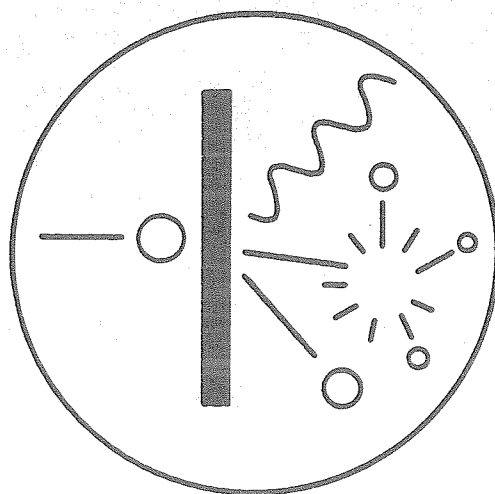


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NEWSLETTER



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

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

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Editors Note

Dear Colleagues,

Like it or loathe it, commercial activities are becoming a part of every target makers activities. Working on the theory "if you can't beat 'em, join 'em" the INTDS Newsletter will accept some advertisements for each edition (relevant ones only please - no used cars!). The main intention is to help laboratories publicize their activities without having to make technical contributions, but other items will be of interest to members: isotopes and equipment for sale, target stocks for sale, special target preparation techniques ...

REMINDER: 16th INTDS World Conference, Padova, September 21 - 25

Abstract deadline: June 30

Hotel reservations: July 15

Chris Ingelbrecht
EDITOR

Preparation of thin deuterated polyethylene targets of large size

P. Demaret

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Louvain-la-Neuve, Belgium

This contribution reports on an improvement of the technique of Bartle and Meyer¹⁾, concerning deuterated polyethylene targets of large size. To obtain a 250 $\mu\text{g}/\text{cm}^2$ thick target, 25 mg of $(\text{CD}_2)_n$ are added to 15 cc of Xylene. The solution is allowed to boil on a hot plate during 5 minutes at a temperature of 145° C. in a beaker covered with a watch glass. After the solution has cooled down slightly, it is poured onto a glass slide which is then covered by an inverted glass vessel.

This setup is placed on a hot plate kept at 70°C, perfectly horizontal and larger than the glass slide. It is very important that the glass slide is very clean, the last cleaning agent being a Kodak optical paper. As important is the fact that the glass slide is kept dust-free during the procedure.

The polymerization takes place slowly at 70°C, in a Xylene-saturated atmosphere. Care should be taken that the condensed Xylene does not fall back onto the glass slide. After about 15 minutes, evaporation of Xylene is completed and the glass vessel can be removed from the glass plate. However, at that time, the surface of the film on the glass slide is still white, indicating an incomplete polymerization. After subsequent warming of the plate up to 110°C, one obtains a transparent film which can be cut into the requested size and pulled off after immersing the glass slide in water. No parting agent is needed. Targets as large as 10x10 cm^2 , i.e. the dimensions of the glass slide, can be produced readily, the thickness inhomogeneity being in the range of 10 to 20 %.

1) C.M. Bartle and H.O. Meyer, Nucl. Instr. Meth. 112 (1973) 615.

Maximum Loading of Resistance-Heated Tungsten Helical Coils with Aluminum *

W. Lozowski and J. Hudson

Indiana University Cyclotron Facility, Bloomington, IN 47408

After years of evaporating aluminum from tungsten helical coils (R.D. Mathis Co., cat. no. F11), we have happened upon a way to minimize the incidence of Al falling from the coil turns during the melting/wetting step. The inspiration to do so was the result of a commitment to add an additional $30\text{-}\mu\text{g}/\text{cm}^2$ deposit of aluminum to large pieces (approx 15 cm x 53 cm) of $1.5\text{-}\mu\text{m}$ thick aluminized Mylar. The area and thinness of the Mylar required a large source-to-substrate distance (40 cm). Consequently, more than 600 mg of aluminum had to be evaporated into an angle of 4π , with some additional aluminum required because of W/Al alloy formation. With the typical amount of Al falling from the source, the desired 111-nm thick deposit of evaporated Al required cycling the vacuum evaporator to air three times to recharge the tungsten source with aluminum.

Our usual procedure of loading aluminum onto a coiled strand of four 0.76-mm diam W wires was the well-worn one: pinching Al wire staples to the vertically lowest positions on the helical coil. We have found that staples load the turns of the coil more evenly than a length of aluminum wire threaded through the helix. Staples produce a more laterally extended evaporation source, which is desirable for evaporation/condensation onto large substrate surfaces. The Al wire used to form the staples was 2.1-mm diam common electrical wire (99.45% Al). Following a vigorous rub with Kimwipe tissues and acetone, the end of the wire was bent over the W coil, pinched tight onto it, and cut to a length of approx 1.8 cm. This process was repeated for five coils, resulting in about 170 mg per coil and roughly 850 mg total aluminum potentially available on the source.

The improved procedure involves cleaning the wire in the same way, flattening it to a thickness of 1.8 mm (we use a metal rolling mill and a stainless steel pack), and then abrading the surface with 1500 grit ($6\text{-}8\ \mu\text{m}$) sandpaper (3M Co. 414Q Imperial Wetordry Color Sanding Paper A Wt.). This paper, in 1000, 1200, 1500, and 2000 grit, is used by automobile body repair and painting shops; local automotive paint supply stores may have it. After the flattened wire is rubbed back-and-forth for about 30 s between a fold in the paper, the surface of the Al has a dark grey color. Immediately thereafter, the dust is removed (using a Kimwipe), the W helical coil is loaded with staples which are pinched tight onto the turns, and the vacuum chamber is evacuated to approx $66\ \mu\text{Pa}$ ($0.5\ \mu\text{Torr}$). The flattened wire staples we have used are about 2 cm in length, resulting in a total charge of Al which is nearly 850 mg, as above. Because the staples rarely fall from the coil, a thickness of $30\ \mu\text{g}/\text{cm}^2$ at 40-cm distance can be accomplished in one cycle.

With regard to possible contamination of the aluminum from the sand paper, at a magnification of 1000:1 no visible evidence could be found of sand particles on the surface of the flattened, abraded wire.

* Work supported by the National Science Foundation grant NSF PHY 90-15957 and Indiana University

TARGET LAB TECHNICAL STATUS *

Prepared for the IUCF Scientific and Technical Report, May 1990-April 1991

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Split-beam operation of the facility continued to keep the number of targets solicited quite large. Production of more-or-less typical targets and diagnostic foils for the accelerators required 32 FTE and 18 professional man-weeks. The balance of the lab effort was applied to the target development projects summarized below.

- Micro-ribbon development for the Cooler was pursued in several ways. One focus was a quest to optimize the surface texture of the substrates used in the vacuum evaporation/condensation process. We were rewarded with carbon ribbons which present a factor of three fewer atoms to the Cooler beam than any others previously mounted and handled successfully at IUCF. The ribbons, typically 23.5-nm thick ($4.2 \mu\text{g}/\text{cm}^2$) x 6.6- μm wide x 33-mm long, have improved regularity in the width, throughout the length. As a result of the elimination of narrower (weaker) areas along the ribbons and other factors related to the surface structure of the substrate, they exhibit increased resilience and strength. Similarly textured larger glass surfaces (50 mm x 75 mm), were used to produce 32-mm wide open-edged stripper foils of $4 \mu\text{g}/\text{cm}^2$ which have less tendency to curl at the open edge.

- Measuring and improving the electrical conductivity of the carbon ribbons were tasks undertaken with P. Heimberg, B. v. Przewoski, and H.O. Meyer. It was established that coating the ribbons with a thin deposit of aluminum (by vacuum evaporation/condensation) was undesirable because it produced ribbons with a value of electrical resistance which increased over time. However, a technique of annealing mounted ribbons at 1273 K for 10 min in an oven (assembled within the high-vacuum evaporator) increased their electrical conductivity by a factor of 10^3 , with reproducibility. In-beam charging of the carbon ribbons was finally attributed to insufficient electrical conductivity of the graphite/cyanoacrylate ester mix used to glue the ends of the ribbons on the target frames. A silver paint (colloidal silver in water) is now used for this purpose.

- Our first attempts to produce vacuum-evaporated copper ribbons achieved ribbons which were $19 \mu\text{g}/\text{cm}^2$ x 25 μm wide x 60 mm long. Glass slide pieces dip coated with a saturated solution of polyvinyl alcohol (water soluble PVA) functioned respectively as the substrate and release agent. Much thinner copper ribbons are likely to be realized with additional trials.

- The accuracy of the lab quartz crystal monitor (Maxtek TM-100) for 7-11 $\mu\text{g}/\text{cm}^2$ deposits of carbon was verified through several vacuum evaporations of thin carbon layers onto monitor foils. For each evaporation, two foils were positioned in the space normally occupied by the ribbon-forming wire grill. The head of the crystal monitor was in the customary position: 16 cm from the e-gun. An ultra-microbalance was used to register the weight changes of the monitor foils.

- The 6-mm thick x 70-mm high x 50-mm wide crystal mosaic target of yttrium

ethyl sulfate (Y E S) was completed in August. It fell short, by about 6%, of the polarization achieved with the target made several years earlier by Lynn Knutson. Analyses (using a direct insertion probe into a mass spectrometer and by energy dispersive x-ray) of samples from both sets of crystals were not adequately sensitive to determine if our method of crystal growth failed to produce the desired 0.01 at% Yb/Y (46 μg Yb/gm Y E S) in the crystals. Additional analysis will be done by a more sensitive method.

- A precision-cut wire saw was designed and built to allow the Y E S crystals to be cut close to the final dimensions required. The saw has a kerf loss of only 254 μm and features an auto-reversing mechanism which prevents the wire splice from traveling through the material being cut. The crystals were sawn without lubrication of the 203- μm diam diamond-impregnated wire (Laser Technology Inc., North Hollywood, CA) and with minimal generation of heat, known to cause the heat-sensitive crystals to crack.

- An H^+RSO^- (strongly acid cation exchanger) type ion exchange resin was used to convert Na^{37}Cl starting material to H^{37}Cl , which was then converted to $^{40}\text{Ca}^{37}\text{Cl}_2$ by direct combination with $^{40}\text{CaCO}_3$. The overall efficiency of the process was 85%. Pressed powder targets of 30 and 120 mg/cm^2 were made with the final product. Because the (p,n) experiment for which they were made was sensitive to sodium contamination at the 1% level, care was taken with the preparation and use of the ion exchange column. An energy dispersive x-ray analysis detected no sodium peak in the converted target material. The fact that the analysis was sufficiently sensitive at the 1% Na level was confirmed with doped samples of reagent grade CaCl_2 .

- A PC-based DA&C system with software packages was purchased for the lab. It will allow much better monitoring and control of short-time processes such as vacuum evaporation of micro-ribbons as well as longer term ones e.g., annealing and curing processes in the lab ovens and temperature monitoring of the controlled temperature bath during months of crystal growing. A 386SX/20 Mhz PC was also purchased for the lab to support the new system and allow background operation.

PREPARATION OF THICK CADMIUM TARGETS

H.J. Maier, H.U. Friebel, D. Frischke and R. Grossmann
Sektion Physik, Universität München, W-8046 Garching

The preparation of high purity cadmium targets in the thickness range between $10 \mu\text{g cm}^{-2}$ and several mg cm^{-2} by high vacuum evaporation-condensation has been described recently [1]. Here we report an alternative method for targets $> 1 \text{ mg cm}^{-2}$, which shows a higher degree of utilization of the costly isotopic material. Starting up with the oxide, an electrolytic reduction is first performed, followed by pelletizing of the resulting metal flakes and subsequent formation of a bead in an induction melter. Finally, foils of the required thickness are obtained by pack-rolling.

For the electrolytic extraction of the Cd metal, typically 30-50 mg CdO are dissolved in 2 cm^3 of $4\text{n H}_2\text{SO}_4$ in a platinum beaker. Heating and ultrasonic treatment are of advantage to speed up complete solution. The platinum beaker is operated as the anode of the electrolytic cell. A 2 mm diameter platinum wire is used as cathode. Adjusting the current to a high value of 150 mA cm^{-2} results in a spongy Cd deposit, which can easily be scraped off from the cathode. Finally, the metal is thoroughly washed in distilled water, rinsed with pure ethanole, dried in a porcelain crucible under an IR lamp and pressed to a pellet of 5 mm diameter. The yield of this procedure is confidently higher than 90 %.

The high volatility of Cd requires a special melting technique to form a bead suitable for rolling. Induction heating in a hydrogen atmosphere was found to give excellent results. The Cd pellet is placed in a small pyrex tube of 15 mm diameter, which is surrounded by a 2 turn induction coil with a clearance of 25 mm. The pyrex tube is evacuated to a pressure of 10^{-4} Pa with a turbomolecular pump and backfilled to $1.2 \cdot 10^7 \text{ Pa}$ with hydrogen of 99.99 % purity. Heating is performed with a 500 kHz high frequency generator operated at a power of 2 kW. Within 1-2 minutes melting comes to a completion. The material losses of this step are about 5 %.

Application of standard pack rolling techniques allows the preparation of target foils down to 1 mg cm^{-2} thickness and a size of $1\text{-}3 \text{ cm}^2$. In the beginning, small reduction steps of 3 % at the utmost prevent sticking of the foil to the stainless steel pack. In the later stages, a drop of ethanole is added to the pack to immerse the foil. Then rolling is continued cautiously. After 3 or 4 passes, the thin ethanole film allows removal of the foil from the pack without breaking, using a scalpel and tweezers as usual. This procedure is repeated until the required thickness is reached.

[1] H.J. Maier, H.U. Friebe1 D. Frischke and R. Grossmann, Nucl. Instr. and Meth. A 282 (1989) 128

PROGRESS REPORT OF THE TARGET LABORATORY FROM THE PELLETRON ACCELERATOR OF THE UNIVERSITY OF SÃO PAULO

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A chemical process to convert KCl into KI is being reproduced, as low Z contaminants are not tolerated in K targets. The KI was obtained by ionic exchange using special anionic resin (Amberlite IRA 400).

The KCl passed through a column of 20 cm height (~ 30 g resin), then it was treated with NaOH (2N). After that, the analitic qualitative teste of Cl ions was performed. The column was washed until pH 6 was obtained. Then a very slow flux of HI solution was permitted to afford the maximum exchange. The column was then washed until neutral pH was reached. Finally the solution of KCl was let through. The obtained solution supposed to be of KI was then dried. The vacuum evaporation of KI was then performed by electronic bombardment with Ta crucible with a lid. Only 15 to 18w are enough. Targets of $\sim 80 \mu\text{g}/\text{cm}^2$ were obtained but were not tested yet.

The following targets were made under request of the nuclear physicists users of the Pelletron accelerator facilities.

Z	Element	Thickness $\mu\text{g}/\text{cm}^2$	Backing	Method	Chemical Form	Crucible (e.b.) Boat (joule)	Power (e.b.) Current (joule)	Parting Agent
6	C	15		Arc		C	400 W	-
8	O	50 to 200	Al	e.b.	Al_2O_3	Ta	100 W	-
8	O	100 to 500	Al	e.b.	SiO_2	Ta	100 W	-
13	Al	500 to 800	-	joule	Metal	W	2,5 to 3,0 A	RBS
14	Si	70 to 180	Al	e.b.	SiO_2+Si	Ta	80 W	-
19	K	10	C	e.b.	KI	Ta	30 W	-
19	K	10 to 20	C	e.b.	KCl	Ta	20 W	-
19	K	600 to 700	C	e.b.	KCl	Ta	20 W	-
19	K	20	C	e.b.	K_2CO_3	Ta	20 to 40 W	-
19	K	80	C	e.b.	KI	Ta	20 W	-
	Mn+Al+Cu	20 to 30	Mylar	joule		W	50 A	-
26	Fe	100 to 250	Mylar	joule		W	3,0 to 3,5 A	-
27	Co	40	C	e.b.		C	50 to 80 W	-
29	Cu	1 to 2 mg/cm^2		joule		W	4 A	RBS
29	Cu	20 to 30	Mylar	e.b.	CuS	Mo	50 A	-
42	Mo	20	C	e.b.		W	100 to 120 W	RBS
42	Mo	30	C	e.b.		W	100 to 120 W	-
47	Ag	1 mg/cm^2		joule		W	2,0 A	RBS
47	Ag	20 to 30	Mylar	joule		W	2,0 A	-
51	$^{121}, ^{123}\text{Sb}$	500	Au	e.b.		Ta	20 to 30 W	-
50	^{124}Sn	500	Au	e.b.		Ta	70 to 80 W	-
79	^{197}Au	300 to 1,5 mg/cm^2	-	joule	Metal	W	15 to 20 W	RBS e CoI
82	^{208}Pb	13 mg/cm^2	Ni+Cu	joule		Ta	2,0 A	-
82	^{208}Pb	50 to 140	C	joule		Ta	2,0 A	-
82	^{208}Pb	5 to 30	C	joule		W	2,0 A	-

Some multiple targets were also made:

1) Pb + Au + Pd + Ta + Pb + C

A special target was fabricated by flash evaporation of various elements, on a carbon backing of $14 \mu\text{g}/\text{cm}^2$, in successive layers as follows: $2 \mu\text{g}/\text{cm}^2$ of Au, $2 \mu\text{g}/\text{cm}^2$ of Pd, $2 \mu\text{g}/\text{cm}^2$ of Ta, $5 \mu\text{g}/\text{cm}^2$ of Pb and finally $15 \mu\text{g}/\text{cm}^2$ of C.

2) CsCl + C on Al

From $500 \mu\text{g}/\text{cm}^2$ to $1 \text{ mg}/\text{cm}^2$ layer of CsCl was evaporated by electronic bombardment, 10 to 20 w, with Ta crucible on $0,5$ to $1 \text{ mg}/\text{cm}^2$ backing of Al.

3) SiO_2 + Al_2O_3

$50 \mu\text{g}/\text{cm}^2$ layer of Al_2O_3 and $480 \mu\text{g}/\text{cm}^2$ of SiO_2 were evaporated on an aluminum backing of $100 \mu\text{g}/\text{cm}^2$ thickness. Electronic bombardment was used. The oxide were filled in Ta crucible.

Floating and Mounting Suggestions - Carbon Foils

John O. Stoner, Jr.
ACF - Metals
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Tucson AZ 85719
Tel. 602-325-9557
Fax: 602-325-9493

Carbon foils are typically shipped either on glass substrates or individually packaged between paper sheets in transparent plastic boxes. Handle the glass substrates by their ends. Foils between paper sheets can be transferred on paper sheets or, with care, using tweezers or forceps. Foils can be cut to size before use; the best method of cutting a foil depends upon its surface density.

Mounting individually packaged foils (surface density > 200 micrograms per square centimeter):

A foil that is individually packaged in a plastic box is ready to be used. [Usually only foils having surface densities greater than 200 micrograms per square centimeter are so packaged.] It has been annealed to reduce its internal stresses so that it shows only slight tendency to curl. Its surface density is marked on the label on the outside of the box. Such a foil can be cut to size by laying it on a glass plate and laying another glass plate on top of the foil with one edge of the top plate used as a straightedge along the foil where you want to cut it. Using a clean single-edge razor blade, make several gentle cuts along that line until the foil is cut through. A single through cut, or a dull blade, may cause the foil to splinter. Cut pieces can be handled gently with tweezers, and glued or clamped to frames.

Removing thick foils (surface density 100-200 micrograms per square centimeter) from their substrates:

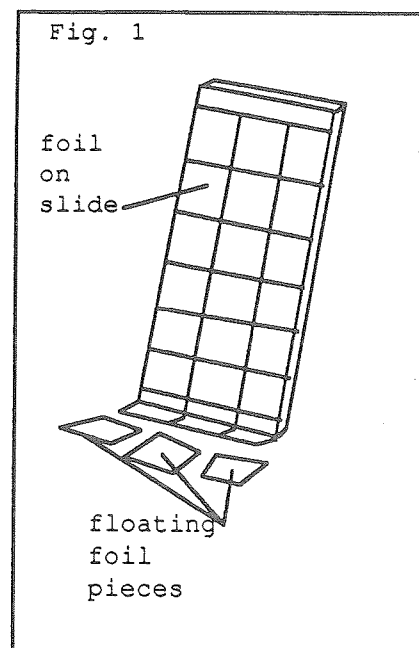
Foils that are on their substrates are handled differently depending upon whether their surface densities are above or below 100 micrograms per square centimeter. If above 100 micrograms per square centimeter, a foil may need to be baked before use, in order to reduce its internal stresses. In that case, the foil will curl into small-diameter tubes having no known use if you float it off without treatment. Write down the number on the tag on the back of the slide; this is the surface density in units of micrograms per square centimeter. If you wish to cut the foil before baking it, do so as described in the above paragraph. Then lay the slide horizontally, gently, foil-side down on a clean glass plate and place the assembly in a vacuum oven or in an oven that is flushed with dry nitrogen. Raise the temperature to 260-300 degrees Celsius and let it cool. The total heating and cooling cycle should be no more than an hour. If an air oven is used, the foil will disappear during baking. Remove the assembly and carefully lift up the upper glass plate (the foil's substrate). Often the foil will simply fall freely onto the bottom plate, and then can be handled as described above. If it is nearly free, ease the foil completely free from its substrate first by scraping around the edge of the foil with a razor blade, then sliding a dissecting needle between foil and slide. If the foil has not mostly released, float it off and pick it up in the usual manner (see below). A floating foil can be cut to size while on the water surface, using sharp scissors, making sure that the points of the scissors do not come together during the cut.

Floating off thin foils (surface density < 100 micrograms per square centimeter) from their substrates:

Write down the number on the tag on the back of the slide; this is the surface density of the carbon layer in micrograms per square centimeter. Scrape around the edges of the foil with a razor blade. Use a straightedge (but not in contact with the foil) and a razor blade or other sharp tool to cut the foil into pieces of the desired sizes. If

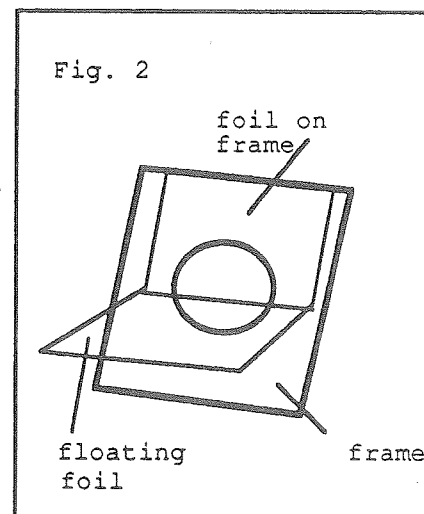
you cut off about one millimeter's width of the foil all around the perimeter, the remainder of the foil often floats more uniformly. Gently blow off any loose fragments. Some people like to exhale onto the foil to humidify it just before floating it.

Use a mechanical arm or your own steady hand. Hold the slide, foil side uppermost, at about 45 degrees to the horizontal. Lower the slide slowly into a dish of clean (preferably distilled or deionized) water at room temperature. Foils will pick up ionic impurities from the water used to float them [Raith *et al.*, Nucl. Instrum. Meth. 142, 39-44 (1977)]. Alternatively, the slide may be held fixed and the dish of water raised below it using a laboratory jack. The smoothest method, preferred for large foils, is to keep both slide and dish fixed, and siphon water from an auxiliary container into the dish. If the auxiliary container has smaller volume than the dish, it will not overflow the dish if your attention is distracted. Alternatively, the slide with foil uppermost may be laid horizontally on the bottom of a dish, and little drops of water placed with a medicine dropper on the intersections of the scribed lines on the foil. After a few minutes, the water will have crept under the foil and more water may be added. When the foil has released completely, water may be siphoned into the dish to lift the foil as needed.



When the water level reaches the foil, it will float on the water surface (Fig. 1). If the foil tends to stick, or is pulled below the water surface, you are doing it too fast. When the foil has floated entirely off of the slide, remove the slide carefully so it won't be in your way for the pickup process. If desired, clean the undesired fragments from the water surface by picking them up with the corner of a paper towel, or sucking them up with a pipette tip, or by allowing them to flow into a little upright empty beaker that you first immerse gently in the water almost to its rim.

Sometimes foil pieces tend to stick together while floating. They can be separated conveniently by touching a boundary between foil pieces with a needle moistened in methanol (W. Lozowski, private communication, 1990).



Frames on which foils are to be picked up should be planar, thin, and clean. It is convenient to have at least one straight edge on a frame. The best thickness for frames is perhaps 0.1-0.2 mm. Thicker frames can be used but the fillet of water that develops at the perimeter of the hole puts extra stress on foils. We like to use copper, aluminum, stainless steel or brass frames, briefly acid-etched to produce a fresh surface, and kept under water until they are used. Hold the frame with tweezers or forceps under water at an angle of about 60-90 degrees to the water's surface, and lift the frame below a piece of floating foil until a corner or edge of the foil drapes over an edge of the frame (Fig. 2). Continue lifting until the foil drapes itself over the hole to be covered. Alternatively, a floating piece of foil can be trapped between the frame and a side of the dish, and picked up without draping it over an edge of the frame. Without touching the foil itself, gently blot off any excess water from the frame. When foils are dry, they are ready to use.