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PREPARATION OF SOURCE MOUNTS FOR  $\alpha$  COUNTING

by

E P JOHNSON

JANUARY 1991

REPORT  
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PREPARATION OF SOURCE MOUNTS FOR 4 $\pi$   
COUNTING

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AUSTRALIAN NUCLEAR SCIENCE  
AND TECHNOLOGY ORGANISATION

LUCAS HEIGHTS RESEARCH LABORATORIES

PREPARATION OF SOURCE MOUNTS FOR  $4\pi$  COUNTING

by

E. PHILIP JOHNSON

ABSTRACT

The  $4\pi\beta/\gamma$  counter in the ANSTO radioisotope standards laboratory at Lucas Heights constitutes part of the Australian national standard for radioactivity. Sources to be measured in the counter must be mounted on a substrate which is strong enough to withstand careful handling and transport. The substrate must also be electrically conducting to minimise counting errors caused by charging of the source, and it must have very low superficial density so that little or none of the radiation is absorbed. The entire process of fabrication of VYNS films, coating them with gold/palladium and transferring them to source mount rings, as carried out in the radioisotope standards laboratory, is documented.

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ACETATES; ANSTO; CALIBRATION STANDARDS; COATINGS; COPOLYMERS; FABRICATION; FOILS; FOUR-PI COUNTING; GOLD; MANUALS; OPACITY; PALLADIUM; PVC; RADIATION SOURCES; SOLUTIONS; SUBSTRATES; SURFACE COATING; THICKNESS; THIN FILMS; VACUUM EVAPORATION.

#### EDITORIAL NOTE

The Australian Nuclear Science and Technology Organisation (ANSTO) replaced the Australian Atomic Energy Commission (AAEC) on 27 April 1987. Reports issued after April 1987 have the prefix ANSTO with no change of the symbol (E, M, S or C) or numbering sequence.

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## 1. INTRODUCTION

Pate and Yaffe [1955] reported on the use of VYNS\* resin as a material for making thin films which are suitable for source mounts in  $4\pi$  counting and beta-spectrometry. In a comprehensive report, details were given of a method to convert the powder into thin plastic films; several methods for determining the superficial densities of the films produced; information on the deposition of gold onto the films to render them conducting; and a technique for measuring the amount of gold deposited. D.W. Green [1961] reported a new method of making the thin VYNS film.

The author has been informed that many overseas laboratories are still having difficulties with the manufacture of source mounts.

In the radioisotope standards laboratory at ANSTO, procedures based on the works quoted above have evolved which have a reasonably low failure rate and are relatively fast.

This manual describes in great detail the entire process of source mount manufacture, as carried out in this laboratory. Where lists of instructions are given, they are specific to the equipment in the radioisotope standards laboratory at ANSTO, but the general method would apply to any similar system.

## 2. MANUFACTURE OF THIN VYNS FILMS

### 2.1 Apparatus

A plastic developing tray 63cm long by 53cm wide by 8cm deep rests on a draining board with part of the long side over a sink (figure 1). Tap water is filtered and passed slowly into the tray at the far end. A slot cut 2.5cm deep and 15cm long allows water to drain from the surface into the sink so there is a constant slow replacement of the water in the tray and floating dust is skimmed off. A drain hole over the sink is plugged when the apparatus is in use. The inside surface of the tray is painted black and an illuminated white board above it allows easy inspection of the film when it is floated onto the water.

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\*VYNS is a copolymer of polyvinyl chloride and polyvinyl acetate introduced by the Bakelite Company and usually supplied in powder form.

Aluminium frames are used to support the films as they are removed from the surface. These are rectangular, 8.3cm by 5.1cm on the outside and 7.3cm by 3.8cm on the inside; 1.25mm thick and slightly rounded on the corners. They are pre-painted on one side with VYNS solution to help them stick to the film.

Tweezers are used to place the frames onto the VYNS spread. These should be of a design which allows the side of the frame to be held and the frame placed down so that no damage is done to the film.

Perspex storage boxes with slots in the sides have been specially designed to minimise damage to the films on insertion or extraction and during transportation. Each box holds fourteen films and twenty such boxes is a useful number.

The VYNS solution is contained in a 250ml bottle and a Pasteur pipette with rubber stopple is used to transfer the solution onto a 7.5cm by 5.0cm microscope slide, thence to the water surface.

Ideally the room temperature should not exceed 20°C and the water temperature should not exceed 15°C. This is easily achieved in winter. At other times an air conditioned room and ice in the water would be required.

## 2.2 Preparation of VYNS Solution

The VYNS solution is prepared the day before a batch of films is to be made, i.e. no stored stock solution is used. It is made by stirring 25ml of the VYNS powder into 100ml of pure cyclohexanone. The one to four ratio produces films predominantly of superficial density  $20 \mu\text{g}\cdot\text{cm}^{-2}$ . Other concentrations may be used if thinner or thicker films are required. 100ml is far more than is needed to make a large batch of films but the remainder can be used for other purposes such as coating source mount rings.

Initially the cyclohexanone is in a 200ml beaker inside a fume cupboard. It is stirred slowly by a glass paddle driven by a motor mounted above it, whilst the powder is cautiously introduced. The speed of the stirring is such that no air is forced into the solution. It takes about two hours to dispose of the powder and stirring can continue after that. At the end of

the working day the stirrer is removed and the solution left overnight to ensure that it is homogeneous. Figure 2 shows the preparation of the solution.

### 2.3 Spreading the Film onto the Water Surface

This work is best done seated comfortably in front of the tray.

The microscope slide is held in one hand lengthwise between forefinger and thumb, best long edge uppermost and furthest from the hand. Using the pipette, a thin line of VYNS solution is deposited on the slide surface close to this edge, but with a gap of about 1cm left at each end. The pipette is replaced into the solution with no relaxation of finger pressure on the stopple till the end is beneath the surface so that no air bubbles enter the pipette. Forefinger and thumb of both hands are used to hold the slide with the solution towards the water, and the slide is tilted down at an angle of about  $45^\circ$  and lowered to the water until the VYNS solution just starts to spread out across the surface, and is then held steady. When spreading is finished, the slide is lifted out slowly with the film still attached and drawn over the edge of the tray to anchor the film onto it. The slide is then cleaned with a tissue and laid in a clean convenient place.

Whilst spreading, the film goes out into an oval shape much wider than the slide so that large areas of uniform film can be obtained.

There are three ways in which the average thickness of the final spread of film can be influenced during this process:

- (i) If a greater quantity of solution is deposited on the slide, the spread will be thicker,
- (ii) A shallower angle of approach of the slide to the water will result in a thinner film, and
- (iii) The depth below the surface of the edge of the slide. If the slide is halted just as it touches the water surface, the thinnest film will result.

The temperature of the water also influences the thickness of the film but there is evidence that films made at temperatures above  $15^\circ\text{C}$  are more likely to break during long term storage.

#### 2.4 Removing the Films on the Aluminium Frames

An aluminium rectangle or frame is grasped with the tweezers by an edge so that it is held in a horizontal plane, with the pre-painted side down, and the tweezers do not protrude through the hole. An area on the floating film is selected with good uniform appearance and required thickness (section 2.5). The frame is lowered slowly all the way onto the film and released only when it is in contact. As many frames may be set down as the size and uniformity of the spread of film will allow.

Removal of the frame with its attached film requires great care. (The whole task requires great patience). A side of the rectangle is lightly supported with one hand whilst the film is carefully torn away from around its perimeter using the tweezers. The film has preferential directions of tearing so care must be taken to ensure that a split does not extend under the edge of the frame into the required area inside. A slight lifting of the film whilst tearing it away from the metal edge may help here. It may be necessary to rotate the floating frame a few times while doing this. When floating film has been cleared from around the frame, it is removed from the water in the following way. A long edge of the rectangle is held firmly with one hand, and an opposite corner is pushed slowly under the surface until the whole film is under water. The frame is then lifted vertically through the water surface slowly and steadily and usually comes out free of water droplets. If water droplets are seen on the film before it is completely out of the water, it should be re-immersed and brought out again in an attempt to get rid of them. Water droplets which dry out may leave weak spots on the film.

The metal frame with its enclosed film can now be placed into a storage box. When all the frames have been removed from the surface, unwanted film can be collected up on the tweezers. This should be squeezed, away from the tray, to remove water, and then discarded. A lint-free towel may be used to dry the hands before repeating the procedure. Many films will be lost by breakage during manufacture, and it is essential to accept this philosophically because a tranquil mind is required for optimum success in this task.

## 2.5 Sorting the Films According to Thickness

Determination of the superficial density of VYNS films has been dealt with in detail by Pate and Yaffe [1955]. A brief account of the method used in this laboratory is included only for completeness.

The films are inspected by observing the effect of reflecting white light from their surfaces. Up to  $10 \mu\text{g}\cdot\text{cm}^{-2}$ , the reflected light is seen as shades of grey or white. Above that, colours due to interference appear. As they are inspected the films are graded into labelled boxes.

Colour or Shade of White observed	Thickness of Film in $\mu\text{g}\cdot\text{cm}^{-2}$
Dark Grey	5
Light Grey	7
White	10
Pale Yellow	20
Yellow/Brown	25
Purple	30

## 3. EVAPORATION OF GOLD/PALLADIUM ONTO THE FILMS

### 3.1 Preamble

To prevent charging of the sources in the  $4\pi$  gas counter, the films are coated with a thin layer of gold/palladium by evaporation in vacuum. An alloy of 80% gold, 20% palladium is used. The films are coated on the same rectangular aluminium frames they were made on.

### 3.2 Preparation of Films for Coating

#### 3.2.1 Selection of films

The Perspex boxes which hold the films are stored in a wall cupboard. Each one is marked 5, 7, 10, 15 or 20, according to the mass per unit area of the films which it holds in  $\mu\text{g}\cdot\text{cm}^{-2}$ . To avoid confusion, only films from the same box should be coated at any one time.

#### 3.2.2 Placement of films in coating position

##### a) Removal of bell jar

Figure 3 shows the layout of the vacuum system. Figure 4 is a photograph of the vacuum system. The bell jar is normally left at ambient pressure. Before removing the safety shield this can be checked by slowly turning the air inlet valve (5) anti-clockwise.

If a hissing noise is heard, the bell jar is under vacuum and air should be allowed to enter until the hissing noise ceases. The safety shield can now be removed. After the bell jar is removed it is laid on a bench horizontally so that the greased seal will not get dirty.

b) Placement of films in jig

This is best explained by listing a series of steps:

- (i) The components inside the bell jar are cleaned. In particular, the electrical insulators must not be allowed to accumulate a coating of gold/palladium. The inside surface of the bell jar should also be cleaned.
- (ii) The glass slide is removed from its holder, cleaned and replaced.
- (iii) From time to time the tungsten filament breaks. It is renewed with 0.5mm diameter tungsten wire. A piece about 6cm long is cut and a vee shaped notch about 5mm deep is made at the centre. This is to hold the gold/palladium. The filament is pressed into the slots in the upright posts (electrodes) with the notch pointing down.  
Before it can be used, the new filament must be annealed and de-oxidised. This is done with no films inside the bell jar. The system is pumped down (see 3.3) to less than 13mPa and the filament current is slowly wound up to 12.5 amps. After one minute the current is slowly wound back to zero. The system is then returned to ambient pressure, the bell jar removed, and the procedure continued.
- (iv) A 1cm length of the 0.5mm diameter gold/palladium wire is bent into a hairpin shape. It is placed onto the tungsten filament so that it hangs from the bottom of the notch.
- (v) The molybdenum "flipper" is lowered to cover the gold/palladium loop. This is to catch droplets when the loop first melts. At this stage the action of the flipper may be checked if required.
- (vi) The required box of films is taken from the wall cupboard and placed on the bench near the coating plant. It is left there throughout the coating process so that the grade of the films will not be forgotten.

(vii) The films are carefully lifted one by one and placed into the slots in the brass jig (figure 5). The side of the aluminium rectangle to which the film is attached should be furthest away from the filament at the centre of the jig. This operation has to be done carefully because it is easy to damage the films if they are touched by the fingers. The lid is replaced on the Perspex box.

(viii) The bell jar is replaced.

(ix) The safety shield is replaced around the bell jar.

### 3.3 Starting the Vacuum Pump

Since it is possible to isolate the bell jar for film changing as required, the vacuum pump may be started at any convenient time. Here it will be assumed that the bell jar and safety screen are in position.

The sequence of operations, which takes about three hours, is as follows:

- (i) The power supply to the entire system is switched on.
- (ii) The bell jar air inlet valve (5) is closed.
- (iii) The rotary pump air inlet valve (6) is closed.
- (iv) The rotary pump is switched on.
- (v) Cooling water is turned on. The water circulates around the pump and then flows into a drain near the tap.
- (vi) Liquid nitrogen is poured into the trap until it is full.
- (vii) The diffusion pump is switched on.
- (viii) The rotary pump valve (1) is opened.
- (ix) The diffusion pump backing valve (2) is opened.
- (x) When the pressure is down to 50 microns or less, the diffusion pump backing valve (2) is closed.
- (xi) The bell jar roughing valve (3) is opened to evacuate the bell jar. This must be done very slowly so as not to break the films. The Pirani gauge can be watched to observe that the pressure only drops slowly.
- (xii) When the pressure in the bell jar is down to 50 microns or less, the diffusion pump backing valve (2) is opened. N.B. When the diffusion pump is on, it should not be isolated from the rotary pump for longer than 20 minutes.
- (xiii) The bell jar roughing valve (3) is closed, and as soon as possible the flap valve (4) is opened.

(xiv) The Penning gauge should be on its least sensitive range at this stage, and as the pressure in the bell jar goes down slowly, the gauge will come into operation. Evaporation of the gold/palladium should not commence until the pressure is at most 13 mPa, and if the pressure is even lower, say 5 mPa, the evaporated coating will be better.

### 3.4 Evaporation of Gold/Palladium

#### 3.4.1 Preamble

Coating is done by passing a current through the tungsten filament. The gold/palladium wire melts and forms a spherical bead at the bottom of the notch in the centre of the tungsten filament which slowly evaporates. Atoms stream radially outwards onto the films, coating them evenly over their surfaces. At the same time a glass microscope slide is also being coated and this is used to monitor the progress of the coating. A beam of light passes through the glass to a photoresistor connected to an external ohmmeter. As the glass becomes darker the resistance of the photoresistor increases providing an indication of the degree of coating of the films.

#### 3.4.2 List of steps to be taken

- (a) Polarity of the photoresistor connections; the green lead from the photoresistor is connected to the d.c.- terminal of the external ohmmeter (in our case an Avo 8).
- (b) Set in the  $\Omega$  position, the Avo terminals are short circuited and the pointer adjusted to zero.
- (c) The shorting bar is removed and the "LIGHT" control adjusted until the Avo reads about  $200\Omega$ .
- (d) The heater is switched on and the variac turned up to about 90 volts and adjusted to give a steady current of 12 amps.
- (e) If a new piece of gold/palladium wire is on the tungsten filament it is watched until it melts and forms a sphere and then a few more seconds allowed for splashing of fine particles to cease.
- (f) The "FLIPPER" switch is depressed and released. This activates an electromagnet which moves the molybdenum shield away from the gold/palladium bead.
- (g) The variac is turned down slowly and the heater switched off when the Avo indicates the desired resistance. This will be found by trial and error, but  $800\Omega$  is suggested for an initial attempt.

### 3.5 Isolation of the Bell Jar

Should it be necessary to remove the bell jar with the pumps still running (in order to change the films for example), the sequence of events is as follows:

- (i) The Penning gauge is switched to range 1 or its least sensitive range.
- (ii) The flap-valve (4) is closed. A springy resistance will be felt so a little extra force must be applied but there is an unmistakable positive stop when the valve reaches its fully clockwise position.
- (iii) The bell jar air inlet valve (5) is opened very slowly. When the valve stops hissing the safety screen and bell jar can be removed as described in section 3.2.2 (a).
- (iv) The coated films, now called foils because they are metallised, can be lifted carefully from the brass jig and may be put back into the Perspex box from which they came until the amount of coating is measured.

### 3.6 Re-evacuation of the Bell Jar

If it is desired to establish the high vacuum in the bell jar again (in order to coat another set of films, for example), the bell jar and safety screen are replaced and the following operations from section 3.3 are performed in the sequence given:

3.3 (ii), (ix), (x), (xi), (xii), (xiii).

### 3.7 Closing Down the Vacuum System

The system is normally left with the bell jar and the rotary pump at atmospheric pressure, and the diffusion pump and other plumbing under vacuum. To close the system down to its rest position, the following operations must be performed:

- (i) The Penning gauge is set on its least sensitive range.
- (ii) The flap valve (4) is closed.
- (iii) The bell jar air inlet valve (5) is opened slowly.
- (iv) The diffusion pump is switched off and the pump allowed to cool for at least 20 minutes with water flowing, then:
- (v) The diffusion pump backing valve (2) is closed.
- (vi) The rotary pump valve (1) is closed.
- (vii) The rotary pump is switched off.
- (viii) The rotary pump air inlet valve (6) is opened.
- (ix) The mains is switched off at the wall.
- (x) The water supply is turned off.

Note: It is advisable to open the air ballast valve on the rotary pump occasionally in order to expel water vapour from the oil. This is best done when the pump has been running for some time. One quarter of a turn anti-clockwise is sufficient.

#### 4. DETERMINATION OF THE MASS PER UNIT AREA OF THE Au/Pd COATING

##### 4.1 Preamble

After removal from the vacuum system, the foils should have been placed in a box marked on its ends with the relevant number for the mass of VYNS per unit area. This is because once they have been coated, the thickness of the VYNS cannot be redetermined, so their identity must be preserved throughout all the preparations.

For each foil the optical density is measured and the amount of gold/palladium deposited is then found from a calibration chart. Optical density is the logarithm of the reciprocal of transmission.

##### 4.2 Instruments Used

A Baldwin transmission densitometer mark 4B connected to a photometer M.N.D. mark 5 is used to measure the optical density of the foils.

##### 4.3 Procedure

One foil is inspected at a time. A few measurements of optical density may be made over the area of the foil, and an average value taken mentally. It should be borne in mind that the foil will later be transferred onto two brass source rings, so attention should be concentrated on the two points where the sources will later be deposited. These points are on an imaginary line midway between the two longer sides and about 2cm in from the inner edge of the rectangle.

The following steps are taken:

- (i) From the wall cupboard is taken a Perspex foil box marked on its ends with the appropriate value of mass per unit area of VYNS. If the foils are to be taken directly from the vacuum system, the box should have at least seven empty slots. It is placed on the bench near the photometer to receive the foils as they are measured.
- (ii) The grey plastic covers are removed from the instruments.

- (iii) The power to the instruments is switched on. A light in the transmission densitometer will come on.
- (iv) A warm up period of 20 minutes is given.
- (v) There is a clip which holds down the moving arm of the densitometer. This is released and pushed back and up onto its retaining peg.
- (vi) With the photometer on density range 0, its set zero control is adjusted so that the pointer is on the highest mark on the density scale, just above 2.
- (vii) The black roller at the front of the moving arm is pulled forward. This opens the photodetector shutter and a deflection of the photometer pointer will occur.
- (viii) The arm is lowered and held down firmly but not with excessive pressure. The coarse and fine controls are adjusted so that the photometer pointer is at 0 on the density scale. This adjustment and the set zero on the photometer should be checked from time to time. The arm is then raised back to its rest position.
- (ix) One of the foils is placed onto the densitometer glass plate over the light aperture in the black disc. The side of the aluminium rectangle with the film attached must be uppermost.
- (x) The roller is pulled forward and the arm lowered as before. The optical density will be indicated on the photometer and, as mentioned earlier, the foil may be moved around to obtain an average value.
- (xi) The foil is placed in the Perspex box.
- (xii) The chart below gives the superficial density of the gold/palladium coating in  $\mu\text{g.cm}^{-2}$  according to its optical density. This usually lies within the range 10 to 25.

Density	$\mu\text{g.cm}^{-2}$	Density	$\mu\text{g.cm}^{-2}$	Density	$\mu\text{g.cm}^{-2}$	Density	$\mu\text{g.cm}^{-2}$
0.19	10	0.27	14	0.35	18	0.43	22
0.20		0.28		0.36		0.44	
0.21	11	0.29	15	0.37	19	0.45	23
0.22		0.30		0.38		0.46	
0.23	12	0.31	16	0.39	20	0.47	24
0.24		0.32		0.40		0.48	
0.25	13	0.33	17	0.41	21	0.49	25
0.26		0.34		0.42		0.50	

- (xiii) By use of a chinagraph pencil, the value is marked on the lid of the Perspex box above the slot which the foil was put into.
- (xiv) When all of the foils have been measured, the lid is replaced on the Perspex box which is returned to the storage cupboard.
- (xv) If the instruments are not required for the rest of the day, steps (iii), (v) and (ii) are reversed to set them at rest.

#### 5. TRANSFER OF THE FOILS ONTO SOURCE MOUNT RINGS

The brass rings onto which the foils are transferred to make the complete source mounts are 3.5cm O.D. by 2.5cm I.D. They have three legs about 2.5mm long arranged symmetrically on a 3.0cm diameter. If two are placed side by side it is possible to transfer foil to both of them from a single aluminium rectangle and this represents a considerable improvement in efficiency compared with the original one to one method.

Before the foil is transferred, the top surface of each ring is painted with a solution of VYNS in cyclohexanone and allowed to dry. The film of VYNS on the brass ring facilitates adhesion of the foil. The aluminium frame, with the gold/palladium coating uppermost, is lowered gently onto the two brass rings (figure 6). On most occasions the foil sticks immediately to the rings but if small gaps are seen, gentle breathing on the foil will help to remove them. To remove the excess foil from around the brass rings a scalpel with a small amount of cyclohexanone on the tip of the blade is used. The tip is traced carefully around the rings until all extraneous foil is gone. It is now possible to lift the rings away from the aluminium frame.

One more operation is necessary before the source mount is complete. To ensure a conducting path between the conductive coating and the brass ring a small area of the foil is scraped away to expose the brass surface in three places around the ring close to its outer perimeter. Conducting paint is brushed onto the areas, joining the gold/palladium surface to the brass electrically.

There are a number of different types of source made in the radioisotope standards laboratory and their preparation has been documented by H.A. Wyllie [1988].

6. ACKNOWLEDGEMENTS

Dr G.C. Lowenthal and Mr D.F. Urquhart introduced the methods described herein to the radioisotope standards laboratory at Lucas Heights and supervised the setting up and development of the equipment.

7. REFERENCES

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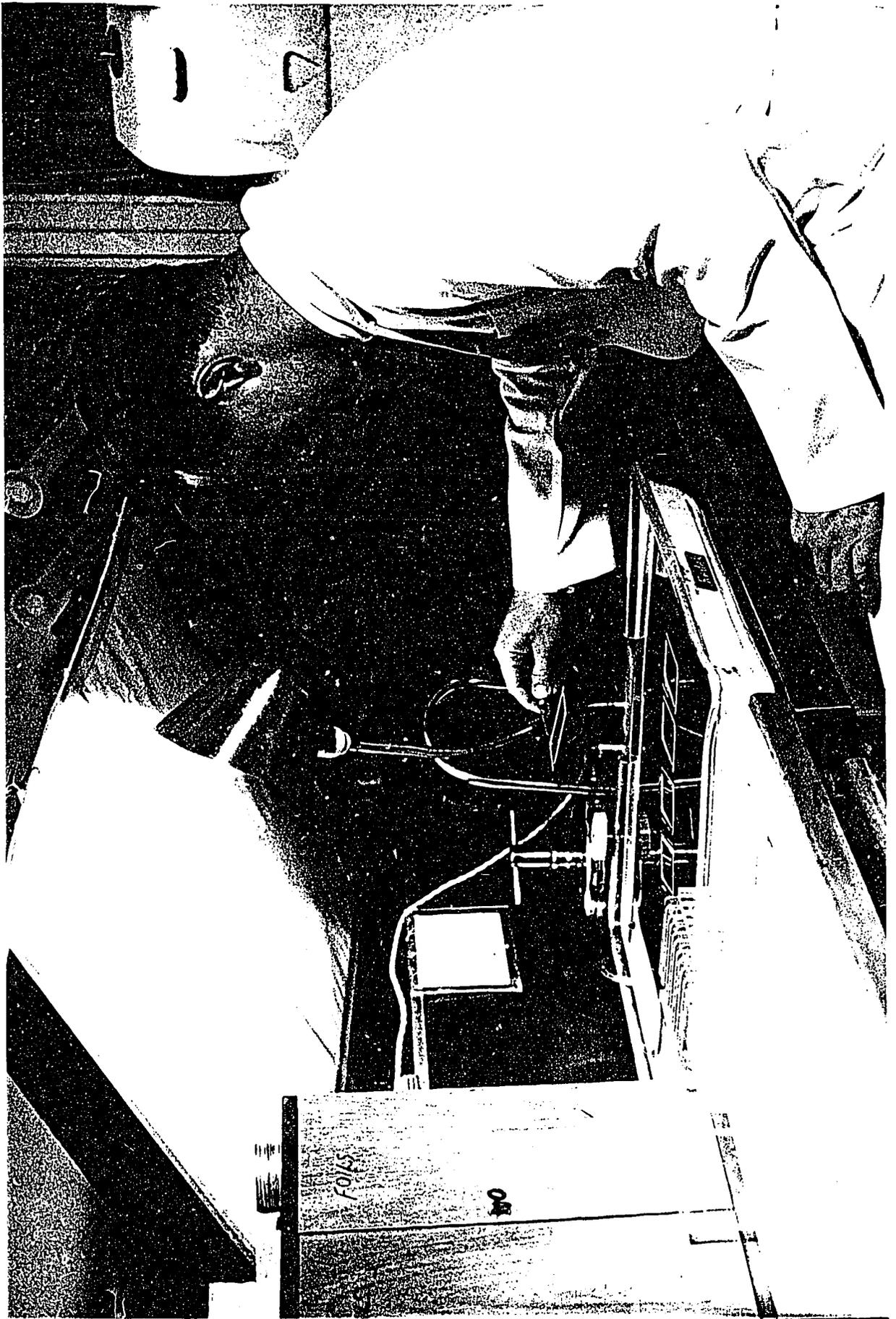


Figure 1. Manufacture of Thin VVNS Films

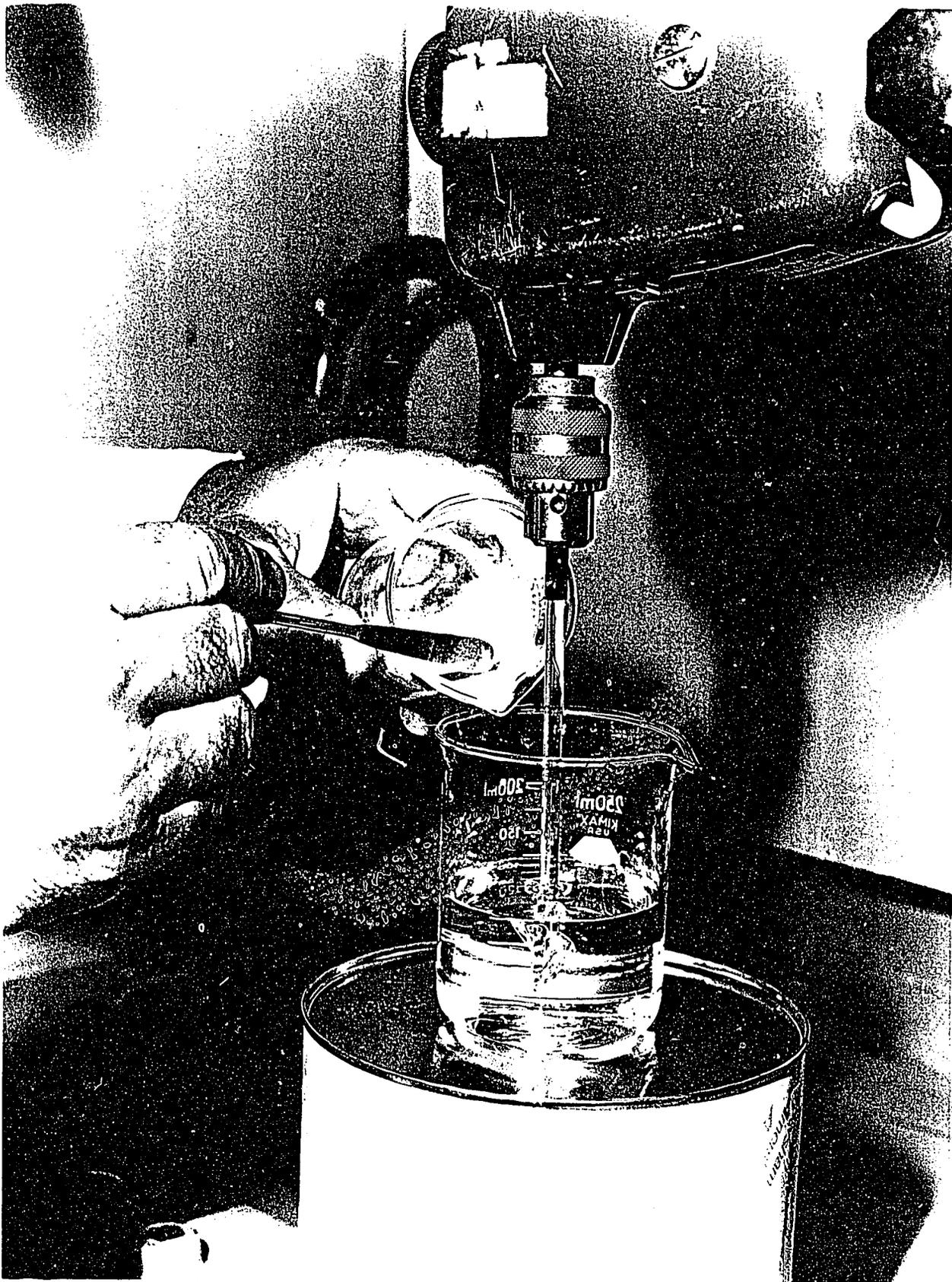


Figure 2. Preparation of VYNS Solution

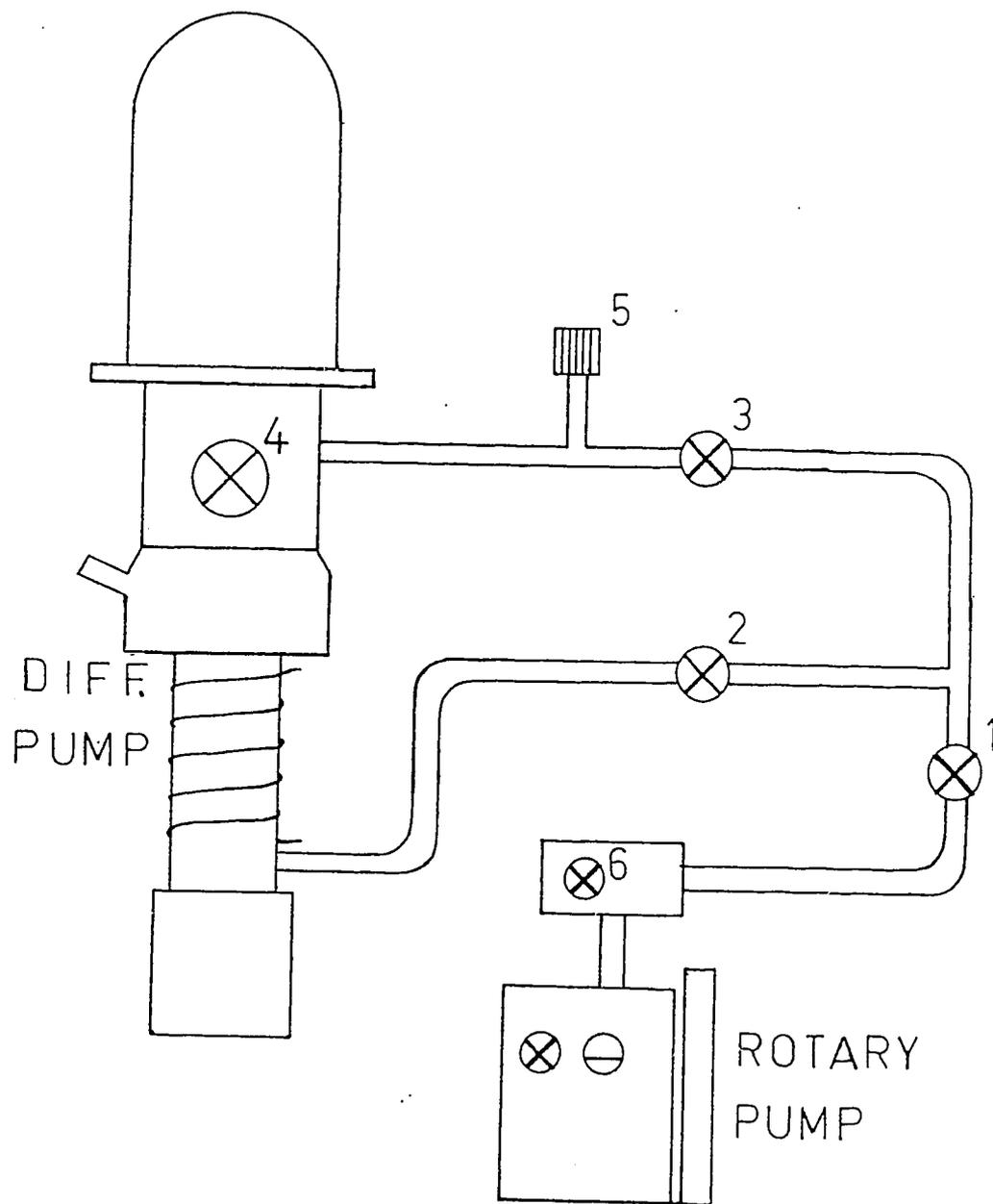


Figure 3. Schematic Diagram of the Vacuum System

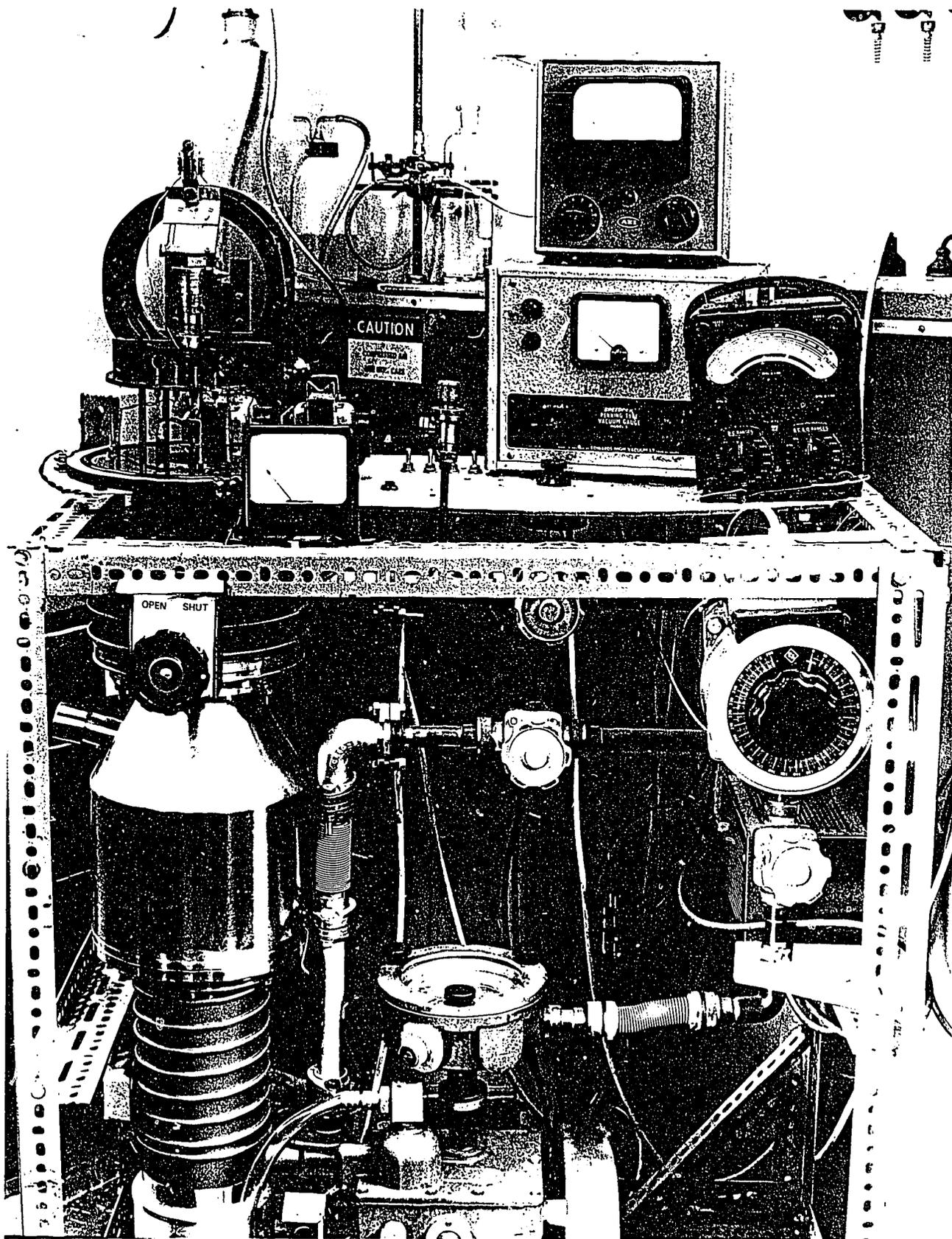


Figure 4. View of the Vacuum System

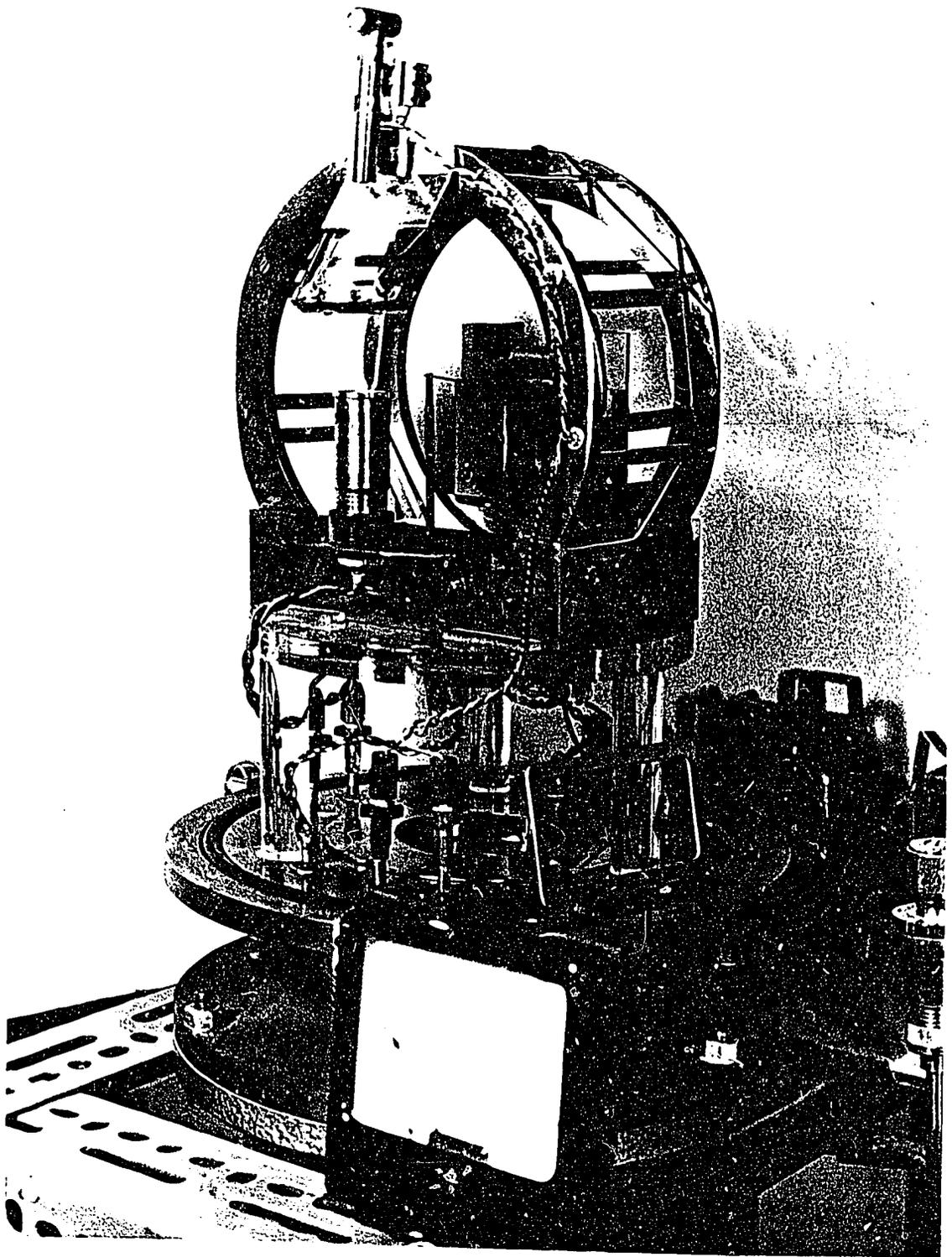


Figure 5. The Brass Jig and Evaporation Apparatus

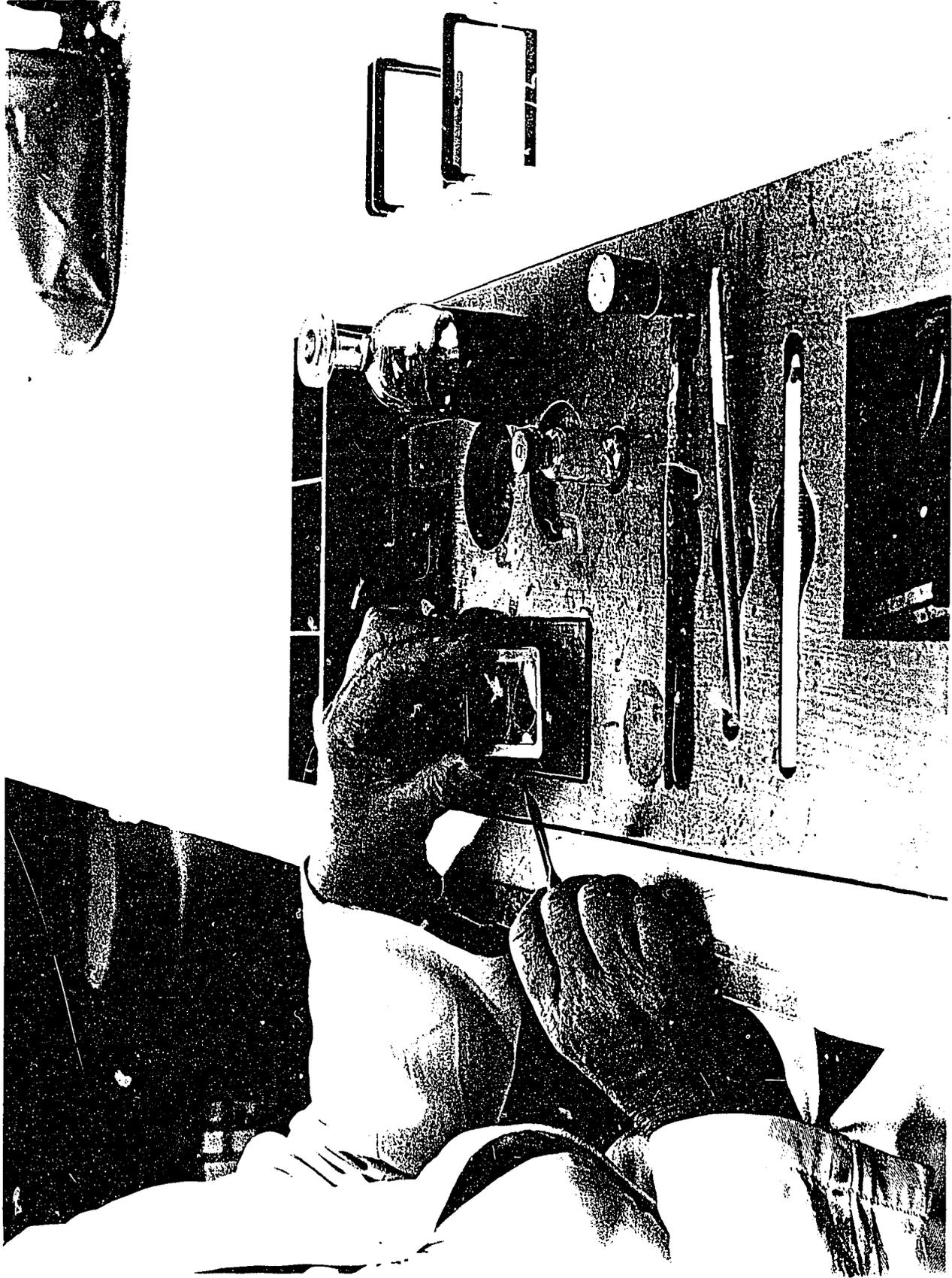


Figure 6. Transfer of Foil onto Source Mount Rings