample together with uncertainties at a desired confidence level. This technique has been routinely used by IRML for mass determinations of vapor deposited films and reactor dosimeter samples.

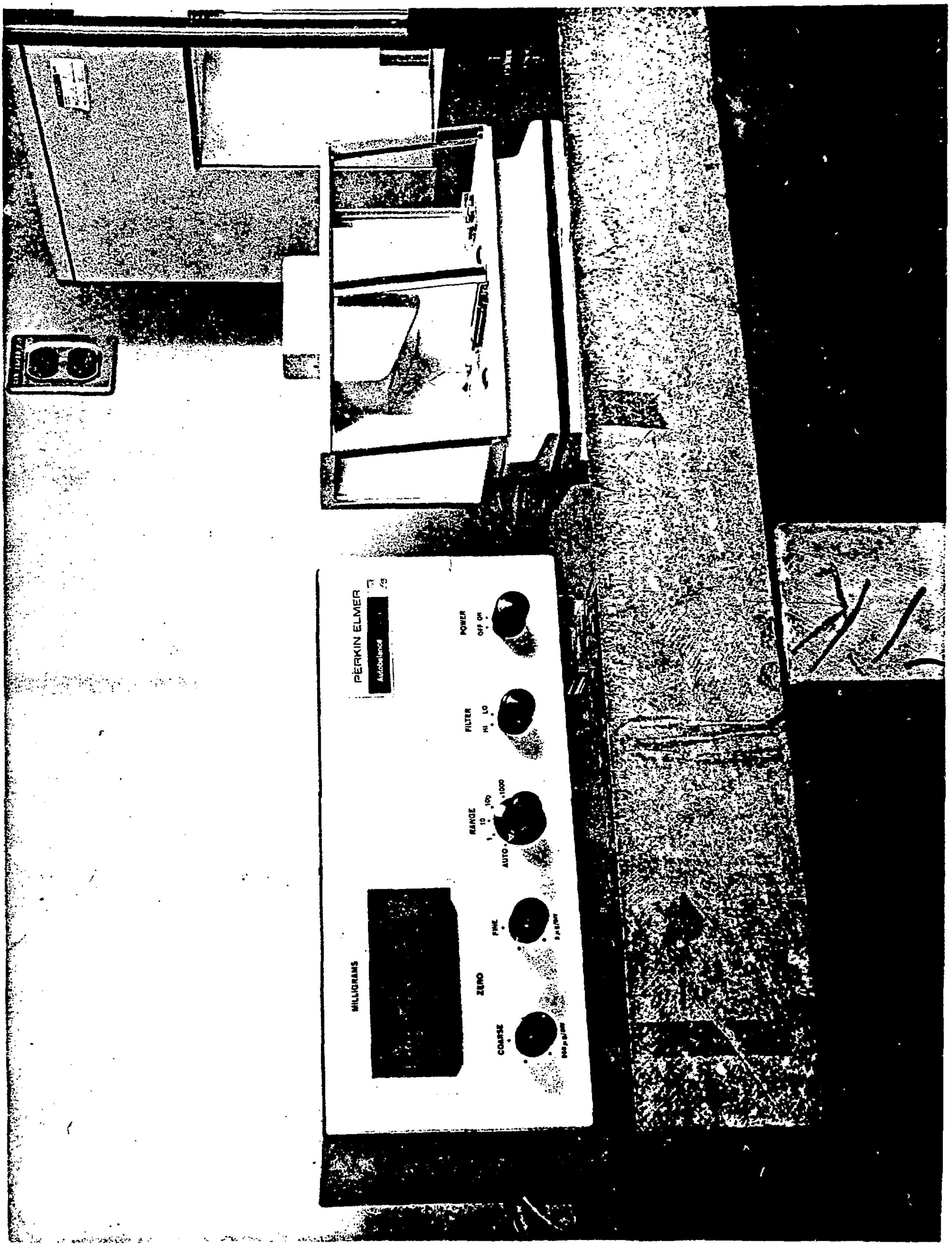
For most mass measurements a Mettler M5 microbalance is used which has a stated precision of 1 microgram. For very small samples a Perkin-Elmer electronic balance or a Mettler UM7 microbalance is used. The Perkin-Elmer electronic balance has a capacity of 100 mg and can read 1 mg to the nearest 0.1 microgram. The UM7 has a 2 mg capacity and a precision of ±0.1 micrograms. The Perkin-Elmer electronic balance and the Mettler

UM7 microbalance are shown in Figs. 5 and 6, respectively. A Mettler M5 microbalance for weighing chemically reactive materials has been installed in a glove box having an argon atmosphere, Fig. 7.

An example of reproducibility in weight determinations is provided in Fig. 8. More than 200 samples of boron and lithium crystals in the weight range 50-300 micrograms were weighed at Atomics International, Canoga Park, California, on a Perkin-Elmer electronic balance. The samples were sent to IRML and reweighed on the same type of balance using the NBS weighing scheme. Figure 8 shows results of this comparison. In almost every case the agreement was better than ±1%.

Many of the errors introduced by weighing samples in air can be eliminated by using a vacuum microbalance. To better define the amount of material deposited on a substrate, a vacuum microbalance was obtained from the Central Bureau of Nuclear Measurements (CBNM) at Mol, Belgium. In addition to weighing targets under vacuum, the balance can be used to calibrate quartz resonant crystals that can subsequently be used for <u>in situ</u> weight measurements during vapor deposition. At CBNM the balance is being used in the production of "standard" targets prepared using boron or uranium and having a layer thickness defined to ±0.3%.

The vacuum microbalance is shown in Fig. 9. A fused-silica balance beam is held to the balance frame by a central suspension composed of two 0.05-mm tungsten wires. One end of the beam is attached to an approximate 3 gram counterweight, a coil, and a mirror; all are fused to the beam. The main fulcrum point for the load position, located on the other end of the beam, is formed using a tungsten triangle arrangement followed by additional pivot points consisting of hooks and rings. Below the fulcrum point is a quartz carrier for two calibration weights and the load. The load, or sample to be measured, is suspended from the balance by a light weight metal, such as aluminum, or by a fused-silica fiber. Calibration rings and the load can alternately be suspended from the balance by stainless steel forks which are raised or lowered by a motor-drive mechanism. The fused-silica balance beam was made electrically conductive by spraying it with a silver paste and then baking at approximately 550°C. A solenoid-type arrangement was prepared by painting a helical trace on a silica cylinder; electric current can be supplied to this coil (located in the field of a permanent magnet)


PERKIN ELMER
Autobalance
POWER
OFF ON
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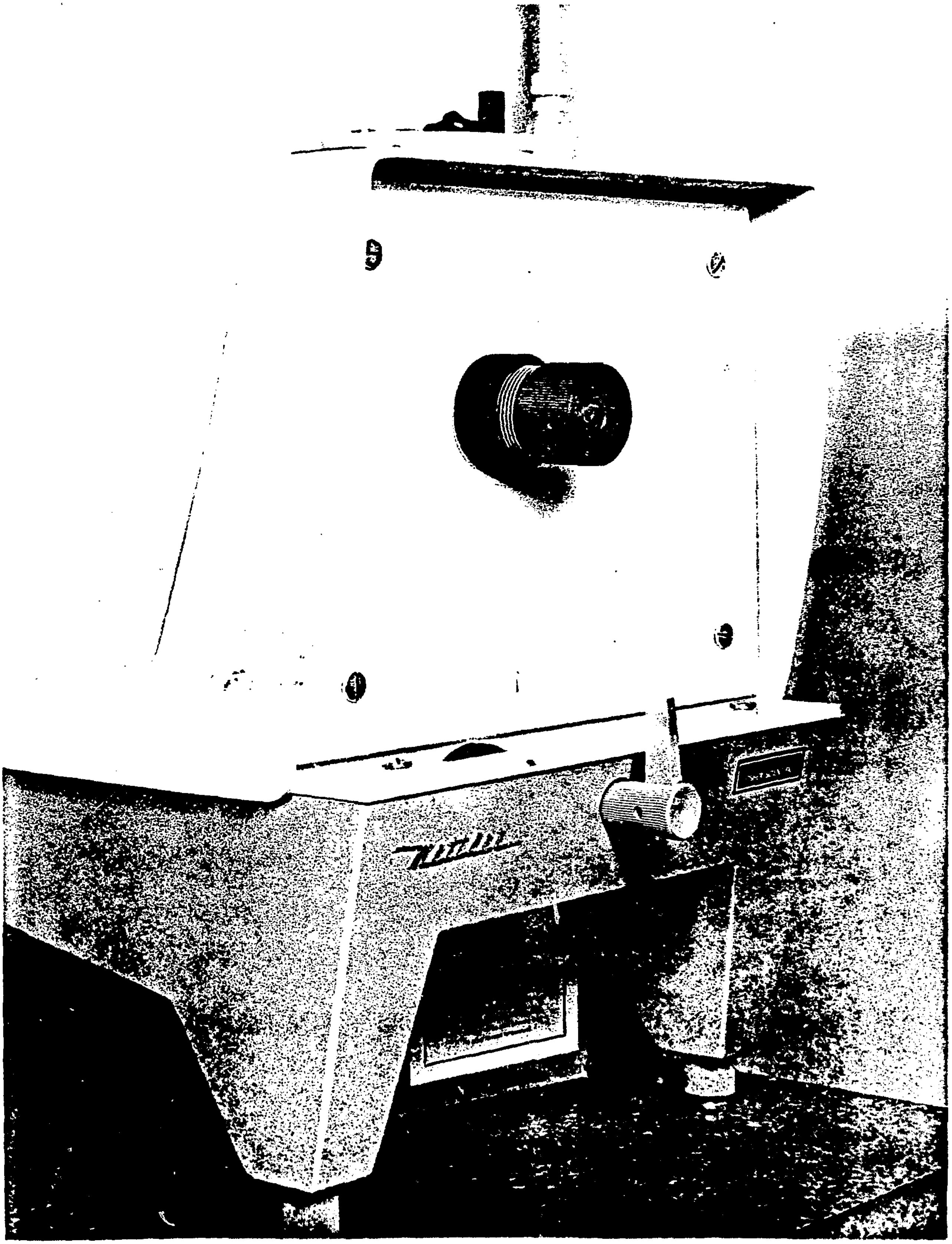
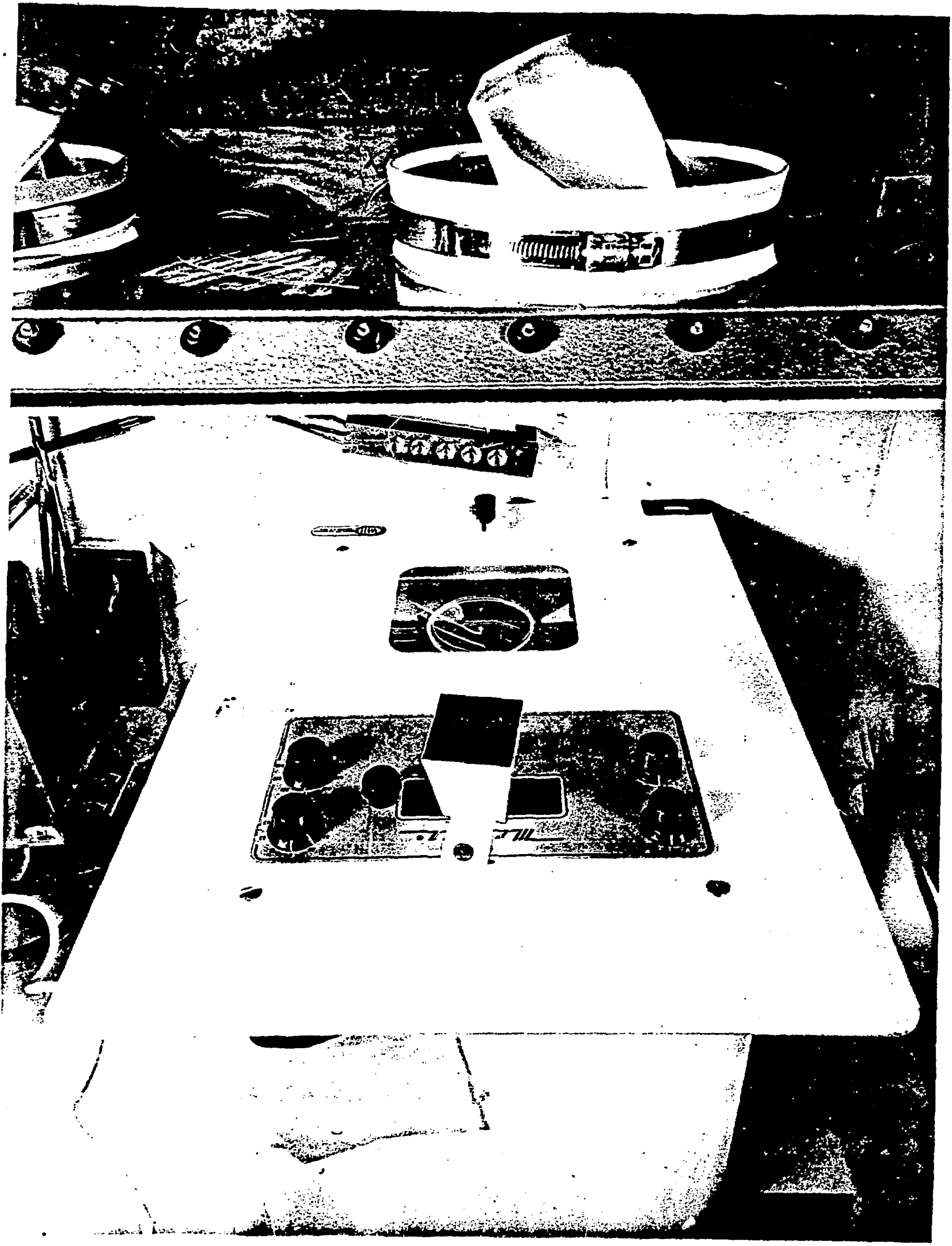
AUTO 1 10 100 1000
MILLIGRAMS
ZERO
FINE
COARSE





ORNL-DWG 75-13195

COMPARISON OF MASS VALUES FOR NATURAL BORON CRYSTALS

AI Crystal Number	Mass Values and Concentrations (μg)	
	AI (3σ)	ORNL (2σ)
DB101	253.8 ±0.5	253.1 ±1.0
DB102	243.5 ±0.4	243.8 ±1.0
DB103	239.7 ±1.5	240.0 ±1.0
DB104	246.6 ±1.5	245.3 ±1.3
DB105	222.4 ±0.3	222.3 ±1.3
DB107	168.9 ±0.2	168.8 ±1.3
DB108	225.0 ±0.2	226.1 ±0.9
DB109*	145.7 ±1.4	145.2 ±1.4
DB110	238.5 ±0.7	239.5 ±0.9
DB111	158.0 ±0.3	-
DB112	202.7 ±0.6	204.2 ±0.9
DB113	208.2 ±0.9	208.8 ±1.4
DB114	181.2 ±0.3	181.8 ±1.4
DB115	228.1 ±1.0	229.8 ±1.4
DB116	256.1 ±0.5	254.3 ±1.2
DB117*	131.3 ±0.3	130.7 ±0.2
DB118	210.9 ±0.7	212.9 ±1.2
DB119	110.8 ±0.9	111.2 ±1.2
DB120	238.7 ±0.2	241.4 ±0.7
DB121*	126.7 ±0.8	126.4 ±0.4
DB122	202.2 ±1.4	201.6 ±0.7
DB123	171.4 ±0.9	174.0 ±0.7
DB124	239.8 ±1.5	239.4 ±1.1
DB125	159.9 ±0.4	160.7 ±1.1

*Enclosed in 0.25-in. vanadium capsule.

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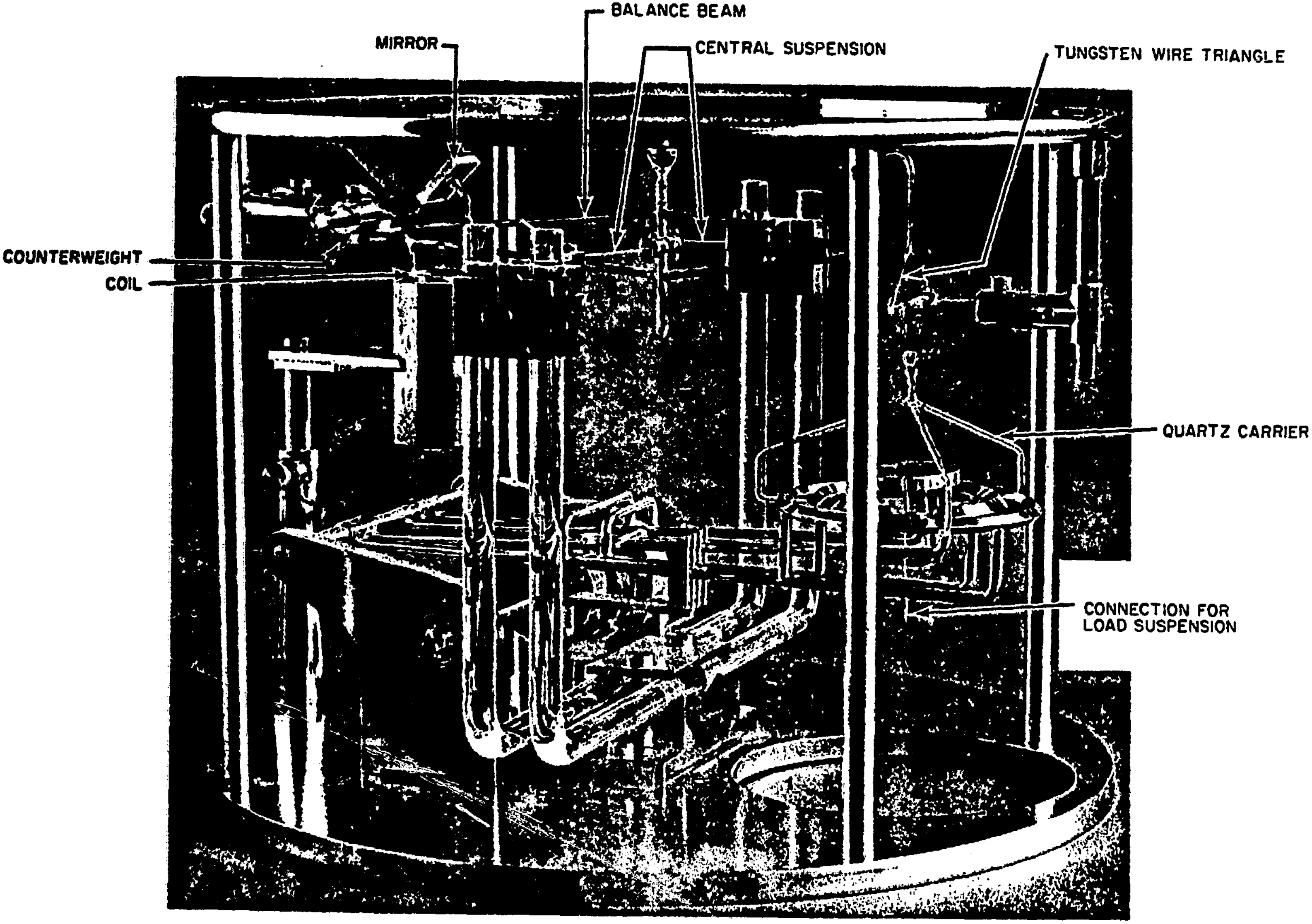
BALANCE BEAM
MIRROR
CENTRAL SUSPENSION
TUNGSTEN WIRE TRIANGLE
COUNTERWEIGHT
COIL
QUARTZ CARRIER
CONNECTION FOR LOAD SUSPENSION

through the tungsten suspension wires and through the balance beam, which has suitable interruptions in the conducting coating to prevent short circuiting. When the balance is not in equilibrium, current supplied to the coil which produces a compensating force, as with an electromagnet, and restores the balance to equilibrium. The amount of current required to restore equilibrium is directly proportional to the load on the balance. The balance is compatible with ultrahigh vacuum and can be baked to 400°C.

A complete weighing using the vacuum microbalance requires three weight determinations. Errors due to sensitivity shift and zero drift of the balance are eliminated by substitution weighing. Figure 10 shows the linearity of the balance which was observed by adding known weights as the load and recording the corresponding current required to restore the balance to equilibrium. Average current per microgram of load was found to be 0.7762 µA with a variation of ±0.4%. Figure 11 shows a schematic of an arrangement by which the balance was used to calibrate a quartz resonating crystal for later use in *in situ* secondary weight measurement.

Quartz Crystals

Quartz crystals are frequently used to determine both the areal density of a deposited layer and vapor deposition rate. A change in the resonant frequency or period of oscillation as material is condensed on the crystal is proportional to the crystal mass. The advantage of crystal monitors is that when once calibrated, they can be used to determine film thickness without having to expose the system to atmosphere. IRML uses crystal monitors to determine if the amount of material deposited is in the desired thickness range, but the ultimate measurement remains the direct mass determination of the target after it has been removed from the vacuum system. Figure 12 shows a water cooled crystal monitor used for monitoring titanium film thickness. Since the ultimate thickness would exceed the range of the crystal monitor, a rotating wheel with a small opening is used to reduce the amount of titanium impinging on the quartz crystal. It is necessary to water cool the crystal since it is heat-sensitive and is operating in an environment of >300°C.

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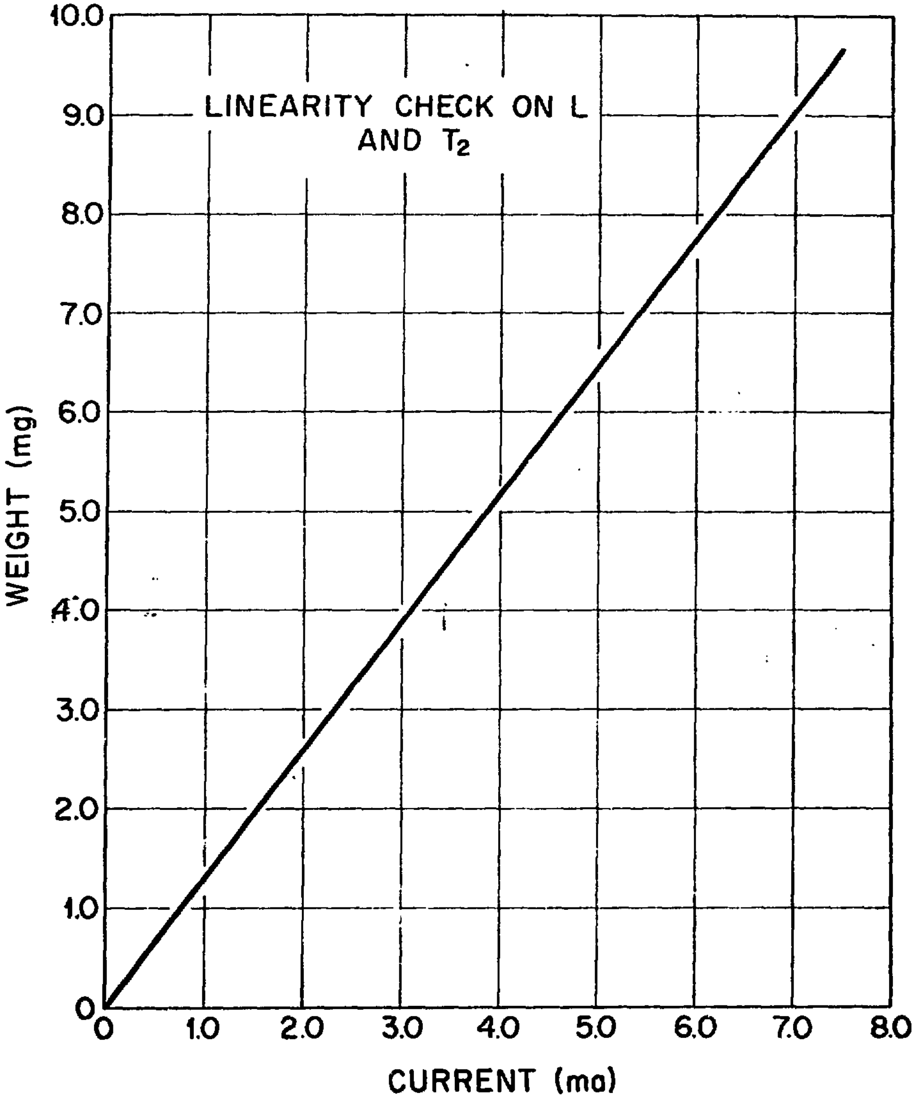
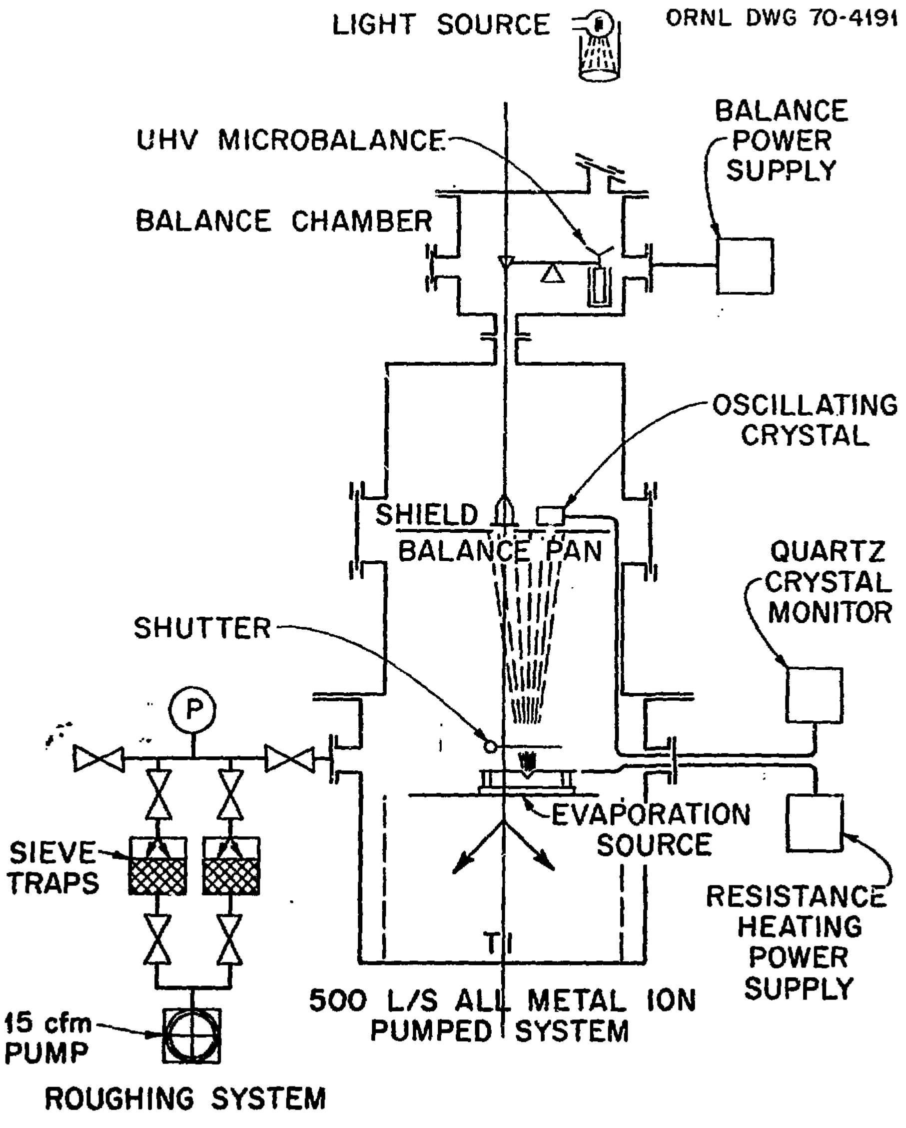
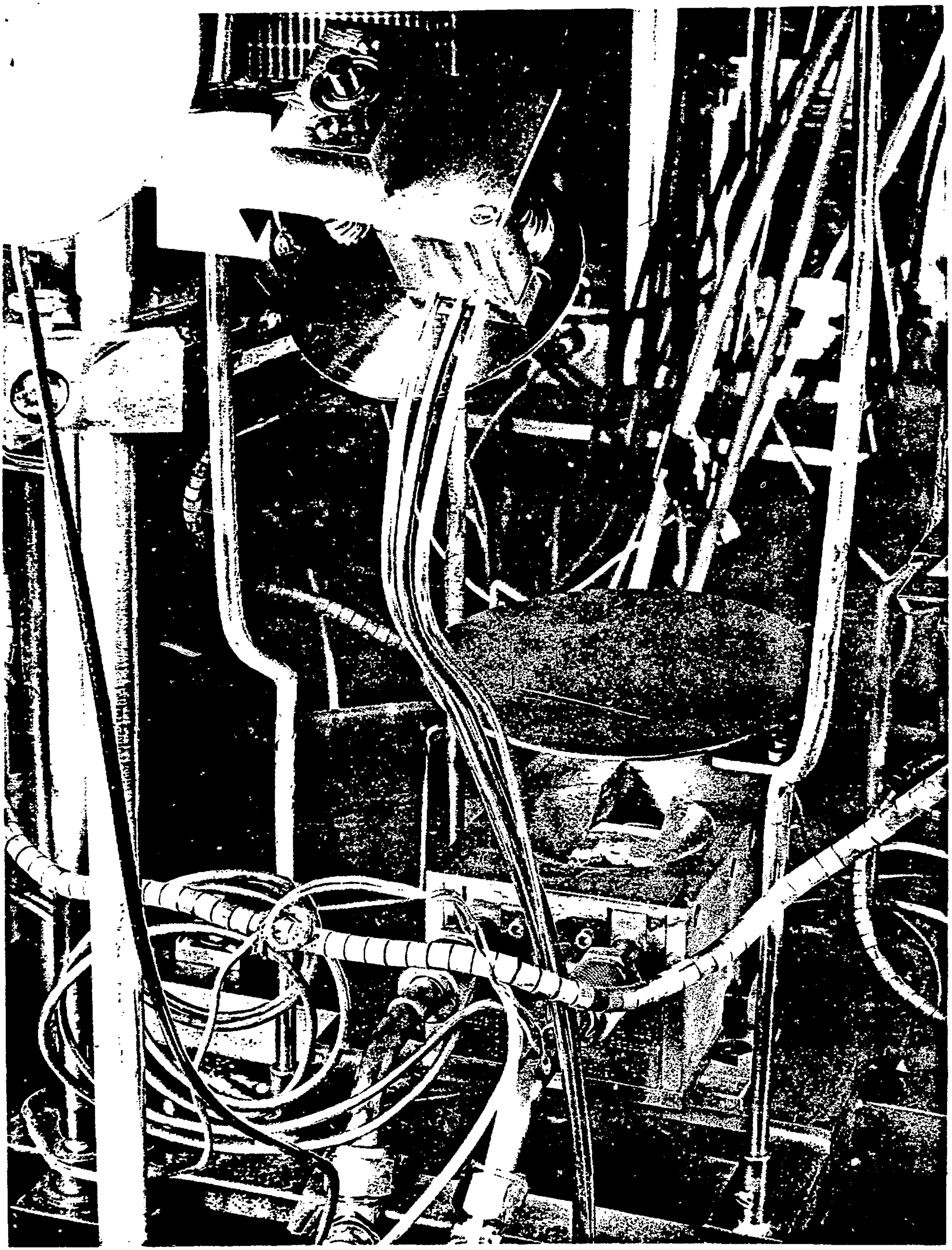



ORNL DWG 70-4191
LIGHT SOURCE
UHV MICROBALANCE
BALANCE POWER SUPPLY
BALANCE CHAMBER
OSCILLATING CRYSTAL
SHIELD
BALANCE PAN
QUARTZ CRYSTAL MONITOR
SHUTTER
P
EVAPORATION SOURCE
SIEVE TRAPS
RESISTANCE HEATING POWER SUPPLY
Ti
500 L/S ALL METAL ION PUMPED SYSTEM
15 cfm PUMP
ROUGHING SYSTEM



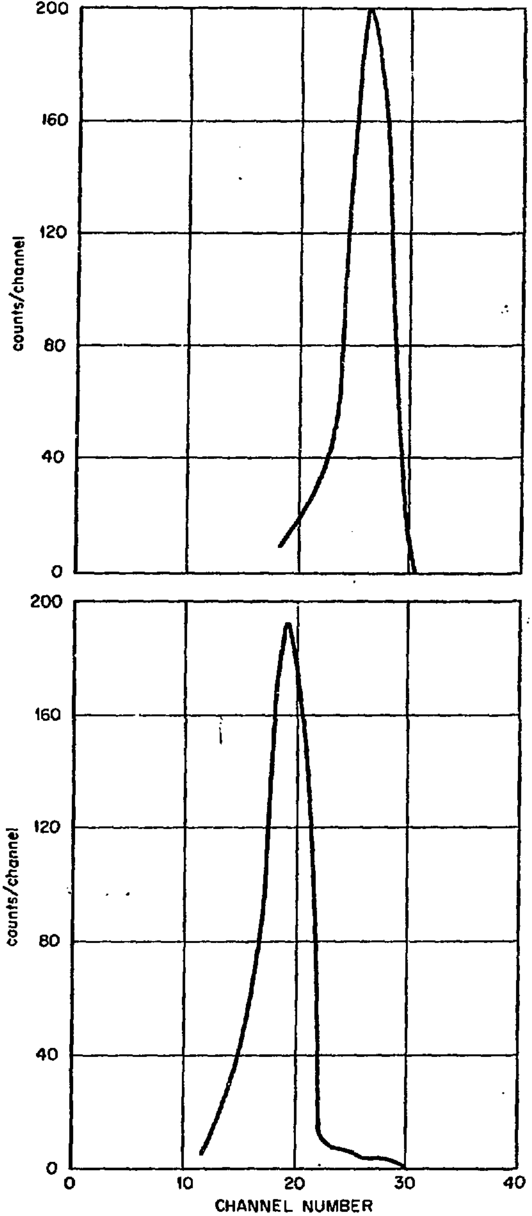
Change in Energy of Charged Particles

Frequently, areal density of self-supporting thin films cannot be determined directly by the above described techniques. The method used most by IRML for such samples is to measure the change in energy of charged particles as they traverse the target film.[10] This is a relatively simple technique and is readily adaptable to all materials whose stopping-power is known and whose thickness does not exceed the range of the incident charged particles. Stopping-power data for numerous incident particles and target materials can be found in the literature, but most of these data have an accuracy of $\geq \pm 10\%$.[11,12] IRML uses both alpha particles and fission fragments to measure target thicknesses which vary from a few μg/cm^2 to several mg/cm^2.

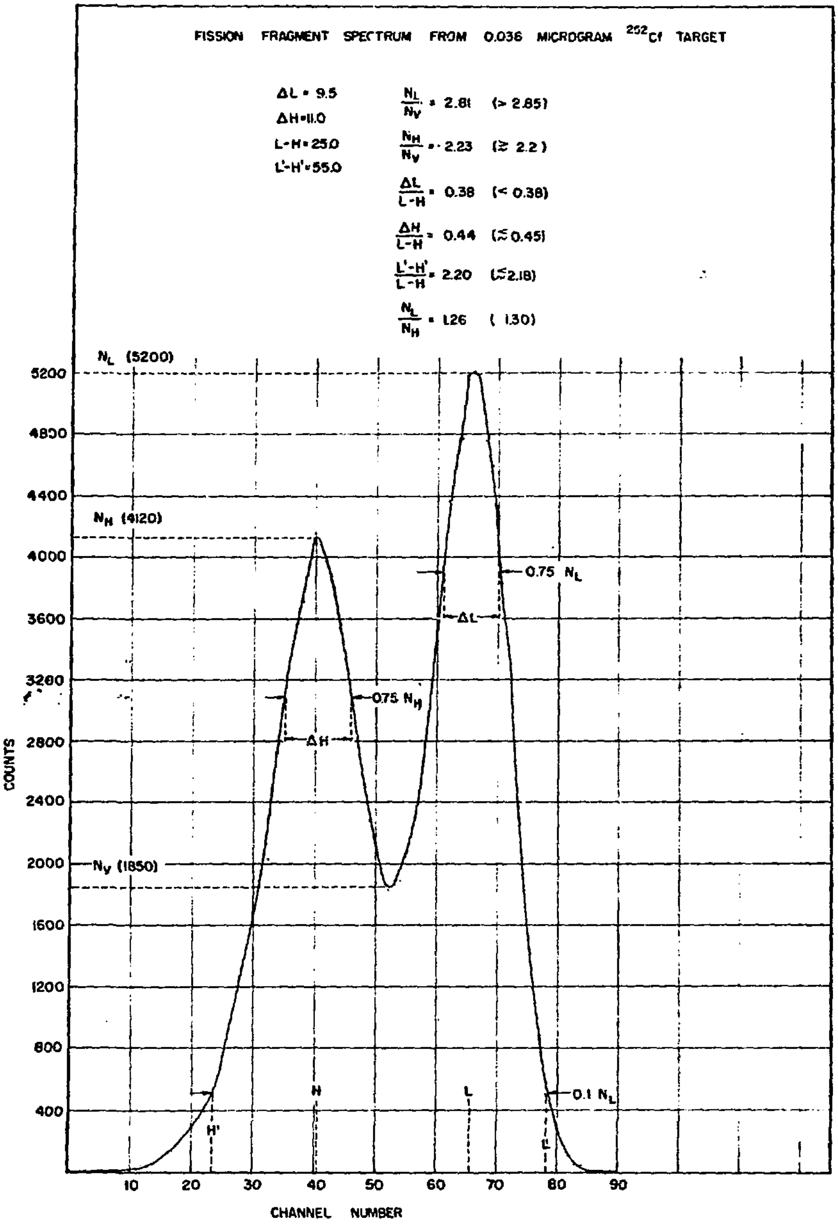
The change in energy of alpha particles as they pass through foils has been used by many investigators to determine target thickness and uniformity.[13-15] Alpha particles from a suitable source such as ^{241}Am are collimated and impinge on a small area of the planar target foil. The energy of the attenuated beam is determined by observing the spectrum shift from its original location obtained with no foil in place using a calibrated multichannel analyzer system. An example of this technique is shown in Fig. 13. By knowing the stopping power, usually expressed in units of MeV cm^2/g, the areal density can be calculated by dividing the value of stopping power into the amount of energy lost by the particle in traversing the film.

Fission fragments are also used to measure target thicknesses. Since these particles are quite massive compared to alpha particles, they experience a greater energy loss per unit target thickness and should be more suitable for measuring the thickness of very thin foils. A thin ^{252}Cf deposit, prepared by the self-transfer process, is used as the source of fission fragments. Figure 14 shows a typical spectrum from such a source. The spectrum parameters indicate a very clean source (very little material other than ^{252}Cf) and good detector performance. The fission fragments are detected with an ORTEC heavy ion silicon surface-barrier detector. The procedure used for thickness measurement using charged particles is described in Reference 15. Beta particles are also used to determine thicknesses of foils in the range of 40 to 500 mg/cm^2; this technique is described in detail in References 15 and 16.

ORNL-DWG 68-2432



ORNL-DWG 69-6315



Light Meter

A calibrated light densitometer is used for thin self-supporting foils, such as carbon, to determine areal density. This method provides a very rapid, semi-quantitative measurement of the foil thickness. Figure 15 shows the calibration curve for carbon foil thicknesses in the range of 5 to 50 μg/cm^2. From the figure, it is apparent that light intensity change is not a very useful technique for carbon films of thickness greater than 40 or 45 μg/cm^2.

Low Geometry Counting

Most often the technique used for determining the amount of radioisotope(s) contained in a target is that of alpha or gamma counting. For evaporated actinide targets, low geometry alpha counting is used to determine the activity present to $\leq \pm 1\%$. A schematic of the low geometry counting chamber is shown in Fig. 16. For highly radioactive samples, a similar but longer counting chamber is used in which the geometry factor can be reduced to 10^{-7} (the number of incident emissions reaching the detector as compared with the total emissions from the source).

A typical alpha spectrum from a $^{235}UO_2$ deposit is shown in Fig. 17. In this figure, the two large peaks between channel numbers 600 and 800 are the 4.77- and 4.72-MeV alpha particle peaks characteristic of ^{234}U emissions. The ^{234}U content of the material as shown in Table 2 is 0.029 atom percent. However, since the specific activity of ^{234}U is much greater than the specific activity of ^{235}U, the number of alpha counts from the ^{234}U activity approach the total number of alpha counts from the ^{235}U material. The remainder of the peaks in Fig. 17 are those characteristic of ^{235}U with small contributions from ^{236}U and ^{238}U. The energy calibration of the detector system is approximately 1.2 keV/channel. For most of the $^{235}UO_2$ deposits prepared, the ^{235}U content is determined initially by weighing. A sample is then placed in the low-geometry alpha counter and a spectrum similar to the one shown in Fig. 17 is obtained. By having an accurate mass analysis of the material, the ^{235}U content can be determined by integrating the number of counts under the ^{235}U peaks and subtracting out contributions from ^{236}U and ^{238}U emissions. The ^{235}U concentration is also determined from the mass analysis and the amount of ^{234}U present. In addition, gamma

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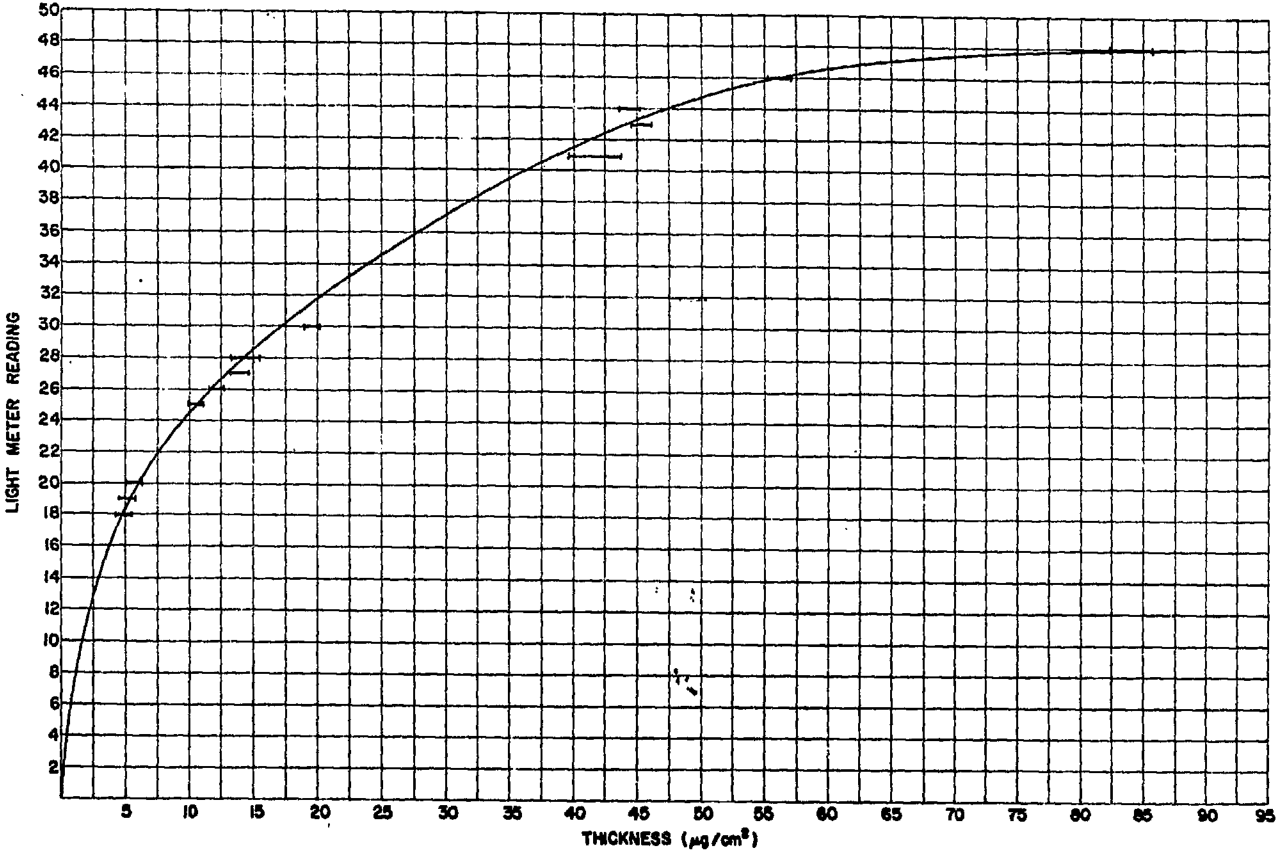
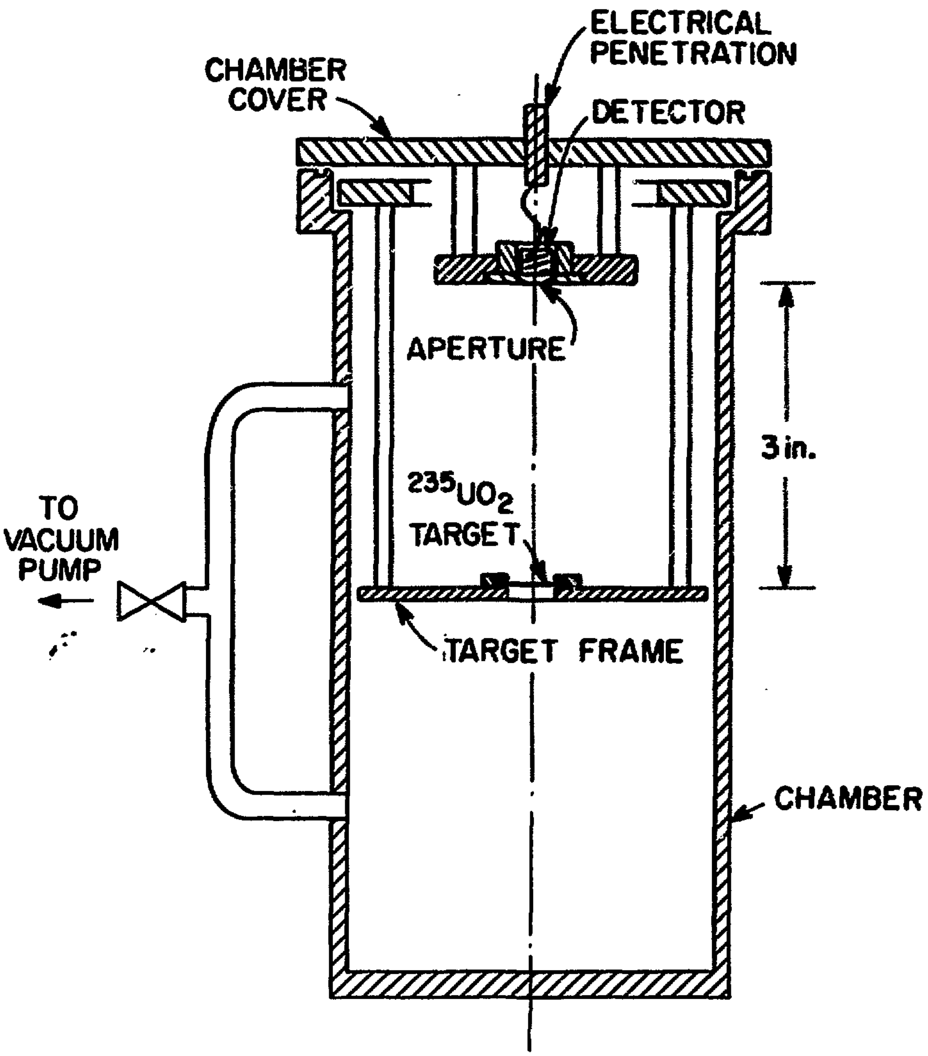
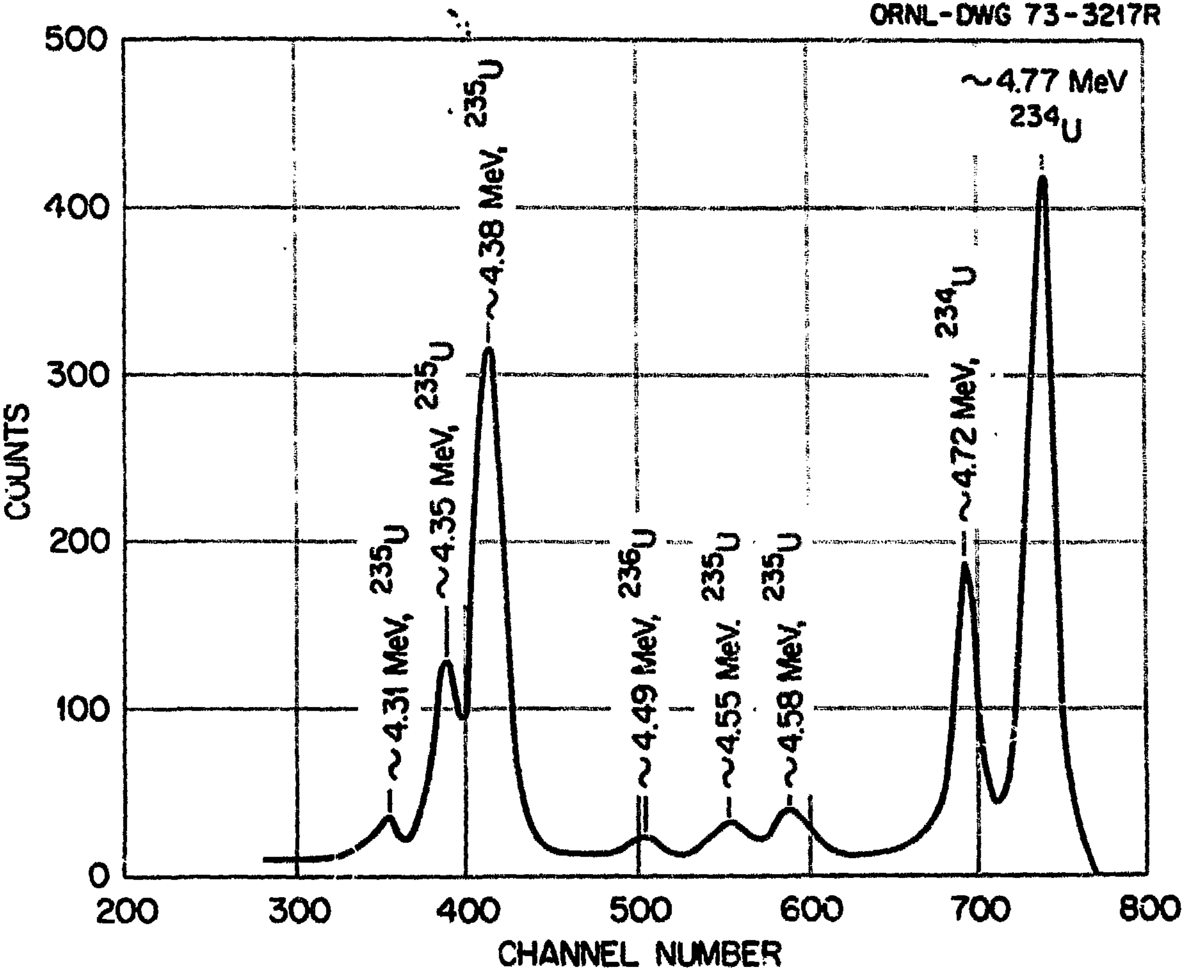


ORNL-DWG 72-7514



ORNL-DWG 73-3217R



counting is used to determine the ^{235}U concentration. A gamma spectrum from a $^{235}UO_2$ dosimeter is shown in Fig. 18. The 144 keV, 164 keV, and 185 keV, gamma peaks are used for ^{235}U concentration determinations.

Table 2. Mass Analysis ^{235}U Material

Isotope	Atom %
^{233}U	<0.0001
^{234}U	0.029
^{235}U	99.91
^{236}U	0.016
^{238}U	0.044

IV. TARGET CHARACTERIZATION BY UNIFORMITY MEASUREMENTS

The uniformity of thin foils can be determined by scanning the foil with a collimated beam of alpha particles. The energy loss of these particles at various locations can be determined and a target thickness topograph drawn. For targets having a thickness in the range of 5 to 500 mg/cm^2, beta particles can be used to determine the target thickness profile by the procedure described in References 15 and 16. Figure 19 illustrates a target thickness profile obtained by scanning a ^{58}Ni rolled foil with β-particles from a ^{204}Tl source. For alpha scanning of stable isotope targets, the ^{204}Tl source is replaced with an ^{241}Am source.

For radioactive sources the surface of the sources can be scanned using a small collimating aperture and a silicon solid state detector; Fig. 20 illustrates a profile obtained by scanning the surface of a $^{239}PuO_2$ deposit. A schematic representation of the system used for target thickness and uniformity measurements at IRML is shown in Fig. 21.

V. TARGET CHARACTERIZATION BY IMPURITY ANALYSES

Characterization of target materials thus far has included only target thickness and uniformity measurements. In addition to knowing how much material is in a target, it is equally important to know what species are present and in what concentration. At the present time this characterization is being done, for the most part, by destructive analyses. Analyses are carried out by the Analytical Chemistry Division at ORNL and include

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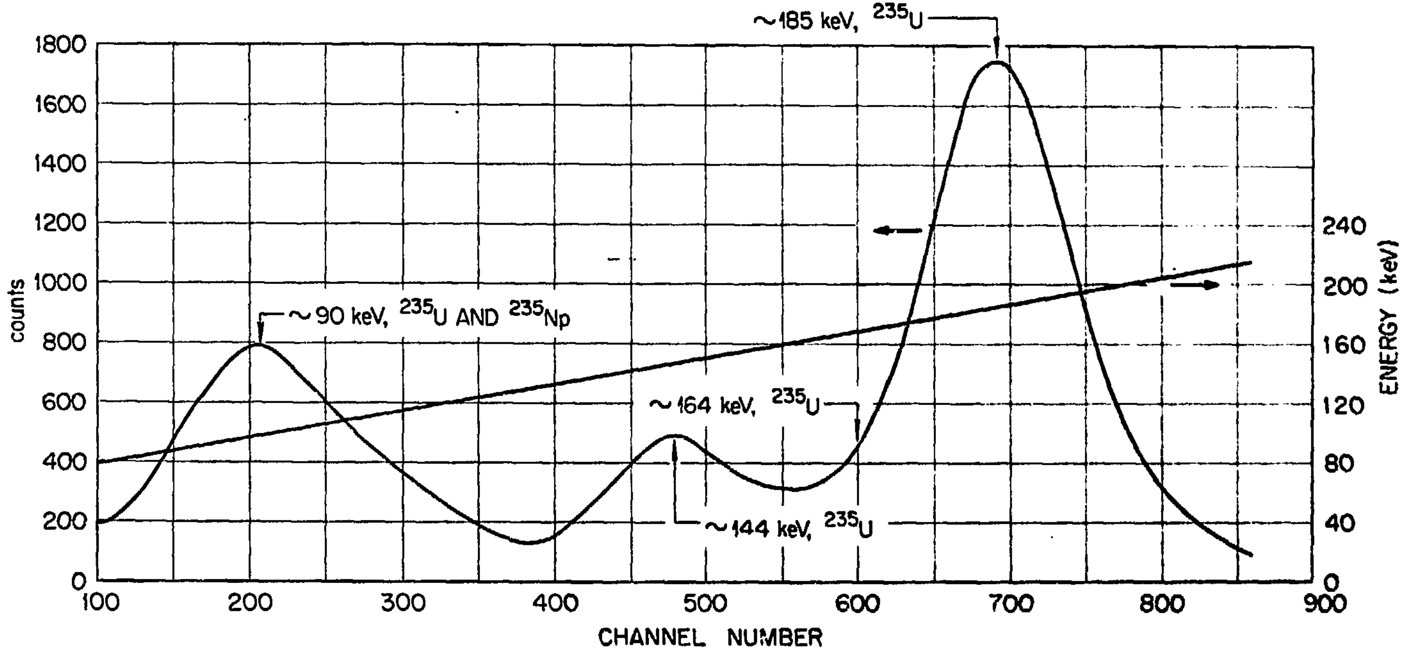
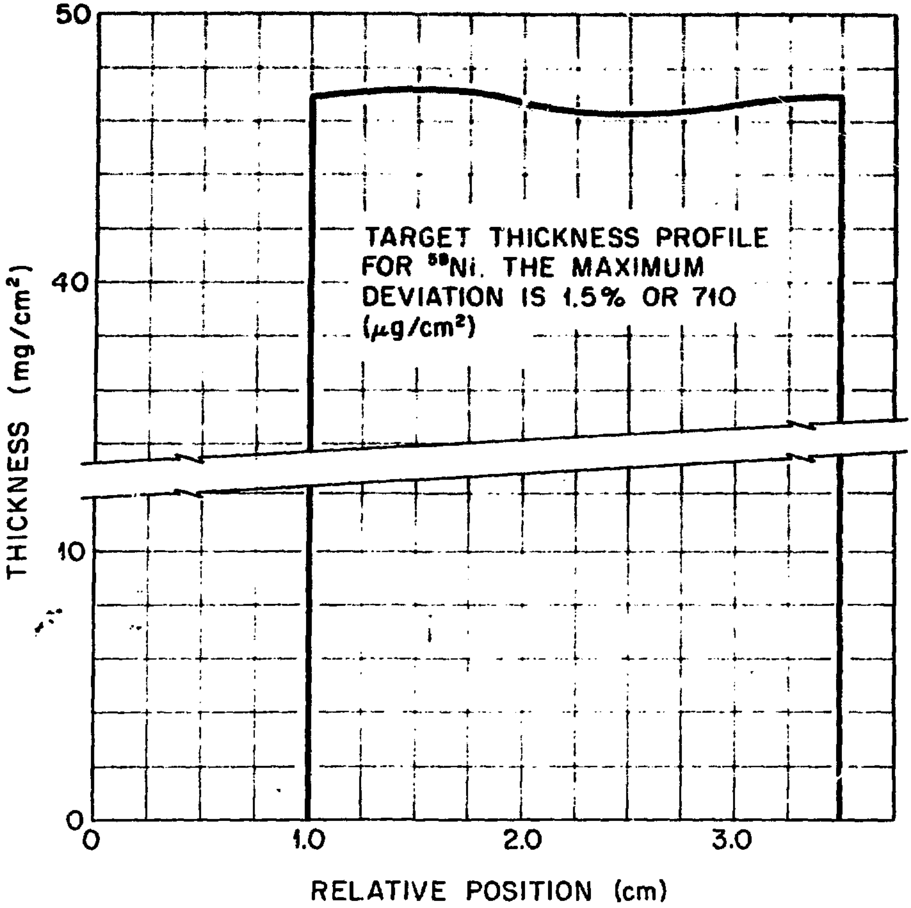


ORNL-DWG 70-5041



TARGET THICKNESS PROFILE FOR ^{58}Ni. THE MAXIMUM DEVIATION IS 1.5% OR 710 (μg/cm^2)

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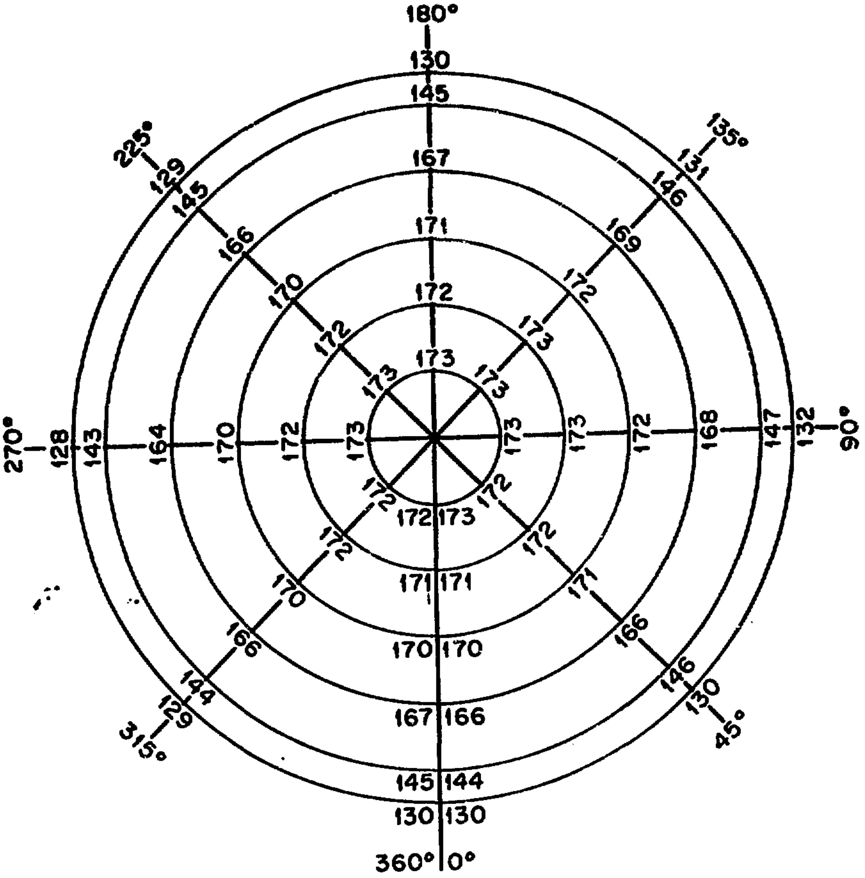

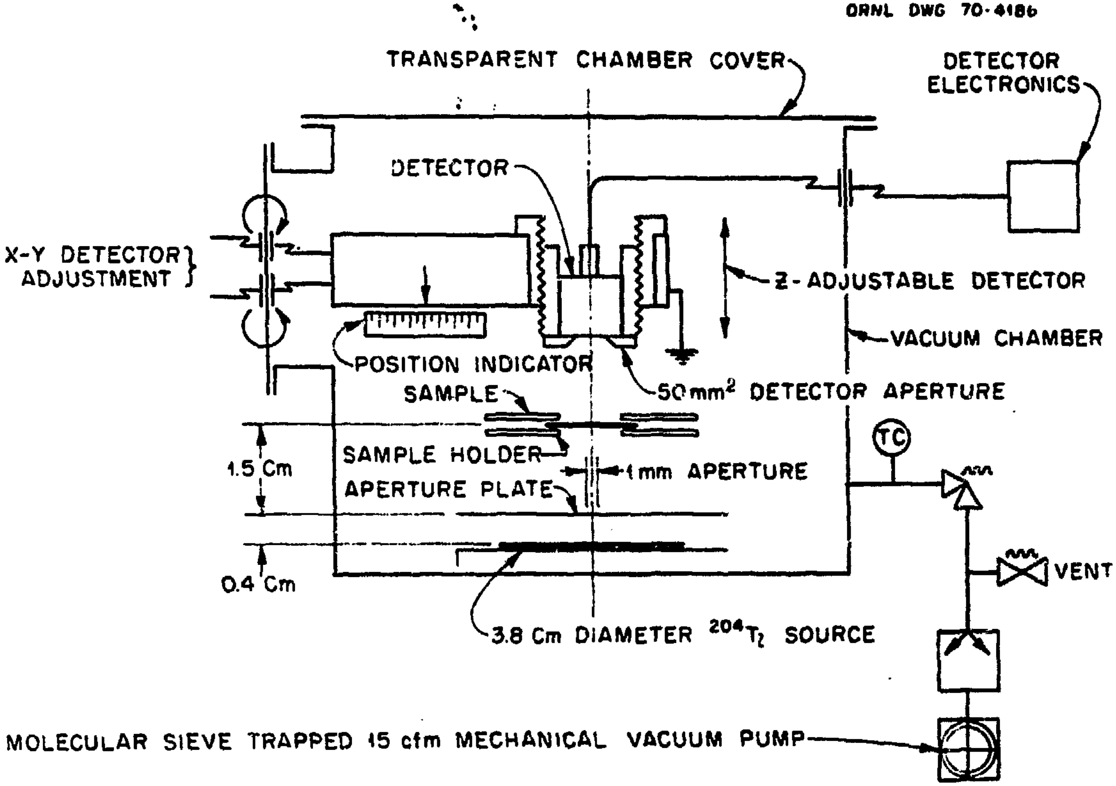
ORNL DWG 70-4186
TRANSPARENT CHAMBER COVER
DETECTOR ELECTRONICS
DETECTOR
X-Y DETECTOR ADJUSTMENT
Z-ADJUSTABLE DETECTOR
VACUUM CHAMBER
POSITION INDICATOR
SAMPLE
50 mm^2 DETECTOR APERTURE
TC
1.5 Cm
SAMPLE HOLDER
1 mm APERTURE
APERTURE PLATE
VENT
0.4 Cm
3.8 Cm DIAMETER ^{204}Tl SOURCE
MOLECULAR SIEVE TRAPPED 15 cfm MECHANICAL VACUUM PUMP

elemental, spark-source mass spectrographic, and mass spectrographic isotopic analyses. It is hoped that in the near future the technique of x-ray fluorescence will be used by IRML to nondestructively analyze completed target samples for impurities that may be detrimental to the desired experimental results.

SUMMARY

A brief description of some of the procedures used by IRML to prepare targets from most of the elements in the Periodic Chart have been described. Emphasis has been placed on the procedures used by IRML personnel to characterize the thickness and uniformity of the target samples.

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FIGURE LIST