2) After the end of the irradiation transfer the zinc into a quartz distillation apparatus together with 50 mg of high-purity tin metal*. See figure.
3) Evacuate, place furnace in position and raise temperature to about 800° C.
4) As soon as all the zinc has collected in the cooler parts of the quartz tube remove furnace. Note 1.
5) After cooling disconnect the distillation apparatus from the vacuum line and cut off quartz bulb at A.
6) Introduce 1.5 ml 6 M HCl into quartz bulb followed by approximately 0.1 ml conc. HNO_3. Place quartz bulb inside a 10 ml beaker and put on hot plate. Note 2.
7) After all the tin has been dissolved (Note 3) add one more drop HNO_3 and transfer the solution onto the top of an anion-exchange column. (Dowex 1 × 10, 100–200 mesh, 0.6 × 15 cm). Note 4.
8) After passage of the solution wash with 1 ml 6 M HCl.
9) Elute copper with 5 ml 2 M HCl. Start collecting the eluant in a teflon cup after one free column volume has been replaced by the 2 M HCl. Note 5.
10) Add one drop of 60% $HClO_4$ to the eluant and evaporate to dryness in a "dust free" evaporation unit [3]. Note 6.
11) Dissolve residue in 0.01 M HCl and adjust to desired volume. Note 7.

Note 1. At the moment when all zinc has been distilled the tin will appear as a dark sponge; a few seconds later it forms a few shiny beads. Any further heating should be avoided.
Note 2. HNO_3 is necessary to dissolve the tin at a reasonable rate. Too much HNO_3 will precipitate hydrated SnO_2.
Note 3. The solution frequently contains a few flakes of a black unidentified material which will be filtered off by the ion-exchange column.
Note 4. Washed with ionfree water, 2 M HCl and finally with 6 M HCl. The change from water to 2 M HCl is used to determine the free column volume.
Note 5. The fractions collected up to this point contain microcurie quantities of ^{65}Ni formed by ^{68}Zn (n, α) ^{65}Ni.
Note 6. When the volume of the solution is down to a few microliters but still contains some HCl a faint yellow coloration of the $CuCl_4^{2-}$ complex will be visible. After complete evaporation a few micrograms of a white flaky material (SiO_2?) will appear.
Note 7. A 24 hr storage in glass shows no loss by absorption at this acidity. The solution can easily be buffered to suit biological applications.

The yield is better than 80% with the main loss occurring during step 9). Complete recovery from the ion-exchange column would require more 2 M HCl which in turn would increase the evaporation time in step 10). The separation can be performed in four to five hours. The radiochemical purity of the final product is at least 99%. The main contaminant which amounts to considerably less than 1% is ^{67}Cu formed by ^{67}Zn (n, p) ^{67}Cu.

A γ-spectrum of the copper taken seven days after its isolation showed only the lines attributable to ^{64}Cu (511 and 1340 KeV) and ^{67}Cu (90 and 180 KeV). Taking into account the decay schemes of the two nuclides and the different detection efficiencies for their respective γ-rays an activity ratio of 1000 for $^{64}Cu/^{67}Cu$ was estimated for this sample at the time of its preparation. The part of the procedure involving the distillation of the target material in the presence of tin as hold-back carrier, is equally efficient for the separation of ^{115}In formed during neutron irradiation of cadmium [^{114}Cd (n, γ, β^-) ^{115}In]. It would appear that this method should be very suitable for the analysis of cadmium metal for trace impurities by neutron activation. In this case the gram quantities of cadmium would be evaporated before the irradiation. The tin which should collect most of the trace impurities can then be irradiated directly in the quartz bulb sealed off from the distillation apparatus. Furthermore, in contrast to conventional methods the danger of an accidental introduction of trace contaminants during the removal of the cadmium prior to the irradiation, should be negligible. Proper choice of the cadmium-tin ratio for the distillation might even render the determination of "blanks" in the high-purity tin superfluous.

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Preparation of Carrier-free Rhodium from Irradiated Palladium

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Carrier-free sources of ^{106}Rh (2.2 hours) were prepared in the following way. Natural palladium was irradiated with 28 MeV deuterons in the Buenos Aires synchrocyclotron. The nuclear reactions which yield ^{106}Rh under our working conditions are:

$$^{106}Pd\ (d, 2p)\ ^{106}Rh$$
$$^{108}Pd\ (d, \alpha)\ ^{106}Rh$$

the relative abundances being 27.33% for ^{106}Pd and 26.71% for ^{108}Pd. Interfering activities of silver, ruthenium and other rhodium nuclides are also produced.

The reported [1, 2, 3, 4] separation methods does not

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result in carrier free activity. Thus the following method was developed:

1) Dissolution of the palladium target.
2) Retention of the palladium on an anion resin. Ruthenium activities are also retained in this step.
3) Repeated coprecipitation of the rhodium activities on $Fe(OH)_3$ in the presence of silver carrier.
4) Elimination of the iron by extraction with ethyl ether.
5) Final purification through a small anion resin column.

This chemical procedure results in a purity greater than 98% with certainty. Several spectra of each source measured with a $3'' \times 3''$ NaI (Tl) crystal showed peaks and half-lives corresponding only to ^{100}Rh (21 hours), ^{101}Rh (4.5 days), ^{102}Rh (206 days), ^{105}Rh (36 hours) and ^{106}Rh (2.2 hours) [5].

The detailed procedure is as follows (Note 1):

a) The metalic palladium target is dissolved by heating in 5 ml of acqua regia and then evaporating to dryness. To eliminate nitrates, concentrated HCl is added and evaporated twice to dryness. The dry chloride is taken up in 4 or 5 ml of 6N HCl.

b) To retain palladium a column of De Acidite FF 530 resin (roughly equivalent to Dowex 21 k) is used with 12 mm internal diameter and 22 cm length. The solution is poured through this column, previously washed with 6N HCl. Then 6N HCl is added on the top of the column, and the first 75 ml passing through are recovered. (This is the Rh + Ag fraction.)

c) 5 mg of ferric ion carrier are added to this eluate.

d) Two or three drops of 0.5 M $AgNO_3$ are added. Then, by addition of 1:2 NH_4OH to the warm solution, $Fe(OH)_3$ is precipitated taking the rhodium with it. (The AgCl precipitate is dissolved during this step.) The liquid is filtered through a collodial filter and washed with aqueous ammonia; the $Fe(OH)_3$ precipitate is then dissolved with 5 ml 6N HCl.

Step d) is repeated twice.

e) The iron is extracted twice with 10 ml ethyl ether in two successive separatory funnels. The aqueous layer is kept and the residual ether in it evaporated carefully by gentle warming.

f) This solution is passed through a small 50–100 mesh Dowex 1 resin column of 5 mm diameter and 10 cm length, the resin being washed previously with 6N HCl. About 20 ml of eluate are recovered. (The possible inpurities of residual iron, palladium and ruthenium are retained here.) This eluate containing the pure rhodium activity, is evaporated to dryness.

g) The dry residue is extracted with 5 ml sulphuric acid solution (Note 2) to electroplate the rhodium. It is electrolized at 15–20 Volt, with a current density of 3 A/cm², at room temperature, on a nickel or palladium foil cathode and with a platinum wire anode, stirring the solution during the process. Cells for the electrodeposition of activities are described in [6] and [7].

Note 1. – As is well known, it is convenient to saturate the glass surfaces with inactive carrier to avoid the adsorption of activity, wetting the walls with the solution and keeping it during several hours. They are then washed with distilled water before use.

Note 2. – The solution is prepared as follows: 10 ml concentrated H_2SO_4 and 500 ml of distilled water according to [8].

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