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THE PREPARATION OF RADIOACTIVE SOURCES WITH RADIOACTIVITIES OF LESS THAN 110 KILOBECQUERELS

by

H.A. WYLLIE

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LUCAS HEIGHTS RESEARCH LABORATORIES

THE PREPARATION OF RADIOACTIVE SOURCES WITH RADIOACTIVITIES OF LESS THAN 110 KILOBECQUERELS

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ABSTRACT

A description is given of the various radioactive sources prepared in the ANSTO Radioisotope Standards Laboratory and the procedures associated with their preparation. ANSTO is authorised by CSIRO to maintain the Commonwealth standard of activity of radionuclides. Counting sources are required for the standardisation of solutions of radionuclides. Calibration sources are required for equipment used to detect radioactivity, such as gamma-ray spectrometers, and can be supplied to clients in other organisations. The maximum radioactivity supplied is 110 kBq.

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The following descriptors have been selected from the INIS Thesaurus to describe the subject content of this report for information retrieval purposes. For further details please refer to IAEA-INIS-12 (INIS: Manual for Indexing) and IAEA-INIS-13 (INIS: Thesaurus) published in Vienna by the International Atomic Energy Agency.

BALANCES; COINCIDENCE METHODS; CALIBRATION; DEAD TIME; DEPOSITION; CALIBRATION STANDARDS; FILMS; FOUR-PI COUNTING; MANUALS; MASS; PYCNOMETERS; RADIATION SOURCES; RADIOACTIVITY; SOLUTIONS; STANDARDIZATION; WEIGHT.

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.

CONTENTS

1. INTRODUCTION

This manual describes the procedures for making radioactive sources with radioactivity of less than 110 kBq in the Radioisotope Standards Laboratory at the Lucas Heights Research Laboratories. Many of the procedures originate from publications referred to by the US National Council on Radiation Protection and Measurements [NCRP 1985]. Other procedures were devised by former members of this laboratory. Descriptions are given of the handling and dispensing of radioactive solutions, the preparation of sources used in the standardisation of radioactive solutions, and the preparation of calibration sources for γ-ray spectrometers used in the indirect determination of radioactivity.

2. THE FILLING AND SEALING OF AMPOULES

Standardised radioactive solutions are stored preferably in sealed standard glass ampoules. This ensures that there is no change in concentration due to loss of water by evaporation and leakage of vapour during storage. When a source is prepared from a solution stored in an ampoule, that part of the solution not required for source-making is transferred to a new ampoule which is then sealed off as shown in **figure 1**. Before starting the transfer, the new ampoule is labelled with the same information as the original ampoule, namely nuclide and solution number.

During storage, water evaporates from the surface of the acid solution then condenses in the top of the ampoule. The water is restored to the solution by centrifuging the ampoule. The centrifuge tubes are fitted with Perspex inserts to accommodate ampoules or vials. Counterweights are available, consisting of ampoules and vials containing varying amounts of water. To eliminate the possibility of the base of an ampoule or vial breaking during centrifuging, the maximum angular velocity is set at 2500 rev min^{-1}, as shown on the liquid (vortex) scale. The horizontal distance from the centre of the centrifuge to the base of the spinning ampoule or vial is 13 cm. At the start of centrifuging, the control knob is turned to its maximum setting and, when 2500 rev min^{-1} is reached, the knob is turned back to the setting for 2500 rev min^{-1} (numeral "1" on the dial).

After centrifuging for one minute, the ampoule is opened by cutting a narrow groove around the neck with a file, the edge of which has been ground to form a sharp saw (**figure 2**), and snapping off the tubing above the file mark. A rubber stopper is lightly inserted in the top of the opened ampoule to prevent evaporation (**figure 3**).

The solution is transferred with a syringe which can be used as a pycnometer in the preparation of sources [Wyllie, 1986]. Before use, the syringe plunger (which is of cruciform cross-section) is rotated about its axis to bring it to the correct angle for gripping by the forceps used during weighing operations (**figure 4**). Except when solution is being transferred, the new ampoule is closed with a rubber stopper. When transferring the solution from the syringe, care should be taken to avoid splashing the upper section of the ampoule. This can be done by using the pycnometer holder designed by Lowenthal and Page [1970] - see **figure 5**. First, air is drawn in above the solution. The

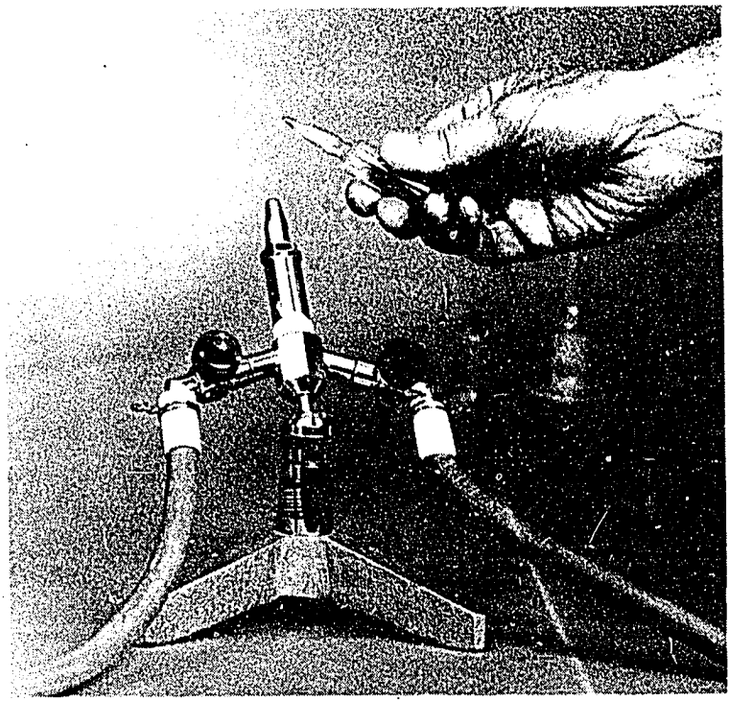


Figure 1

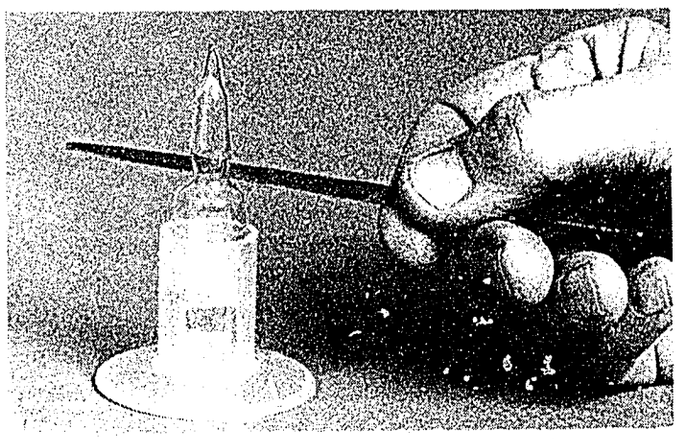


Figure 2

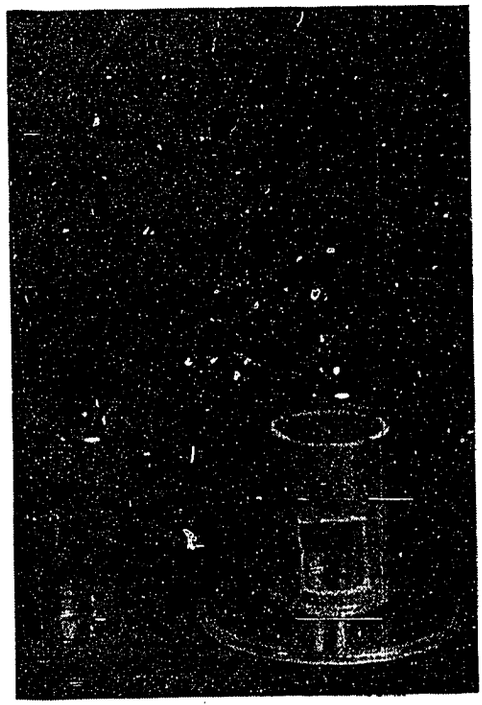


Figure 3

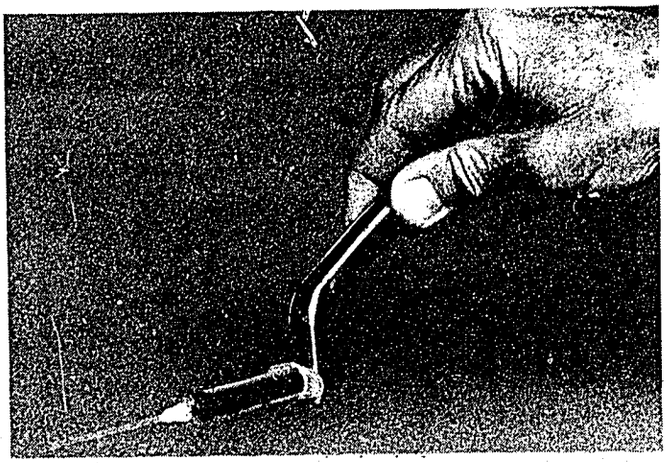


Figure 4

Figure 5

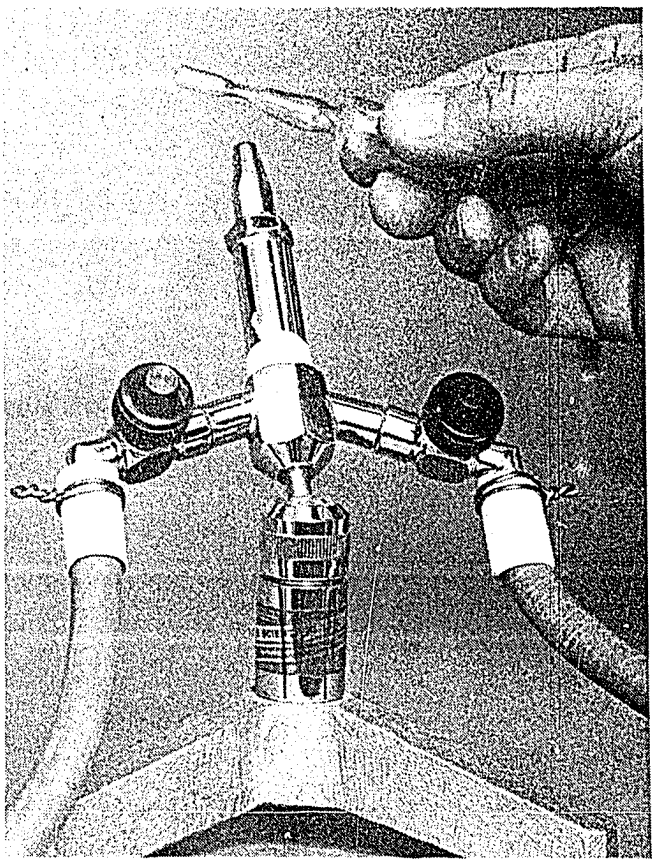


Figure 6

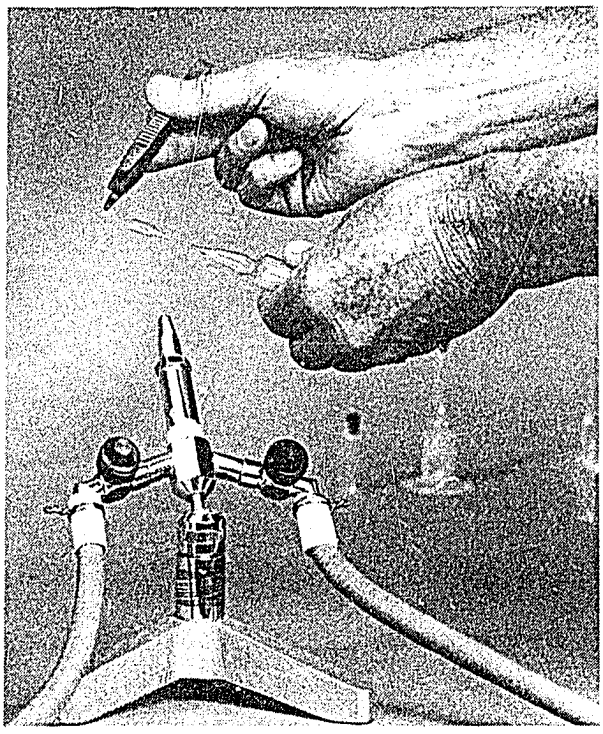


Figure 7

solution is then run into the ampoule drop by drop. When an amount of solution less than the volume of a pendant drop big enough to break away of its own accord remains in the needle, the plunger is withdrawn to draw the solution back to the top of the needle. If the last drop is blown out by a gust of compressed air, there is a possibility of droplets adhering to the outside surface of the needle. When the syringe is withdrawn from the ampoule, the end of the needle should be examined to check that no droplet is on the outer surface. During transfer operations, the old and new ampoules are kept upright in stands made by cutting off the tops of plastic measuring cylinders. After the original ampoule has been emptied it is placed in the radioactive waste receptacle.

The method for weighing the solution in the new ampoule is described in **section 3.** When sealing the ampoule, care must be taken to avoid drawing out the heated glass in such a way that the wall at the point of sealing is left too thin. This can be avoided by thickening the wall of the neck before sealing it off. The stopper is removed and the neck rotated in a small oxy-LP gas flame so that it shrinks lengthwise to form a constriction (**figure 6**). The flame is then concentrated on the top half of the constriction for a short time, the ampoule is withdrawn from the flame, and the neck is drawn out (**figure 7**) with a pair of forceps. The lower end of the constriction is heated to close it completely, the top end of the neck is removed, and the sealed end of the ampoule is rounded in the flame. To avoid strains in the sealed end, its temperature should be lowered slowly by continuing to heat it for a short time in a smoky flame obtained by turning off the oxygen to the burner.

3. MASS DETERMINATION OF SOLUTION DEPOSITED IN AN AMPOULE

Generally, when solution is completely transferred to a new ampoule it is not necessary to weigh it. However, if a diluted solution is to be prepared in the new ampoule, the mass of solution transferred is determined by one of two methods. If more than 0.3 g is transferred, its mass will be determined by weighing the ampoule, stopper and stand on a semi-microbalance before and after filling the ampoule. Depositions of the order of one or two hundred milligrams are determined by weighing a syringe pycnometer on a Mettler M3 electronic microbalance (**figure 8**) before and after the deposition [Wyllie 1986]. For this operation, the balance pan is replaced by a lighter, stainless-steel, wire stirrup (**figure 9**). When making the zero adjustment, the stirrup is loaded with a piece of wire whose mass of 0.401 g equals the difference between the masses of the pan and the stirrup (**figure 9**). Because the pycnometer is too long to fit into the balance, the weighing chamber is extended 4 cm by leaving the left-hand door open and placing across the opening a double-walled glass box which is open on the right-hand side. The gap between box and balance is sealed with adhesive tape (**figure 10**).

Before making the initial weighing, the syringe pycnometer (**figure 11**) containing the necessary amount of radionuclide solution is placed at the back of the weighing chamber for 20 minutes so that it may attain the temperature of the chamber. In **figures 8** and **9**, the syringe can be seen immediately in front of the polonium-210 static eliminator discs (supplied by Amersham Australia Pty. Ltd., Catalogue No. PDV 3611; useful life : one year) which ensure accurate weighing by removing

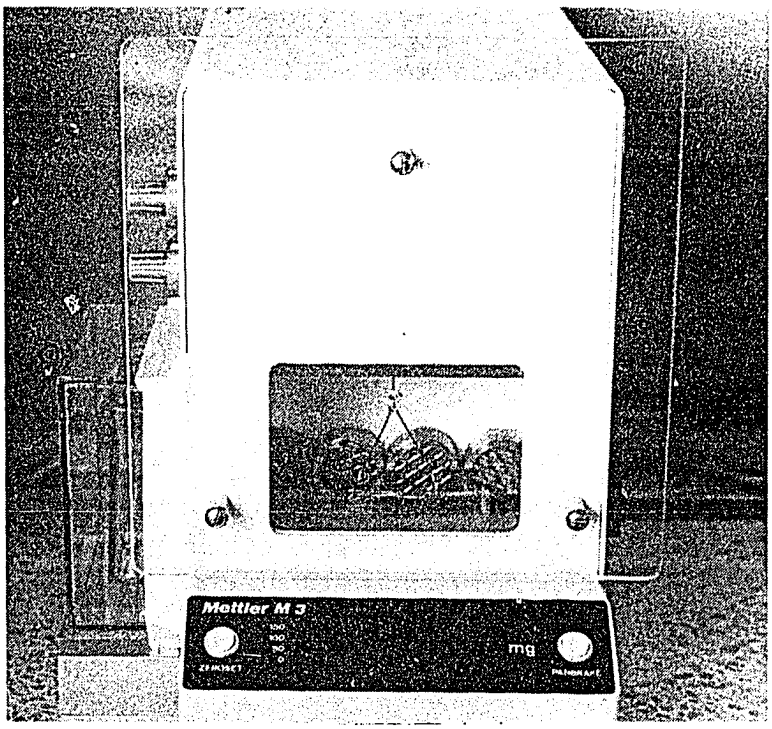


Figure 8

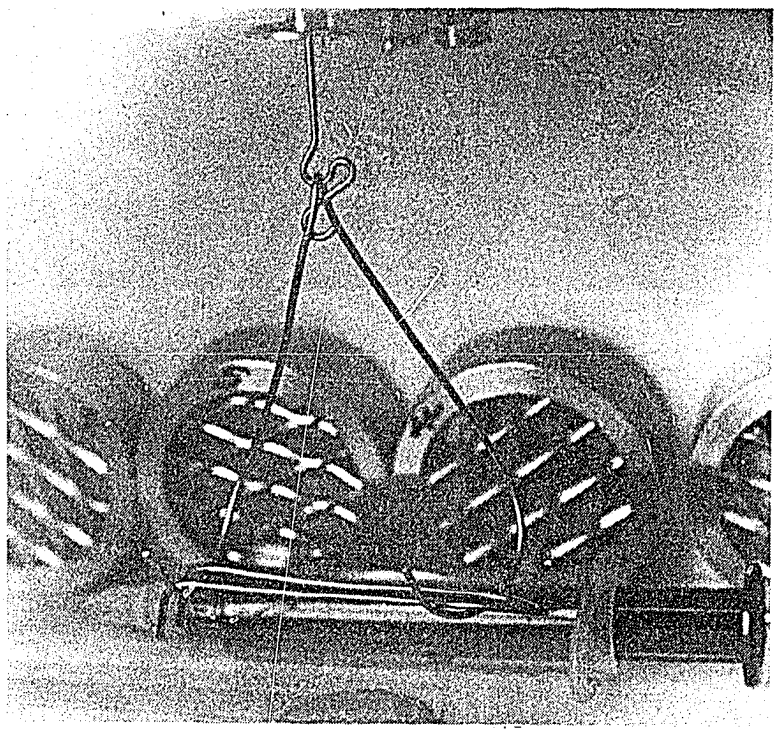


Figure 9

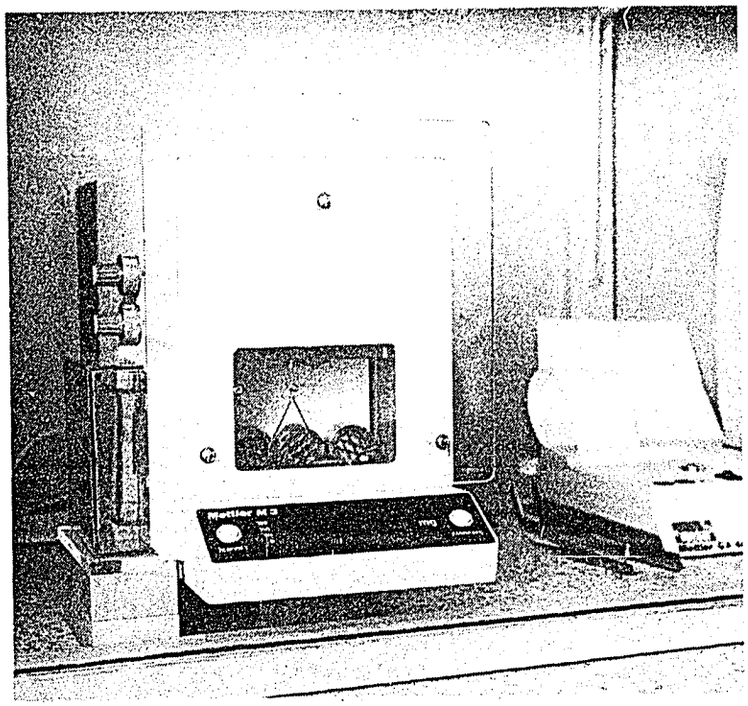


Figure 10

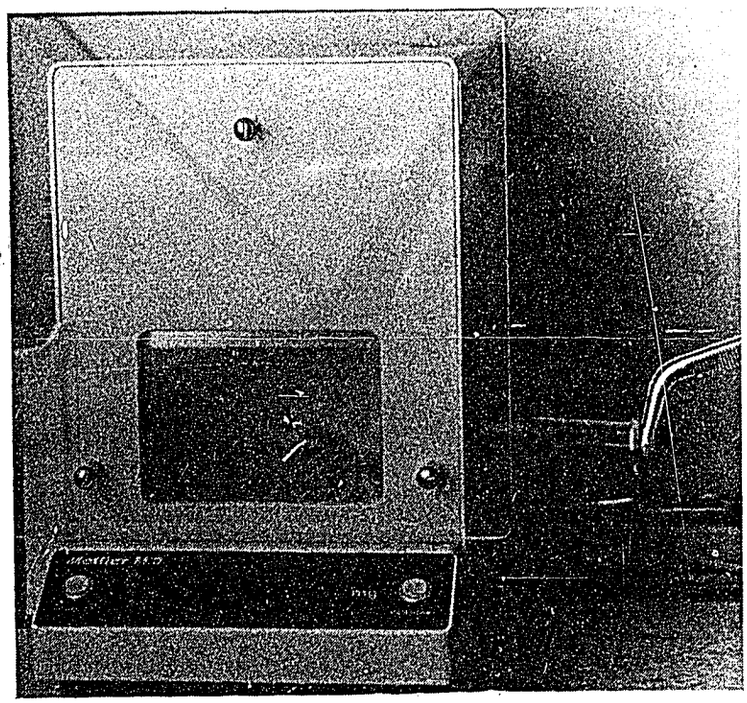


Figure 11

static electricity. To adjust the zero, the balance is released by rotating the lever on the right-hand side of the balance case from position "O" to position "1". To stop movement of the pan suspension (indicated by a red light), the "Panbrake" button is pressed. If the front "electrical" scale is not reading 0.000 mg, the "Zeroset" button is pressed.

To check the calibration of the balance, the lower knob on the left-hand side of the balance case (**figure 10**) is rotated to bring the letter "C" to the front position, and the "Panbrake" button is pressed to damp the ensuing vibrations. If the "electrical" scale does not show 100.000 mg, the balance is recalibrated as follows. The lower left-hand knob is returned to the "O" position, and the "Zeroset" button pressed if the "electrical" scale does not show 0.000 mg. A small screw-driver is then used to press the small, recessed button on the right-hand side of the front console. If "CAL Err" appears on the scale, indicating lack of stability, ensure that the scale reads zero, and that the red light is out before again pressing the recessed button. When "CAL" appears on the scale, turn the lower left-hand knob to show "C" and press "Panbrake". The scale will then show 100.000 mg. The lower left-hand knob is then turned back to zero. After noting that the scale is again reading 0.000 mg, the balance is arrested by rotating the right-hand lever to position "O". It should be noted that after the balance case has been opened and closed, a few minutes must elapse before adjusting the zero or checking the calibration, to avoid undue drift in readings because of air currents and temperature gradients.

The zero-adjustment wire is removed from the stirrup. To place the syringe on the stirrup for the initial weighing, the plunger is gripped by the forceps, the syringe is withdrawn to the right (**figure 11**), then moved to the left and gently lowered on to the stirrup. The balance is released, tare weights are mechanically loaded on to the beam by turning the two left-hand knobs until a reading appears on the front "electrical" scale. The maximum reading is 192 mg, which is the "electrical" weighing range of the balance. After a few minutes, the scale reading is recorded by pressing the "PRT" button on the Mettler GA 40 printer. The balance is arrested and the two left-hand knobs are returned to their zero positions.

The syringe is removed from the stirrup and lowered into the pycnometer holder (**figure 12**), first making sure that the screw of the holder is sufficiently wound back. The deposition is made by winding the screw down to press the plunger. The screw is then wound back, the plunger (**figure 34**) is withdrawn with thumb and finger to bring the meniscus of the solution back to the wide end of the glass needle, and the tip of the needle is examined to see if a droplet of solution has been left on the outside surface. The syringe is again placed on the floor of the weighing chamber in front of the static eliminator discs, the zero-adjustment wire is placed on the stirrup, and the door is closed.

After a few minutes, the balance is released. If the "electrical" scale reading is not zero, the "Zeroset" button is pushed. The balance is arrested, the zero-adjustment wire is removed from the stirrup, and the syringe is re-weighed with the same mechanical tare weights as before.

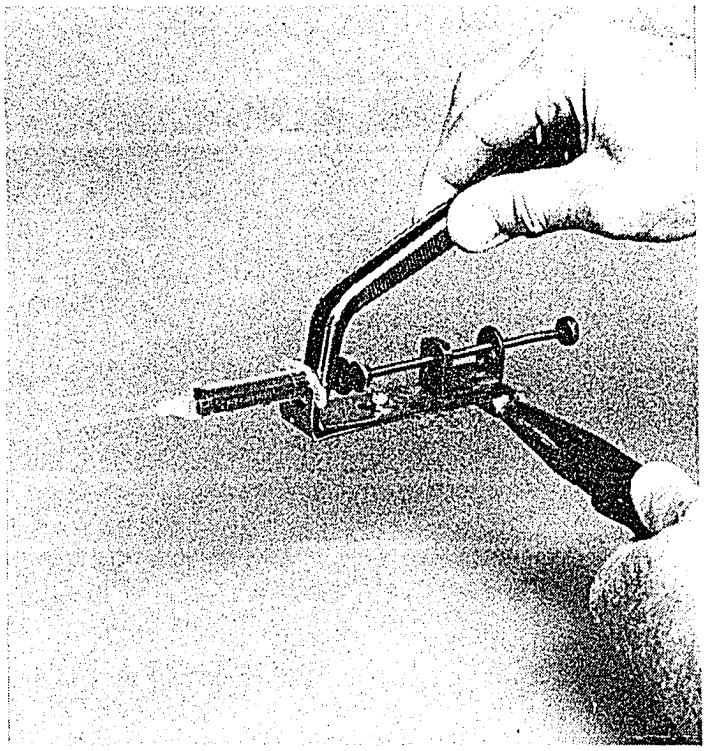


Figure 12

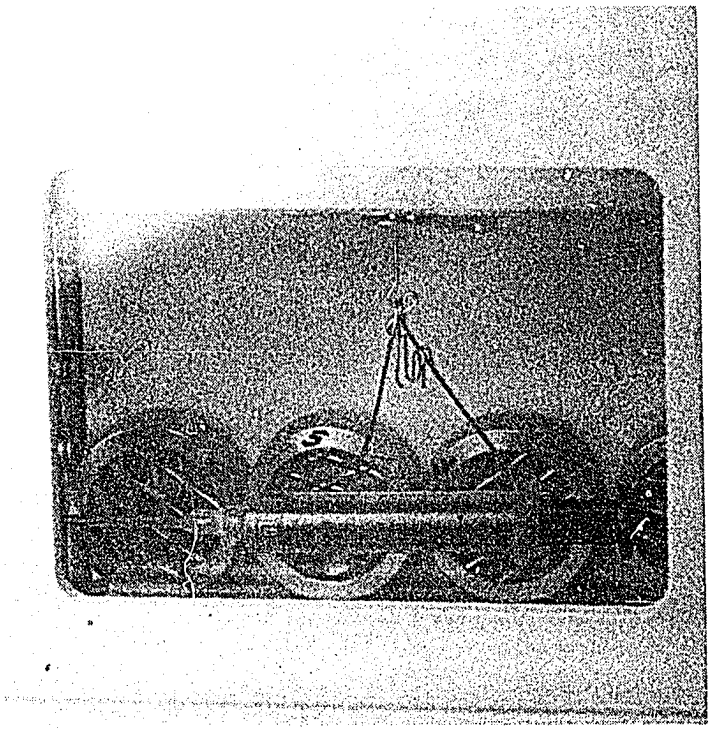


Figure 13

If the above procedure for weighing by difference is followed, the maximum mass of solution which can be deposited is equal to the reading on the "electrical" scale when the syringe is weighed before making the deposition. The mass of the proposed deposition can be increased to the maximum of 192 mg by the following method. The "electrical" scale reading is increased before deposition by adding one or more tare weights to the hook from which the stirrup and syringe are suspended. These weights, which have been made by bending pieces of Nichrome wire, are removed for the zero to be checked after the first weighing, and replaced before the syringe is re-weighed after the deposition.

A deposition of more than 192 mg can be measured with lower accuracy by using one of two tare weights whose masses, measured on the M3 balance, are 119.958 and 184.160 mg, respectively. After the deposition has been made, the syringe is re-weighed with one of the tare weights suspended from the pan hook (**figure 13**) to give a positive reading on the "electrical" scale.

The initial mass of the pycnometer = mechanical tare + initial scale reading + mass of the zero-adjustment wire. The final mass of the pycnometer + mass of wire tare = mechanical tare + final scale reading + mass of the zero-adjustment wire. Therefore the mass of solution deposited = initial mass of pycnometer - final mass of pycnometer = initial scale reading - final scale reading + mass of wire tare.

4. 4 π COUNTING SOURCES

4 π counting sources are used in the primary (*i.e.* absolute) standardisation of a radioactive solution. Standardisation is the term used to describe the determination of the radioactivity concentration of a solution. The unit of radioactivity concentration is becquerel per milligram (Bq mg^{-1}).

A 4 π counting source consists of a radioactive deposit of very low mass spread as uniformly as possible over a circle of 6 to 9 mm diameter on very thin metallised plastic film which is supported by a 25.4 mm i.d. x 34.9 mm o.d. brass ring (**figure 35**). VYNS plastic film is used, the upper surface of which has been coated with an alloy of 80 per cent gold and 20 per cent palladium [Johnson 1985]. The film is prepared from VYNS resin [NCRP 1985] supplied by Union Carbide Australia Limited, Sydney.

To prepare a source mount, it is first advisable to file off the sharp points on the rings (**figure 14**) and widen the gaps slightly preparatory to flattening the rings on the anvil in the device shown in **figure 17**. A brass ring is placed on the anvil, as shown in **figure 15**, and the cylinder with its locating pin correctly oriented is lowered into position, followed by the piston (**figure 16**). The ring is then flattened by hammering the top of the piston (**figure 17**).

The top of the flattened ring is painted with the VYNS solution (**figure 18**) used in the preparation of VYNS foils. After drying, two rings are placed close together on the piece of plate-glass shown in

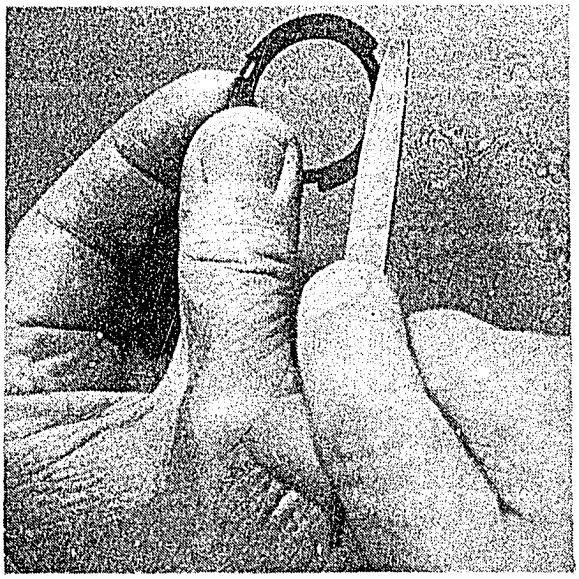


Figure 14

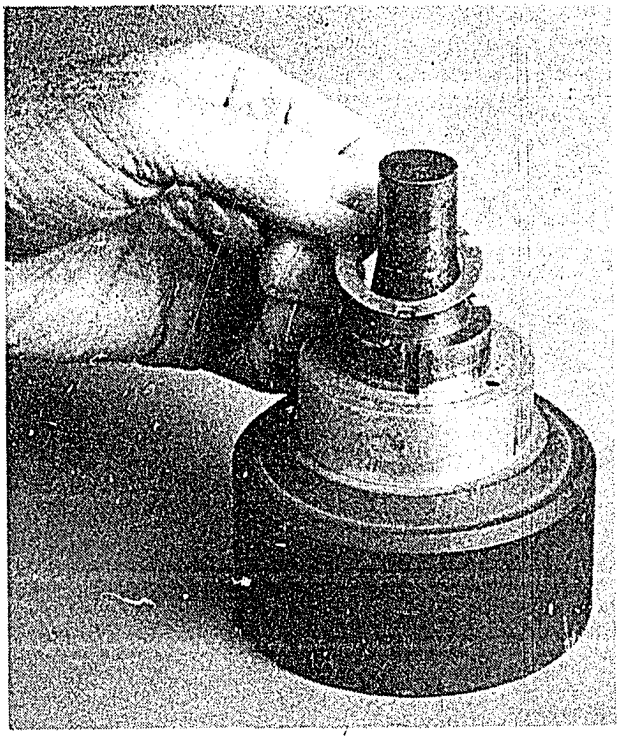


Figure 15

Figure 16

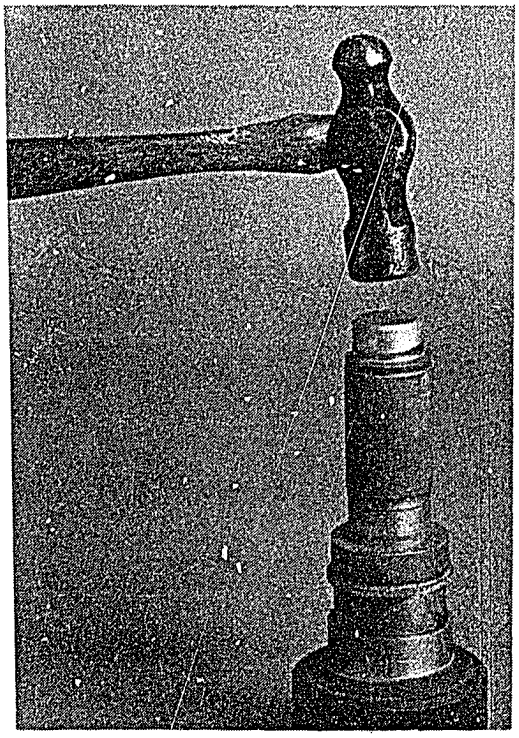


Figure 17

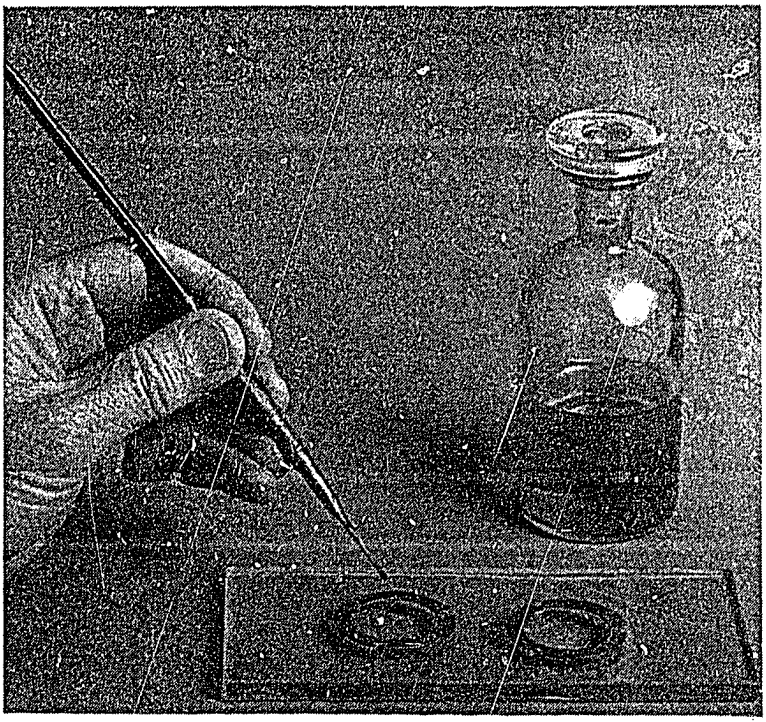


Figure 18

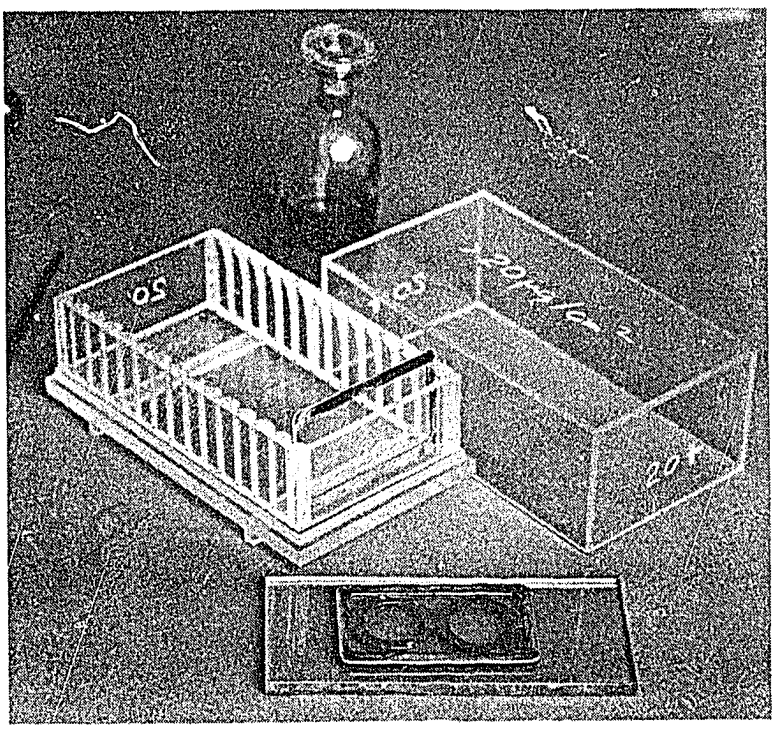


Figure 19

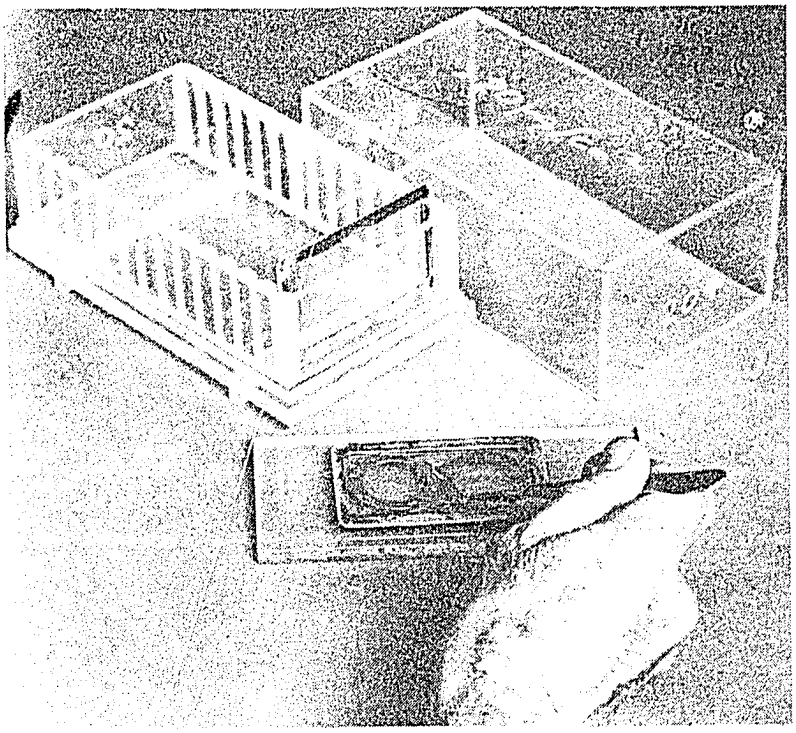


Figure 20

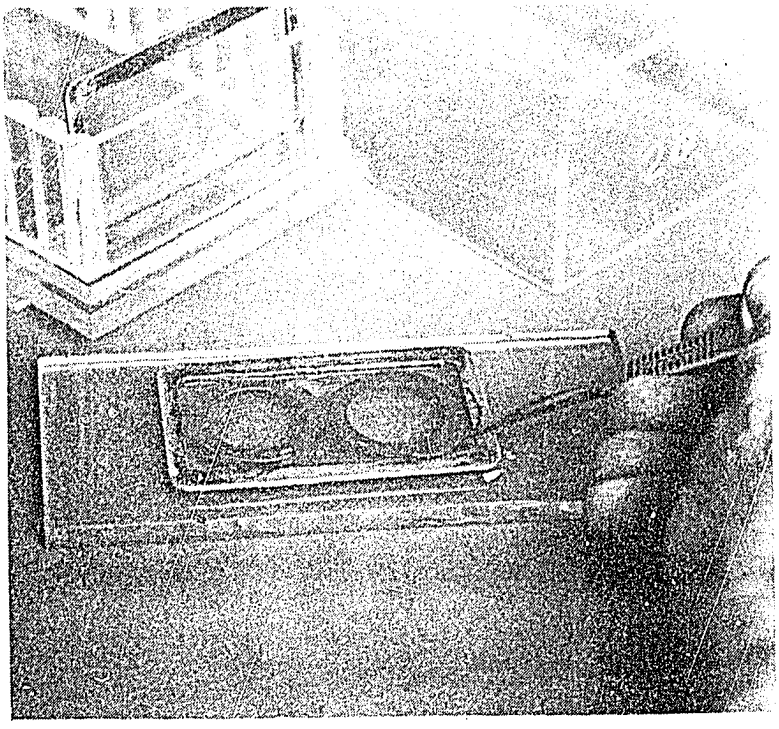


Figure 21

figure 19. A rectangular gold/palladium-coated VYNS foil from the storage box (**figure 19**) is carefully lowered, gold/palladium side uppermost, on to the two rings. Adhesion of the foil to the rings can be ensured by breathing on to the surface of the foil; the layer of condensed moisture hastens the settling of the foil on to the painted brass. The foil is cut away from the edge of the rings by means of a scalpel which has a little VYNS solution adhering to its point (**figures 20** and **21**). The cutting should be carried out well away from the edge of the rings wherever possible.

The edges of the rings are scraped in three places (**figure 22**) and silver paint is applied to the bare metal and the edge of the foil (**figure 23**). This ensures that there is electrical connection between the gold/palladium and the brass. When the paint has dried the counting-source mount is ready for the accurate deposition of a drop of solution whose radioactivity concentration is to be determined. The method of deposition is described in **section 7.2**; the source mounts are stored in boxes described in **section 7.2.3**.

5. 4 π CALIBRATION SOURCES

A 4 π calibration source is more robust than a 4 π counting source, but less efficient. The calibration source consists of a radioactive deposit on Mylar film which is glued to a flat aluminium ring. The source mount is shown in **figure 30**. The radioactivity is protected by superimposing a similar foil and ring, and crimping the two rings together.

To prepare two such sources, the sharp edges are filed from four of the flat aluminium rings (**figure 24**). The two Perspex rings shown in **figure 25** are used to stretch a piece of Mylar film, as shown in **figure 26**. The inner surface of the larger ring and the outer surface of the smaller ring are conical, the angle of the cone being quite small. The Mylar film is laid on the smaller ring and the larger ring is then pushed down over them. The distance between the two conical surfaces is sufficient to allow the film to be stretched tightly. The nominal thickness of the film is 0.0127 mm. The roll of film is always stood with its cylindrical surface vertical to avoid puncturing.

The four aluminium rings are coated on one side with an epoxy adhesive, such as Selley's "Super Strength", and dropped sticky-side down on to the stretched Mylar film (**figure 26**). On the next day, the rings are separated from one another with a scalpel. This has to be done with care because the adhesion of the film to the hardened adhesive is not great. The excess film is removed from each ring by means of a file (**figure 27**). The edge of the ring is filed at the angle shown in order to reduce the possibility of the edge of the film being accidentally prised away from the adhesive. After the required mass of radioactive solution has been deposited on the source mount and dried, as described in **section 7.1**, the source mount is lowered, active face uppermost, into an aluminium ring (of the type shown on the left-hand side of **figure 45**) which has an L-shaped cross-section. The radioactive residue is protected by lowering on to it, as shown in **figure 28**, another Mylar ring, this time with the film bottom-most. The outer ring is then crimped, as described in **section 8.2**, to give the finished source.

Figure 22

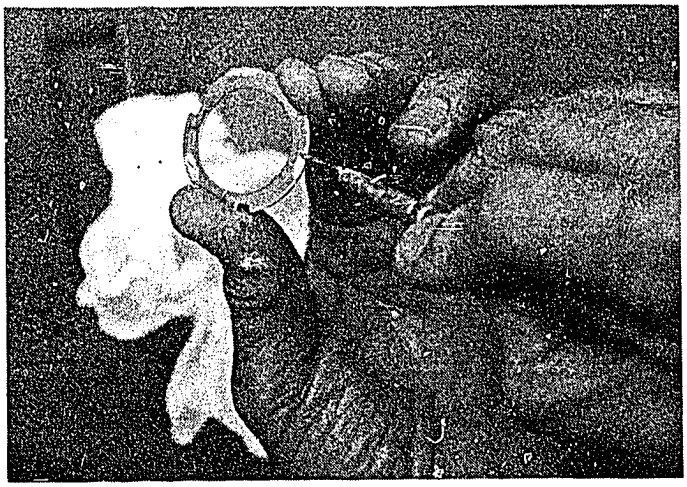


Figure 23

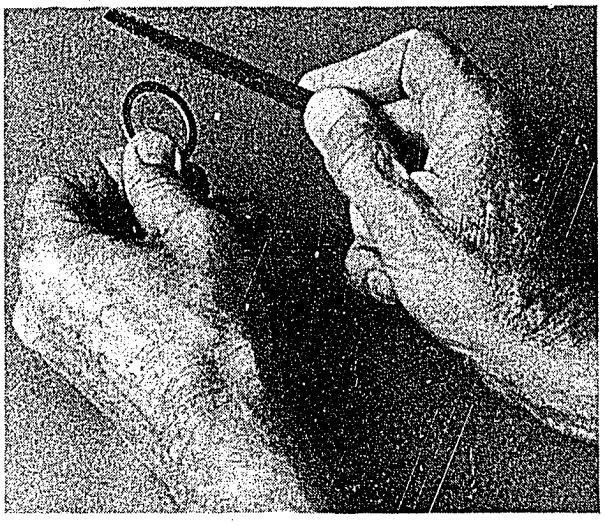


Figure 24

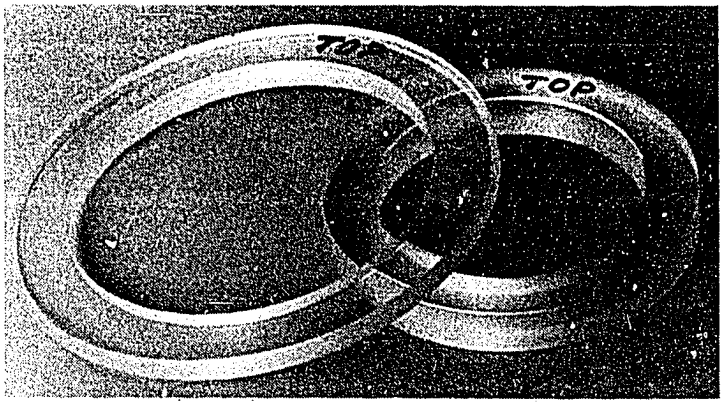


Figure 25

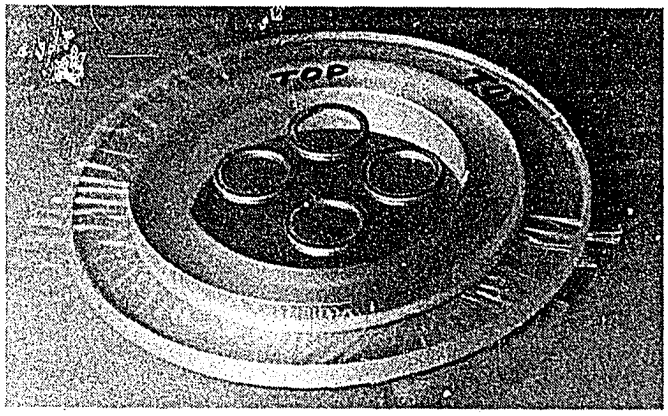


Figure 26

Figure 27

Figure 28

6. 4 π COUNTING SOURCES ON MYLAR FILM

If a counting source of low efficiency is required, the radioactive solution is deposited on Mylar film instead of a VYNS foil. Brass rings of the type described in **section 4** are glued to Mylar film in the manner described in **section 5**. The Mylar source mounts are then coated on their upper surfaces with gold/palladium alloy, using the apparatus and method described by Johnson [1985].

Two of the uncoated Mylar source mounts are attached to the brass holder shown on the left-hand side of **figure 29**. This holder has the same outer dimensions as the aluminium frame of the rectangular VYNS foil shown on the right-hand side of **figure 29**. The holder thus fits into the vacuum-coating equipment used for metallising the VYNS foils.

7. DEPOSITION OF RADIOACTIVE SOLUTIONS

7.1 Deposition on a 4 π Calibration Source Mount

Sources of improved efficiency are prepared by the addition of Catanac solution. The residue produced by evaporating the deposited radioactive solution will then be spread out in a thinner layer and there will be less self-absorption in the source. The Catanac solution is prepared by dissolving Catanac SN spreading agent (supplied by American Cyanamid Co, Bound Brook, New Jersey) in ethanol to give a concentration of 0.01 g mL^{-1}, and then diluting with distilled water to give a concentration of 500 μg mL^{-1}. A drop of the dilute solution is deposited at the centre of the source mount (**figure 30**) and evaporated to dryness in an oven at 50°C. The residue left after the evaporation is shown in **figure 31**. A known mass of a standardised radioactive solution is deposited from the syringe pycnometer on to the Catanac residue, as shown in **figure 32**.

To deposit a pre-determined mass of solution, the source mount is usually placed on the pan of the semi-microbalance (**figure 33** shows a counting-source mount and its storage box on the pan), and the mass deposited is controlled by watching the optical scale of the balance. After deposition the drop is touched with the tip of the pycnometer needle in order to remove any droplet adhering to the outside surface of the needle. The technique is shown in **figure 32**, where, for the sake of clarity, the source mount is outside the balance. The plunger is then pulled back, as shown in **figure 34**, to withdraw the meniscus to the top of the needle. The measurement of the mass of the deposited drop by electronic microbalance, and other details of the deposition are described in **section 3**.

The drop of radioactive solution is dried in the oven at 50°C. When transporting the source to the oven, care should be taken not to shake the source in order to avoid spreading the drop over too wide an area.

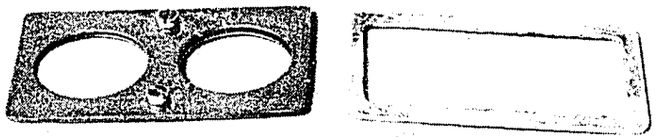


Figure 29

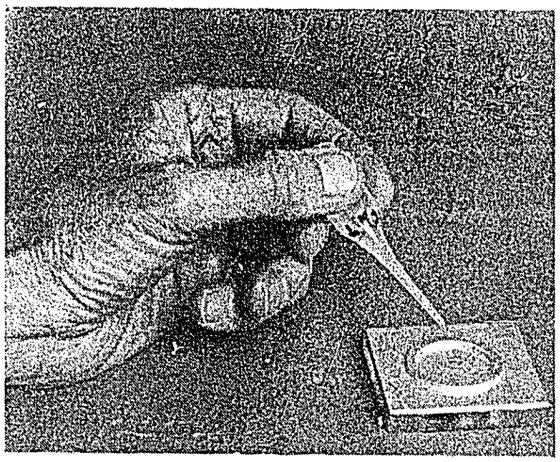


Figure 30

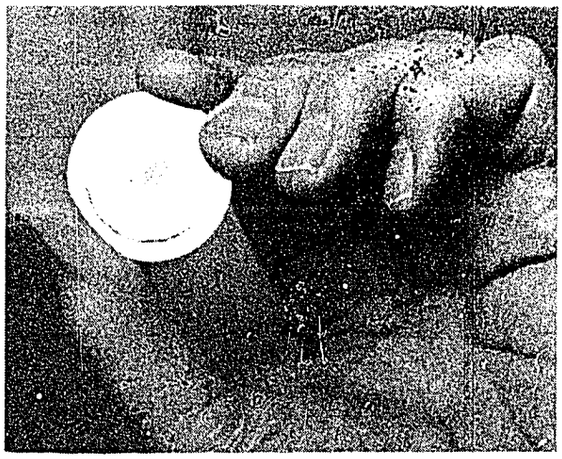


Figure 31

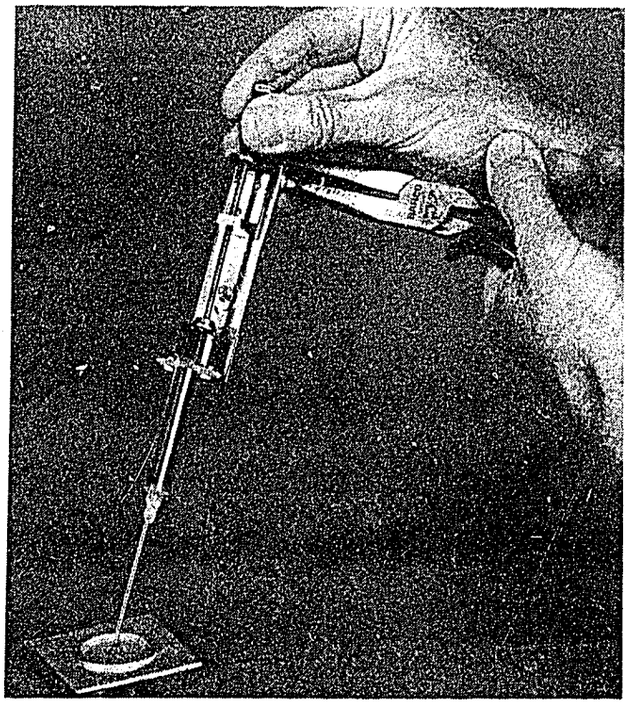


Figure 32

7.2 Deposition on a 4 π Counting Source Mount

7.2.1 Deposition with Catanac

This is carried out in the same way as for 4 π calibration sources. The efficiency of sources made with Catanac is improved if oven-drying is replaced by drying and stirring the drop of solution with a jet of nitrogen [Wyllie *et al.*, 1970].

7.2.2 Deposition on Ion exchange resin

In this method, instead of the radioactive solution being deposited on a Catanac residue, it is deposited on a very thin, electrosprayed layer of ion exchange resin. The method of electrospraying the resin on to the gold/palladium-coated source mounts is described elsewhere [Wyllie 1988].

7.2.3 Storage of 4 π counting sources

Figures 35 and **36** show how the finished source is stored between a Perspex cover and a square piece of aluminium sheet which are held together by an elastic band. The inner ledge of the Perspex cover ensures that the VYNS film will not be damaged if the storage box is inverted. On the Perspex and aluminium are inscribed the nuclide and source number; on the aluminium are also inscribed the thickness ($\mu g\ cm^{-2}$) of the VYNS and gold/palladium layers, and an indication as to whether the metal layer faces up or down.

7.3 Deposition in an Ampoule

Figure 37 shows how radioactive solution is deposited in an International Standard Ampoule by means of a syringe pycnometer. This method is employed when the mass deposited is of the order of one or two hundred milligrams. The diluting acid is then added by means of a syringe with a silicone-treated glass needle [Wyllie 1986], the syringe being held in the Lowenthal-Page holder (**figure 5**). The mass of the diluted solution is obtained by weighing the ampoule on the semi-microbalance before and after filling. The mass of the empty ampoule includes the masses of the rubber stopper and the plastic stand.

7.4 Deposition in a Vial

A vial with a rubber stopper held down by a metal cap (**figure 38**) is suitable for the short-term storage of a standardised solution. Over a short period there is negligible change in concentration owing to leakage of water vapour from between the stopper and rim of the vial. The leakage rate is about 5 $\mu g\ day^{-1}$ for Teflon-faced, butyl-rubber stoppers (20 mm Wheaton, No. 224168, supplied by Edwards Instrument Company, Narellan, NSW). After the radioactive solution is deposited in a vial (**figure 39**), it is touched momentarily with the tip of the glass needle to ensure that no droplet will remain on the outer surface of the needle.

The stoppered vial is capped as shown in **figures 40** and **41**. A polythene ampoule pycnometer can be used to transfer radioactive solution to a vial when a syringe pycnometer is not required for

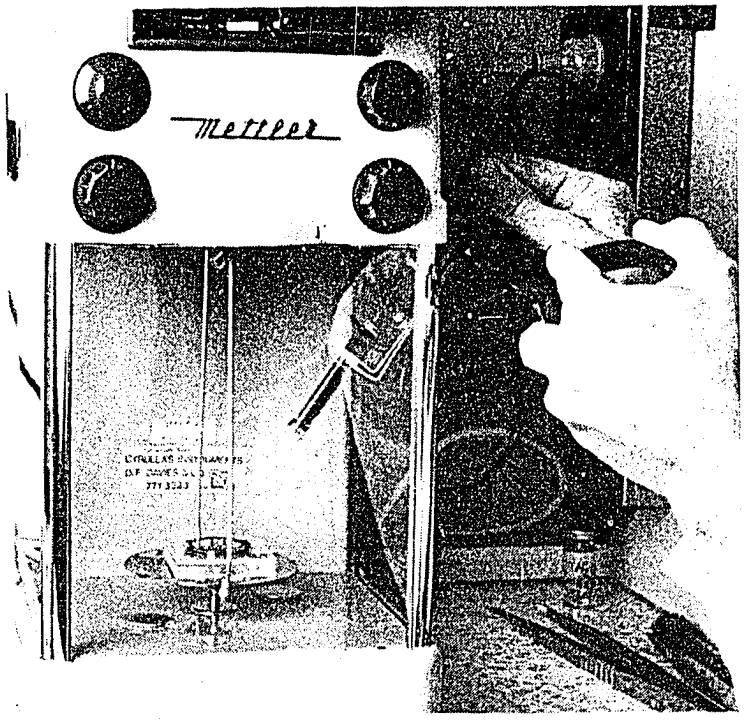


Figure 33

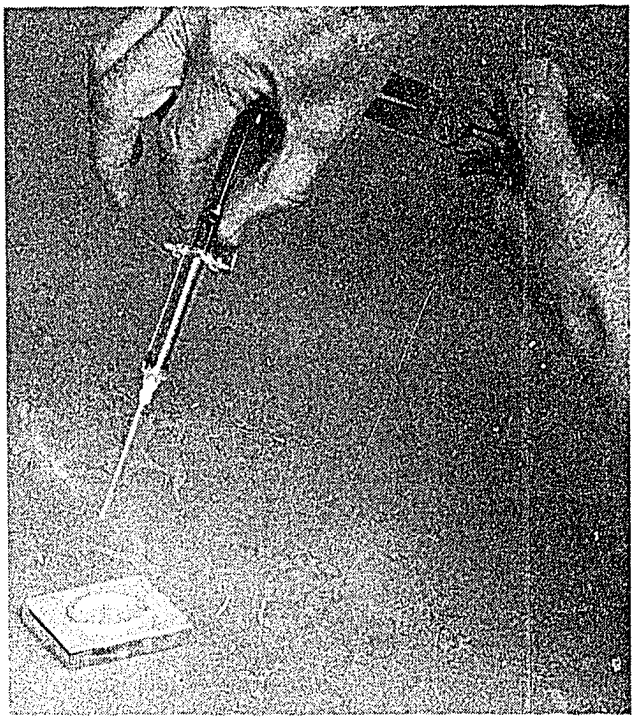


Figure 34

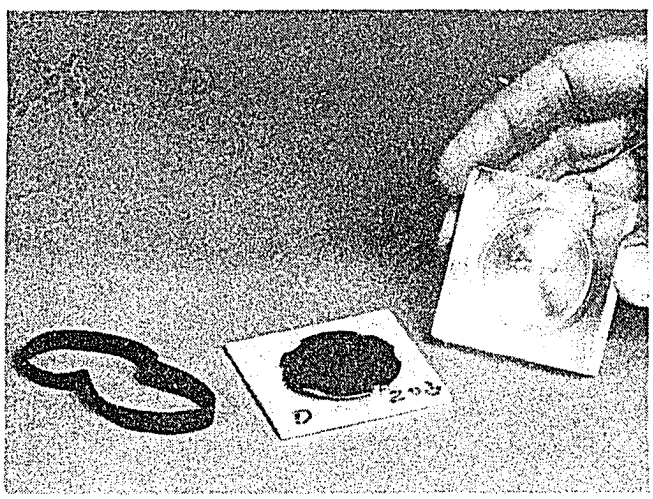


Figure 35

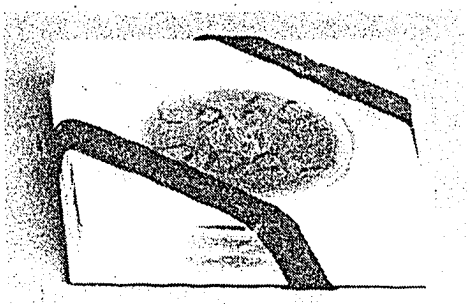


Figure 36

the accurate dispensing of small amounts of the solution. When a polythene ampoule pycnometer is used, the mass of solution in the vial is measured by weighing the vial on the semi-microbalance. The manufacture of polythene pycnometers is described in NCRP Report No. 58 [NCRP 1985 : 229].

8. 2 π γ CALIBRATION SOURCES

8.1 Deposition and Assembly

A 2 π γ source mount consists of a circular disc of 1.59 mm thick polythene cut out of a larger sheet by means of a 32 mm wad punch. The centre of the disc is marked with a felt pen. The tip of the pen is located by placing the disc in an aluminium ring containing a similar disc with a hole in the centre (**figure 42**); the mark is then made through the hole in the latter disc (**figure 43**). The unmarked face is painted with VYNS solution (**figure 44**). Using the centrally-marked dot as an aiming point, the radioactive deposition is made as described in **section 7.1**. Generally, 2 π γ sources are used as point sources, so Catanac spreading agent is not used. After drying the deposited solution in the oven at 50°C, the source is covered with a VYNS foil.

Two 2 π γ sources may be covered simultaneously with one rectangular foil, using the procedure illustrated in **figure 19**. The VYNS film beyond the edges of the discs is cut away with a scalpel which has been dipped into VYNS solution (**figures 20** and **21**).

Figure 45 shows the components of a 2 π γ source before they are finally assembled. Into the aluminium ring are inserted, in the following order, a piece of heat-shrinkable plastic film, the polythene disc with its radioactive face downwards (**figure 46**), a paper disc with the source number and nuclide symbol printed on its upper face, and a blank polythene disc (**figure 47**). After pressing these components into place, the heat-shrinkable film above the rim of the aluminium ring is cut off with scissors (**figure 48**). *Note*: The roll of heat-shrinkable plastic should be stood with its axis vertical to prevent damage to the film.

8.2 Operation of the Crimping Machine

Figure 49 shows the two parts of the machine which is used to crimp together the assembled components of the source. **Figure 50** shows the assembled components being put into place. The aluminium ring must be pressed firmly into the depression to avoid the possibility of it moving sideways and getting mangled during the crimping operation. The top part of the machine is put into place and the slide within it is moved to its central position (**figure 51**). The top part is then wound down as far as it will go. This bends the top edge of the aluminium ring inwards. The top part of the machine is then wound up a few turns and the slide is moved to its open position to check that the first operation has been successful. The slide is then moved to the opposite position (**figure 52**). The top part is wound down to flatten the rim of the source completely, and then wound back and removed. To release the source, it is generally necessary to give the release lever a light tap with a hammer (**figure 53**) while holding the source with the other hand to prevent it from jumping out. The

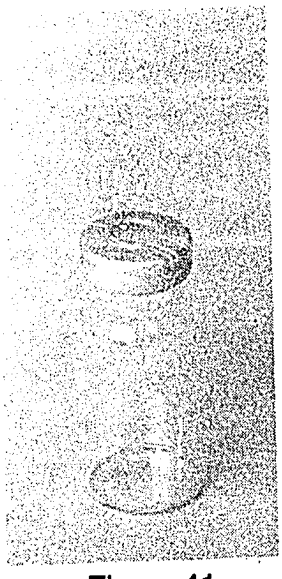


Figure 41

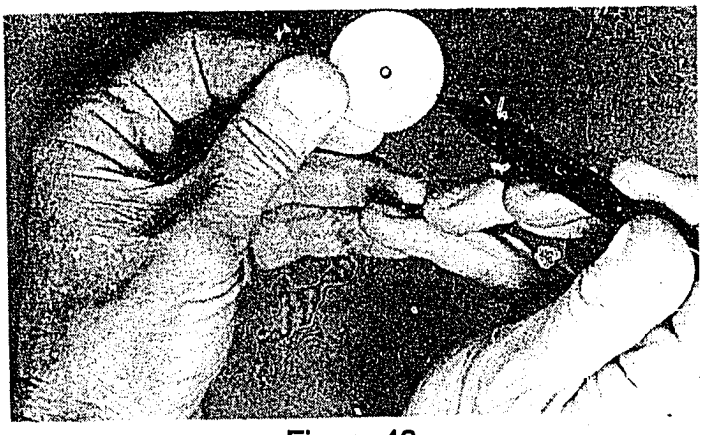


Figure 43

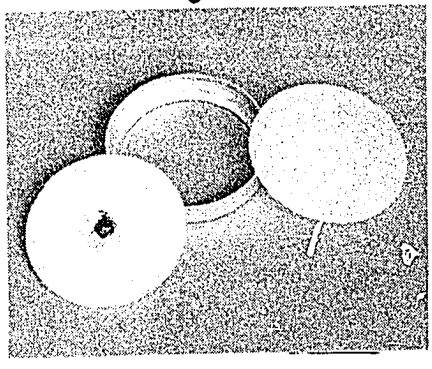


Figure 42

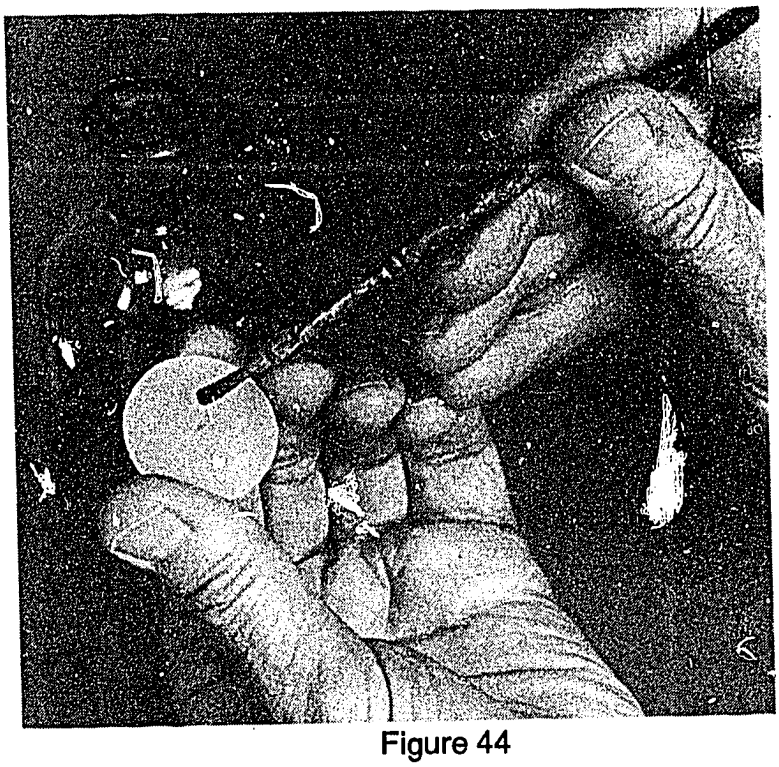


Figure 44

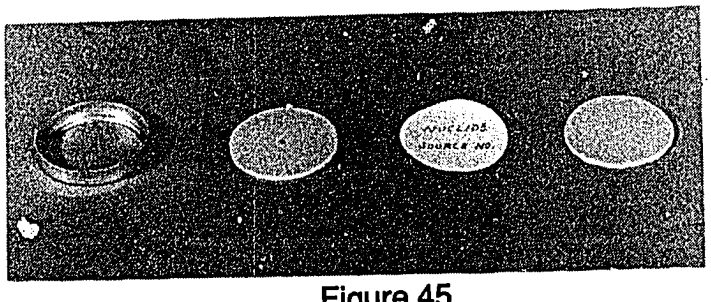


Figure 45

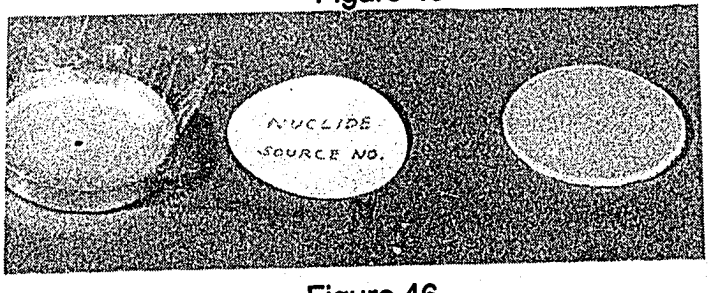


Figure 46

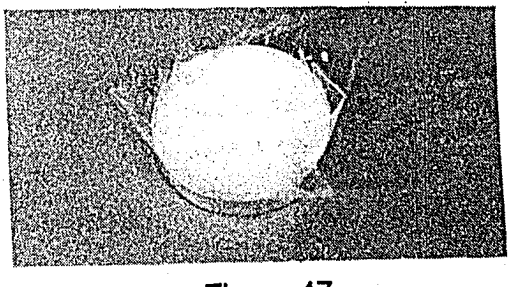


Figure 47

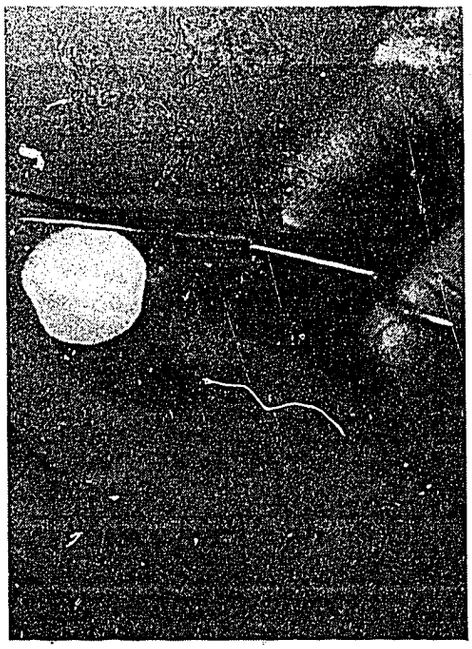


Figure 48

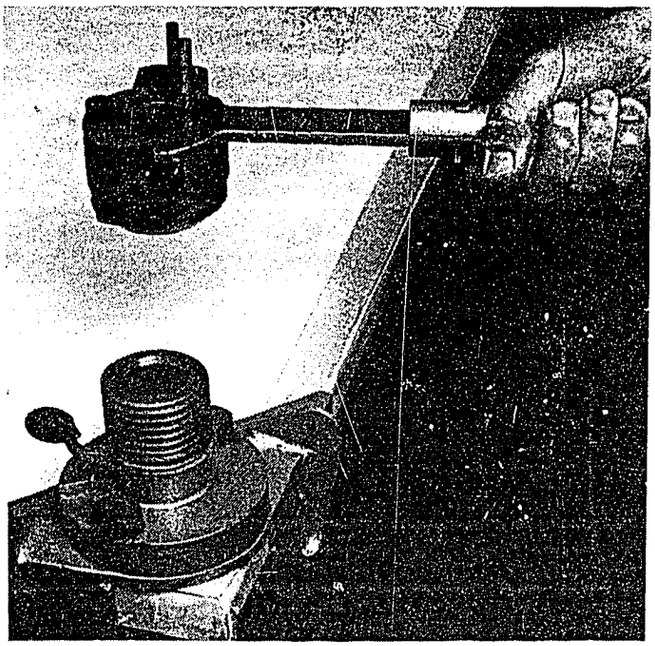


Figure 49

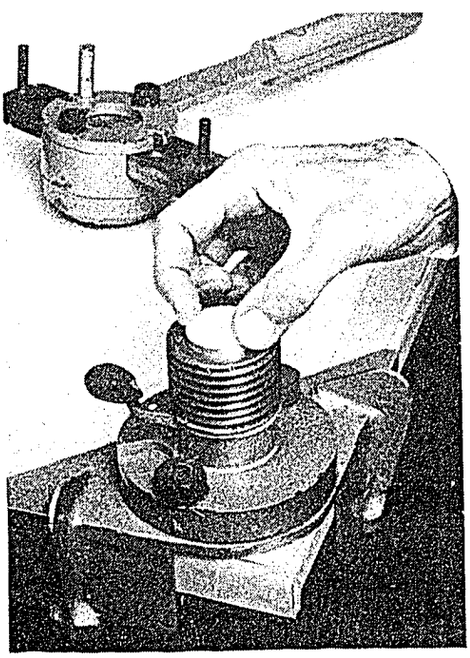


Figure 50

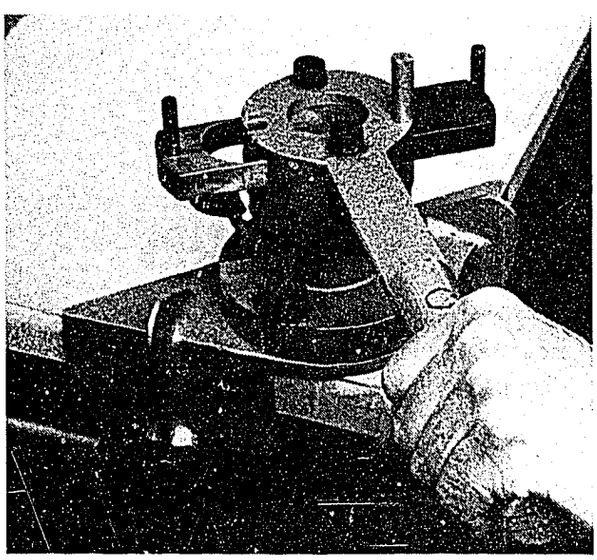


Figure 51

remaining fringe of heat-shrinkable film is trimmed off with a scalpel.

8.3 Heat Treatment

The heat-shrinkable plastic is shrunk by holding it in a stream of hot air from a hot air "gun" (**figure 54**), or under an infra-red lamp. Care must be taken not to overheat, as this will make the source bulge. **Figure 55** shows a completed source. **Figure 56** gives a cross-sectional view of a source with some of the vertical dimensions exaggerated.

8.4 2 π γ Source with a Stronger Cover

A 2 π γ source with a stronger cover is prepared by substituting a polythene disc for the heat-shrinkable plastic film. After covering the radioactive residue with a VYNS foil as described in **section 8.1**, the disc bearing the residue is lowered into the aluminium ring with the radioactive face uppermost. A blank polythene disc is lowered over the radioactive disc, and the assembly is crimped together as described in **section 8.2**.

9. 2 π α AND 2 π β CALIBRATION SOURCES

2 π α and 2 π β sources require a protective covering film of minimum thickness in order that self-absorption be kept as low as possible. A further requirement is sufficient backing for saturation back-scatter. The components for these sources consist of a 4 π source on VYNS, made with electrosprayed ion-exchange resin or with Catanac, a stainless steel disc of 24 mm diameter and 3 mm thickness (**figure 57**), and a protective aluminium ring. The back of the stainless steel disc is engraved with the source number and nuclide symbol. The front is painted with VYNS solution (**figure 58**). After the solvent has evaporated, the 4 π source is lowered, radioactive side uppermost, on to the painted surface of the disc (**figure 59**). The ring is pushed down to break it free from the source (**figure 60**). The source is then generally protected by lowering on to it a VYNS foil. A foil coated on its upper surface with gold/palladium is used if the 2 π count rate of the finished source is to be determined in the gas counter. Half of a rectangular foil can be used if it is first cut across with a scalpel dipped in solvent (cyclohexanone). This is done by lightly touching the film at a series of points with the tip of the scalpel. To ensure that the gold/palladium is earthed, it is scraped at three points on the edge of the disc, which are then touched with silver paint.

The final assembly consists of inserting the source into the protective aluminium ring and bending the lugs of the ring (**figure 61**) over the back edge of the disc with a screw-driver. A top view of the assembly is shown in **figure 62**.

10. ULTRA-THIN SOURCES

Ultra-thin sources [Wyllie and Lowenthal 1984] are used in α and β spectrometry, where the α or β particles should emerge from the sources with negligible loss of energy. As a supplement to the description given in the above reference, **figures 63** and **64** illustrate the source washing procedure

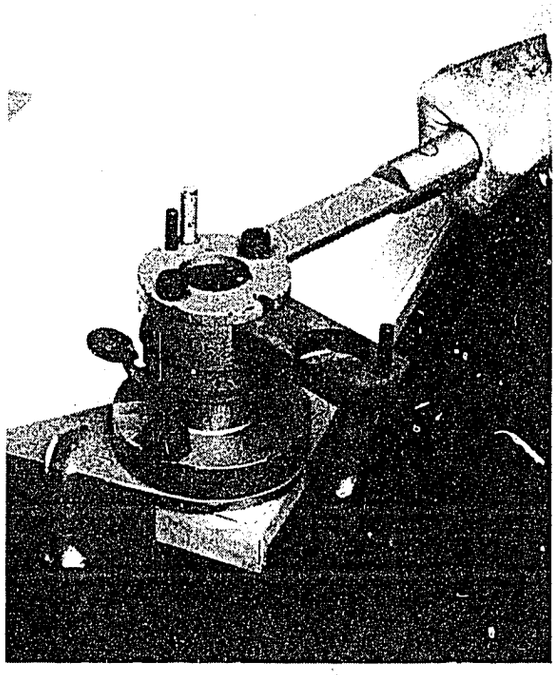


Figure 52

Figure 53

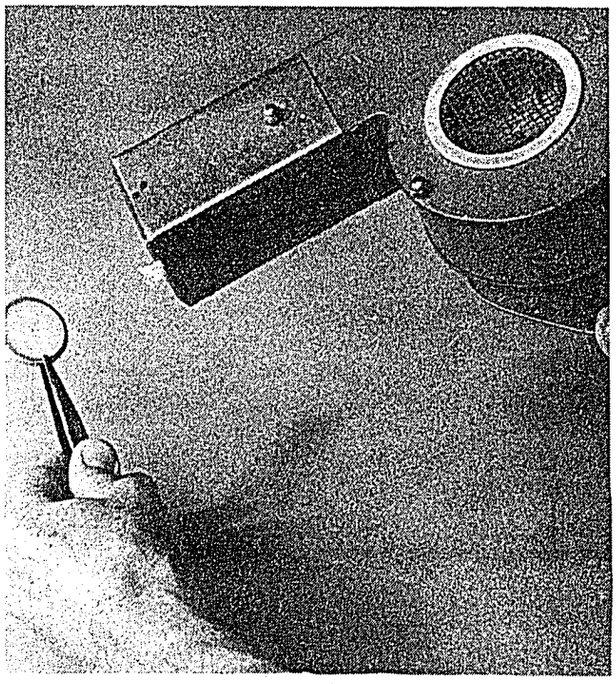


Figure 54

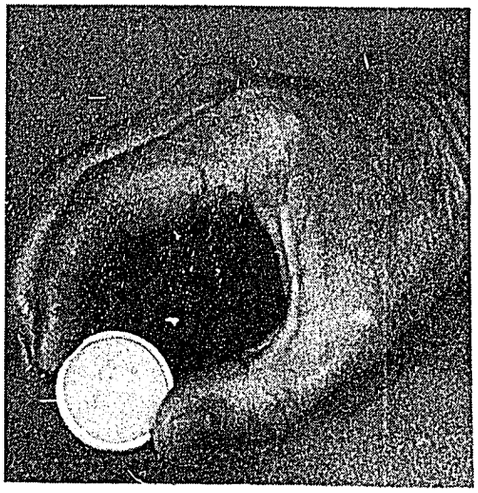
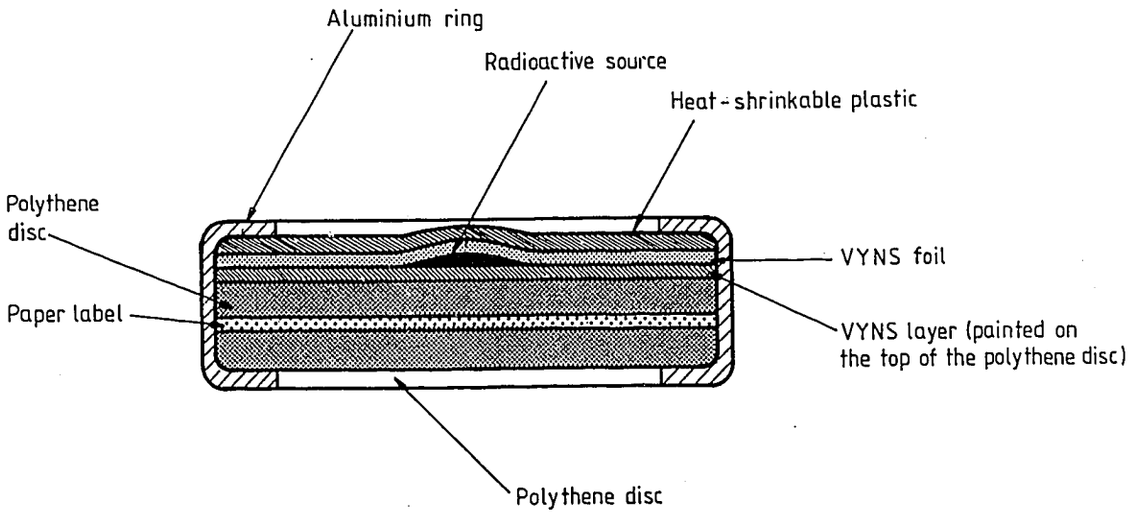


Figure 55



2 π γ SOURCE

Figure 56

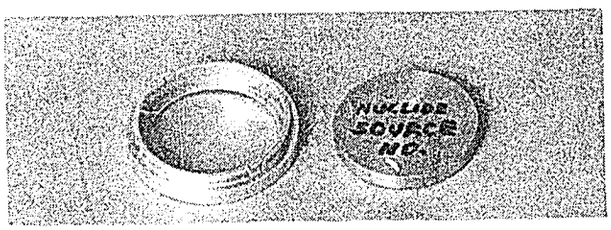


Figure 57

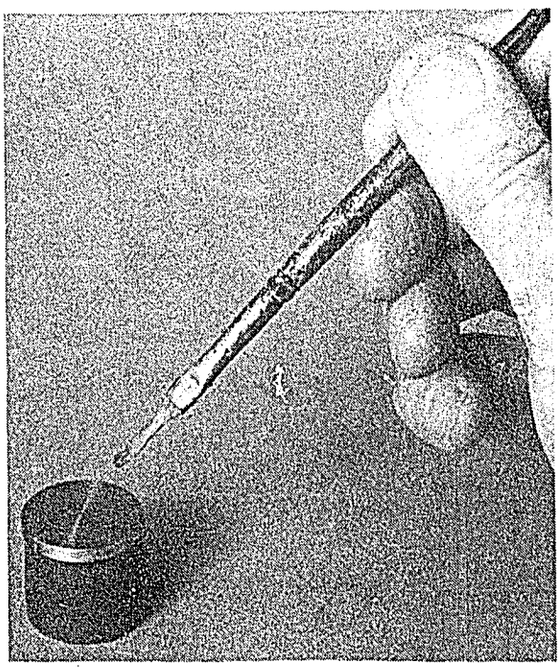


Figure 58

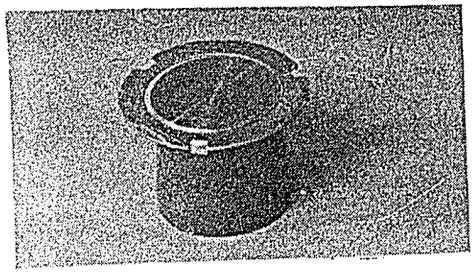


Figure 59

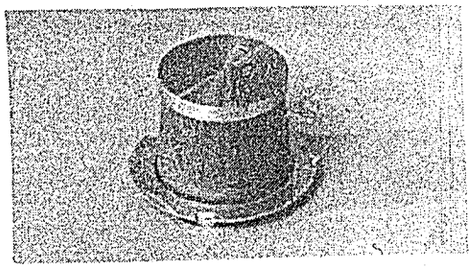


Figure 60

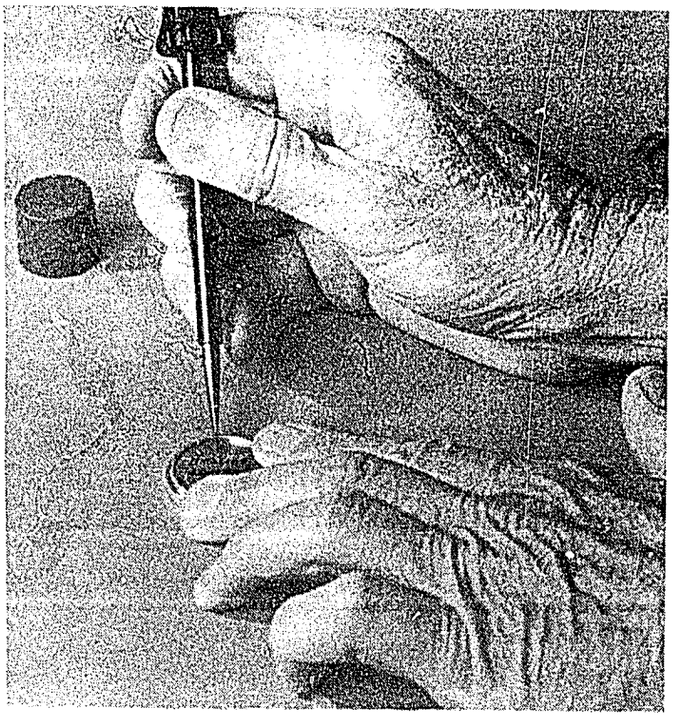


Figure 61

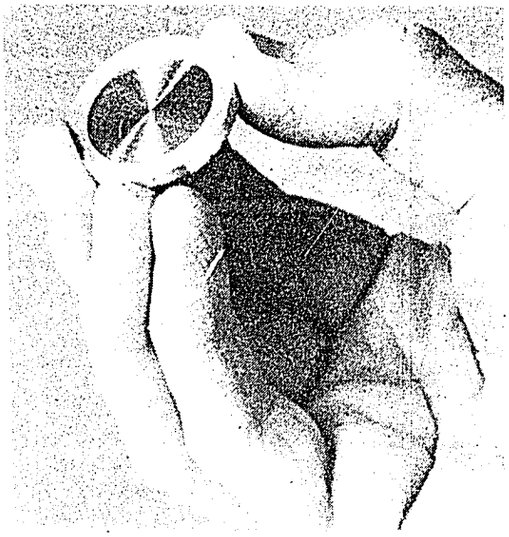


Figure 62

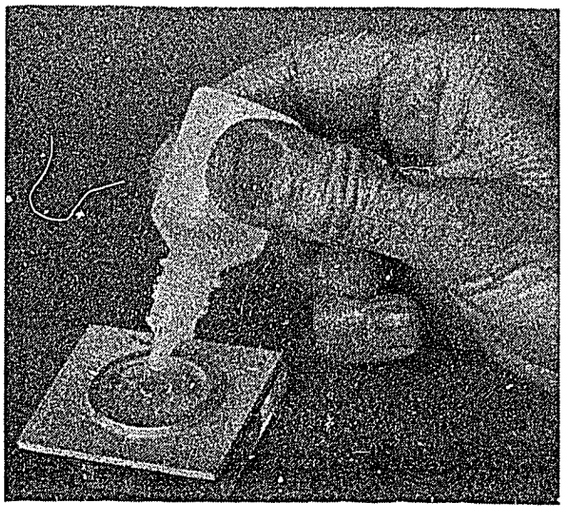


Figure 63

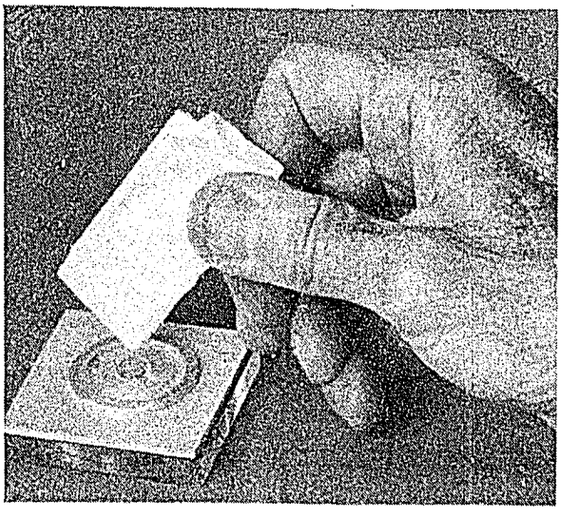


Figure 64

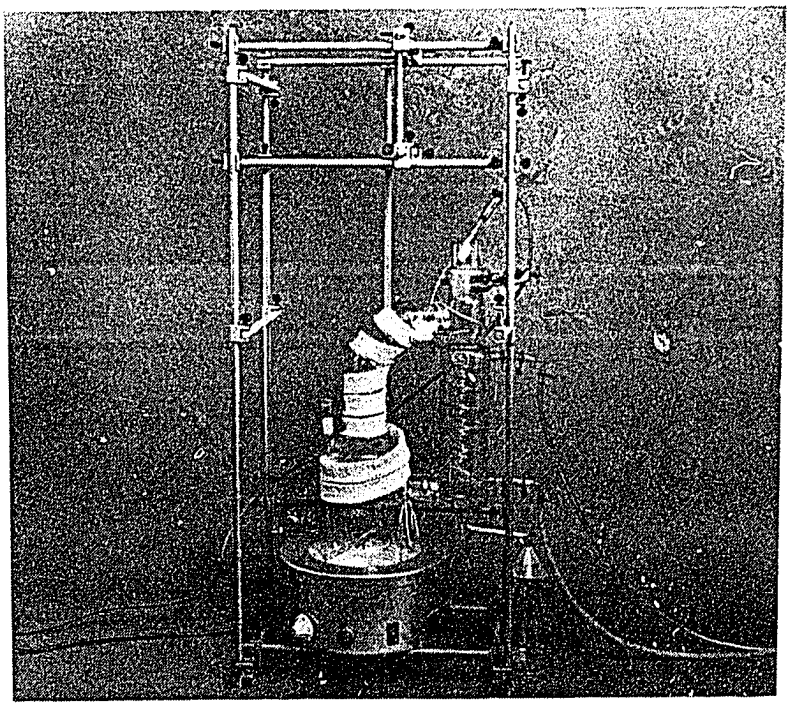


Figure 65

in which distilled water is deposited on to the adsorbed radioactivity from a polythene dropping bottle, and then largely removed by holding a folded cellulose tissue a millimetre or so above the surface of the source mount. The washing procedure removes all soluble material, leaving only the adsorbed hydroxide.

11. SUB-BOILING STILL

The highest-efficiency counting sources are prepared on electrosprayed ion-exchange resin. The source efficiency can be improved by reducing the total dissolved solids in the radioactive solution from which the source is prepared. This can be done by using the best distilled water for diluting the hydrochloric acid which is used for the dilution of the original radioactive solution. The best water is obtained from the sub-boiling still (**figure 65**), which is operated at about 94°C. The operating procedure is given elsewhere [Wyllie 1988].

12. ACKNOWLEDGEMENTS

Some of the procedures discussed were devised by former members of this laboratory, notably, G.C. Lowenthal, V. Page, and E.P. Johnson. The author wishes to thank Mr E.P. Johnson for helpful discussions, and Mr D. Elkins for organising the photography of the equipment and procedures.

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