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ORNL-TM-530 (Revised)

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ISOTOPES DEVELOPMENT CENTER
PROGRESS REPORT
REACTOR- AND CYCLOTRON-PRODUCED ISOTOPES
NOVEMBER-DECEMBER 1962

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ISOTOPES DEVELOPMENT CENTER

PROGRESS REPORT

REACTOR- AND CYCLOTRON-PRODUCED ISOTOPES

November-December 1962

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SUMMARY

A method of dissolving metallic foils (~ 0.001 in.) in varying concentrations of HCl (3.2 N, 6.5 N, 9.7 N and 12.2 N) by alternating current electrostripping is described. Under the conditions described, the amount of metal electrostripped in 30 min was found to be, in mg/in²: 80 (Rh), 900 (In), 90 (Pd), 2.5 (Au) and 12 (Ir).

The production of Rb⁸⁴ utilizing the Sr⁸⁷(p, α)Rb⁸⁴ and the Kr⁸⁴(p,n)Rb⁸⁴ reactions in the ORNL 86-Inch Cyclotron is described. A production rate of 3.3 μ c Rb⁸⁴/hr was found in the irradiation of SrO at a beam current of ~ 180 μ a. A gas target assembly for the irradiation of krypton gas (normal and Kr⁸⁶ depleted) is described. The average production rate from the proton irradiation of krypton was found to be 0.23 mc/hr, 1.1 mc/hr, and 0.5 mc/hr for Rb⁸³, Rb⁸⁴, and Rb⁸⁶ activities. The thick target reaction cross section at 12.9 Mev was estimated to be ≤ 4300 mb, ≤ 1900 mb, and ≤ 2200 mb, for the Kr⁸³(p,n)Rb⁸³, Kr⁸⁴(p,n)Rb⁸⁴, and Kr⁸⁶(p,n)Rb⁸⁶ reactions, respectively.

The production of Co⁵⁷ by the irradiation of nickel with 23-Mev protons is discussed. The preparation and irradiation of high purity, low-cobalt content nickel targets are described. Details are given for the electrostripping of the nickel target and the isolation and purification of the Co⁵⁷ activity. Cobalt-57 activity was obtained with a specific activity of ≥ 7.2 mc/ μ g Co.

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1. REACTOR-PRODUCED ISOTOPES

1.1. Target Processing

In the preparation of the chloride salts of radioisotope products, the effective dissolution of metal targets without the use of HNO_3 , prolonged boiling in HCl solution, or fusion would be very desirable. Table 1.1.1 lists the metals which are difficult to dissolve, the present method used, and time required for preparing the HCl solution of the target.

Table 1.1.1. Reactor Target Metals Which Resist Dissolution

Metal	Hours Required for Dissolution	Method Used for Dissolution
Hf	4	fusion
In	4	boiling with HCl
Ir	4-8	fusion in chlorine atmosphere
Os	1 ^b	boiling in aqua regia
Ta	4-6	fusion
Pt	1 ^b	boiling in aqua regia
Pd	1 ^b	boiling in aqua regia
Rh ^a	4-6	mechanically reduced and boiled in H_2SO_4 and HCl
Au	1 ^b	boiling in aqua regia

^acyclotron target.

^b2-3 hrs are required to rid solution of HNO_3 or nitrate ions.

The presence of HNO_3 is undesirable when the final product is to be used in the human or animal system; therefore, a technique for dissolution which does not require HNO_3 eliminates the need for additional chemical treatment and reduces the time required for processing the target.

A method reported by Yufa and Chentsova¹ employs alternating current to dissolve platinum metals and their alloys in HCl solution. A schematic diagram of the apparatus consisting of a 10-amp Variac, a transformer, and leads, based on the design by Yufa and Chentsova, is

¹T. P. Yufa and M. S. Chentsova, Analiz Blagorodn. Metal. Akad. Nauk SSSR, Inst. Obshch. i Neorgan. Khimi. 1959, 176; CA: 54, 13901b.

shown in Fig. 1.1.1. Preliminary tests were made to evaluate the method by dissolving foils of rhodium, palladium, iridium, indium, and gold, ~0.001 in. thick, in different concentrations of HCl. The maximum current obtainable in the equipment was used in each case. The results (Fig. 1.1.2) indicate that the method will reduce the time for routine chemical processing of targets.

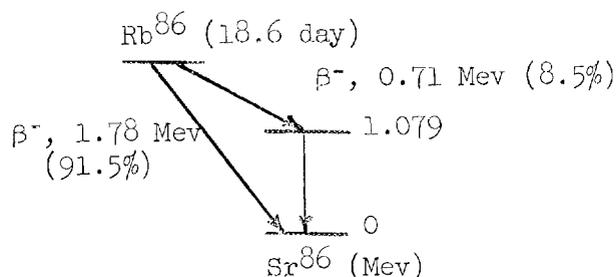
2.0 CYCLOTRON-PRODUCED ISOTOPES

2.1. Production of Rubidium-84

The absence of any radioactive potassium isotope with a half-life between 12.4 hr and 1.3×10^9 yr has been a major deterrent to effective solutions of many problems. Both cesium and rubidium have been used as substitutes for potassium. Of the two, rubidium has properties most similar to potassium.

Since the principal medical use of radioactive rubidium is as a tracer, the dose to the system for a given level of detection should be minimized. Of the three readily available rubidium isotopes -- Rb^{83} ($T_{1/2} = 83$ days), Rb^{84} ($T_{1/2} = 33$ days), and Rb^{86} ($T_{1/2} = 18.6$ days) -- Rb^{83} is least desirable because of its 83-day half-life. It can be seen from the decay schemes²⁻⁵ that detection of Rb^{86} must be based on beta particles (which virtually excludes in vivo measurements) or on the 1.079-Mev photon which is emitted in 8% of the disintegrations.

Rubidium-84 is ~10 times more effective as a gamma tracer than Rb^{86} because about ten times as many photons per disintegration are emitted by Rb^{84} than by Rb^{86} .



(Decay scheme from Ref. 3,4; Half-life from Ref. 5)

²The decay schemes used are from Ref. 3; where necessary these decay schemes are brought up to date (e.g., by use of Ref. 4 or 5).

³D. Strominger, J. M. Hollander, and G. T. Seaborg, Rev. Mod. Phys. 30, 585 (1958).

⁴B. S. Dzhelepov and L. K. Peker (trans. ed., D. L. Allan), Decay Schemes of Radioactive Nuclei, Pergamon Press, New York, 1961.

⁵J. F. Stehn, Nucleonics 18 (11), 186 (1960).

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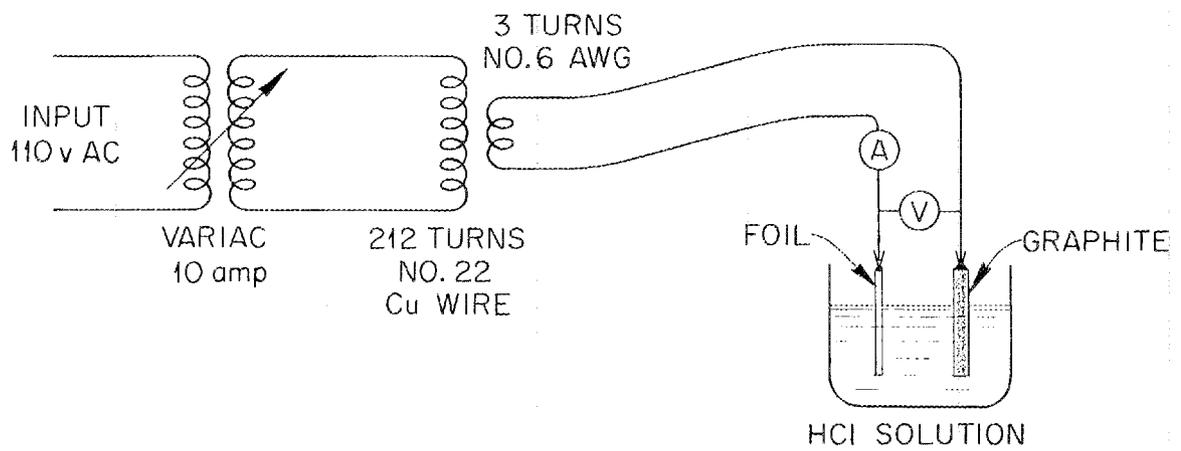


Fig. 1.1.1. Schematic Diagram of Alternating Current Electrostripping Apparatus.

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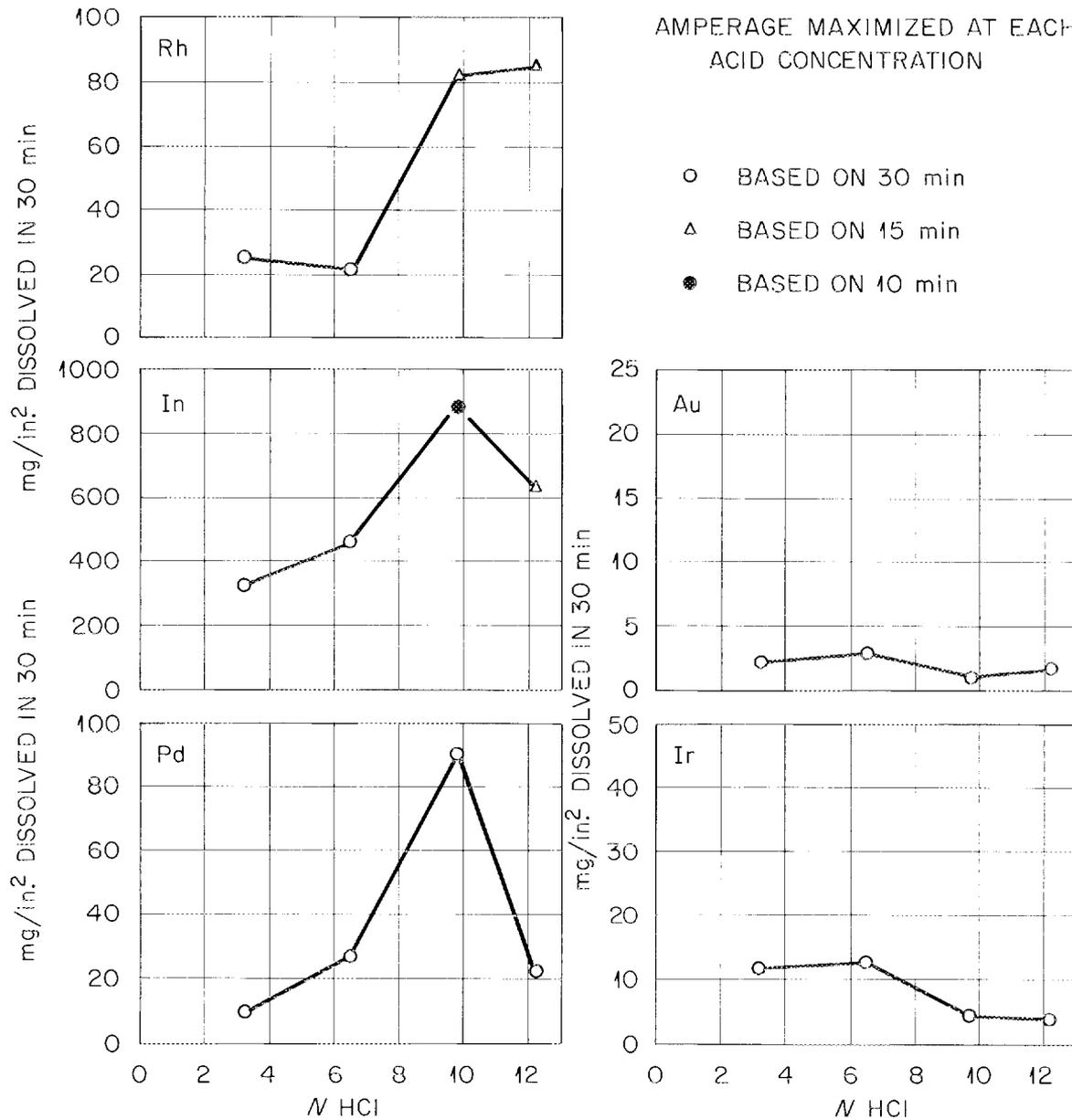
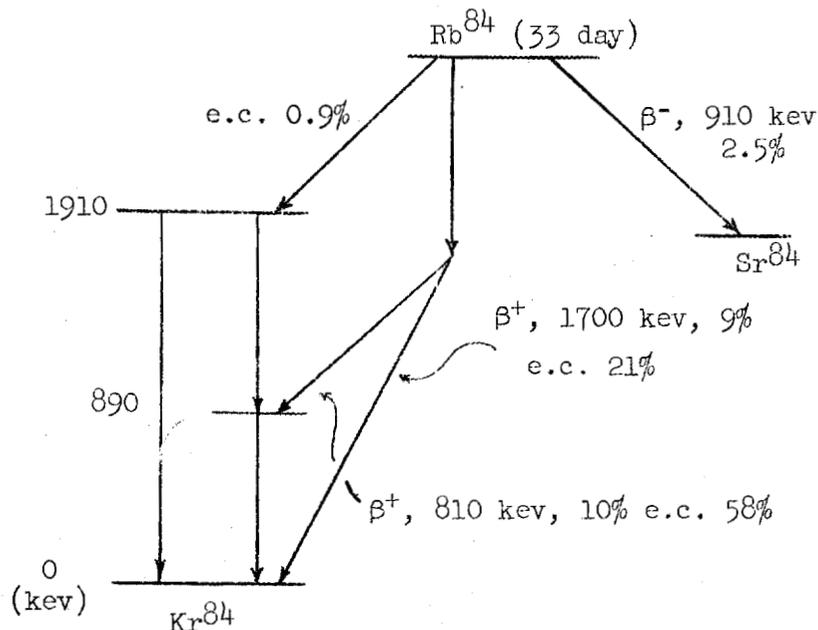


Fig. 1.1.2. Alternating Current Electrostripping of Ir, Pd, In, Au and Rh.



(Decay scheme from Ref. 4)

Nuclear Reactions and Production

Carrier-free Rb^{84} can be produced in the ORNL 86-Inch Cyclotron by the following reactions:

<u>Reactions</u>	<u>Q (Mev)</u> ^{6,7}	
$\text{Sr}^{87}(\text{p},\alpha)\text{Rb}^{84}$	- 0.05	(1)
$\text{Sr}^{88}(\text{p},\alpha\text{n})\text{Rb}^{84}$	-11.67	(2)
$\text{Kr}^{84}(\text{p},\text{n})\text{Rb}^{84}$	- 3.49	(3)

In order to obtain yield information, a target of SrO (containing ~30% SrCO_3) which was infinitely thick to 23-Mev protons, was irradiated at the zero-degree facility.⁸ Approximately 7.3 μC Y^{88} and traces of $\text{Sr}^{87\text{m}}$ and Sr^{85} were detected; no Rb^{84} was detected.

⁶The Q-value is the energy of the reaction and describes energy release (positive Q) or energy absorption (negative Q) as a result of the nuclear transformations. Q values were calculated from tables given in Ref. 7.

⁷A. H. Wapstra, Physica 21, 367 (1955).

⁸T. A. Butler, Reactor- and Cyclotron-Produced Isotopes, July-October 1962, ORNL-TM-463.

A second irradiation was made in the internal beam, where the proton beam current is 10^3 to 10^4 times that in the zero-degree facility. An irradiation of ~ 2 g SrO for 1080 $\mu\text{a-hr}$ produced a total of 19.6 $\mu\text{c Rb}^{84}$ at a rate of 3.3 $\mu\text{c/hr}$. Even the substitution of 95% enriched Sr^{87} , which would raise the target cost to several thousand dollars, would result in a yield of only 0.045 mc/hr . Therefore, this method is not considered practical.

The production of Rb^{84} using the reaction $\text{Kr}^{84}(\text{p,n})\text{Rb}^{84}$ has serious objections associated with the irradiation of a gas: only the deflected beam can be used, only a limited number of target atoms can be presented to this deflected beam, and handling a gas target is inconvenient. The estimated yield per hour, based on experience with other (p,n) reactions of approximately the same mass and atomic number, is 1.1 mc Rb^{84} for a 10- μa beam current.

A gas target chamber, shown in Fig. 2.1.1 was affixed to the deflected beam "T" position.⁸ With this target configuration, beam currents were available in the range of $15 \pm 5 \mu\text{a}$, depending on the operating conditions of the cyclotron. The cross sections for (p,n) reactions normally peak at ~ 12 Mev; therefore, the 23-Mev protons were passed through a 0.063-in. aluminum window to degrade the beam energy to ~ 14 Mev. Since the gas target further degraded the beam energy to ~ 3 Mev, at which energy no nuclear reactions are expected to occur, the target was considered to be infinitely thick.

The nuclear reactions of interest at ≤ 14 Mev are:



A two or three-day cooling period allows the short-lived activities associated with aluminum and its oxides and impurities as well as short-lived krypton and rubidium activities to decay to a negligible level. The primary activities remaining after the cooling period are those associated with rubidium-83, -84, and -86.

The experiments described were first performed with normal krypton and later with krypton depleted in Kr^{86} in order to minimize the Rb^{86} production. No change in Rb^{86} production due to isotopic depletion was detectable. The relative abundances of the krypton isotopes in the target are shown in Table 2.1.1.

The krypton filling and storage system showing a cylinder of normal krypton, a flask of Kr^{86} depleted krypton, a thermocouple ion gauge, and a thick-walled Monel tube with the liquid nitrogen trap is shown in Fig. 2.1.2. The yields of rubidium activities which were observed in both normal and depleted krypton are given in Table 2.1.2.

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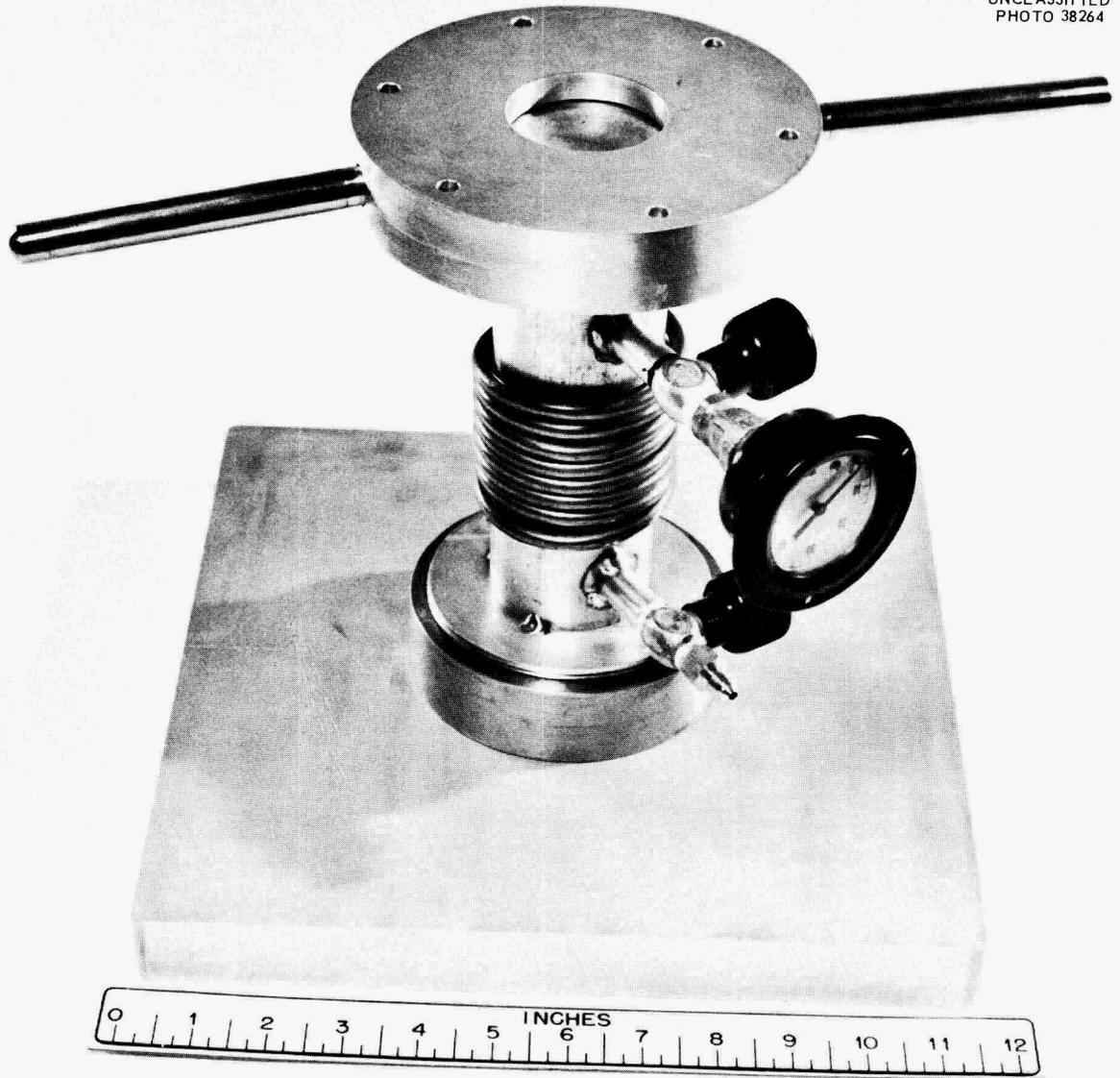


Fig. 2.1.1. Krypton Gas Target Chamber for ORNL 86-Inch Cyclotron.

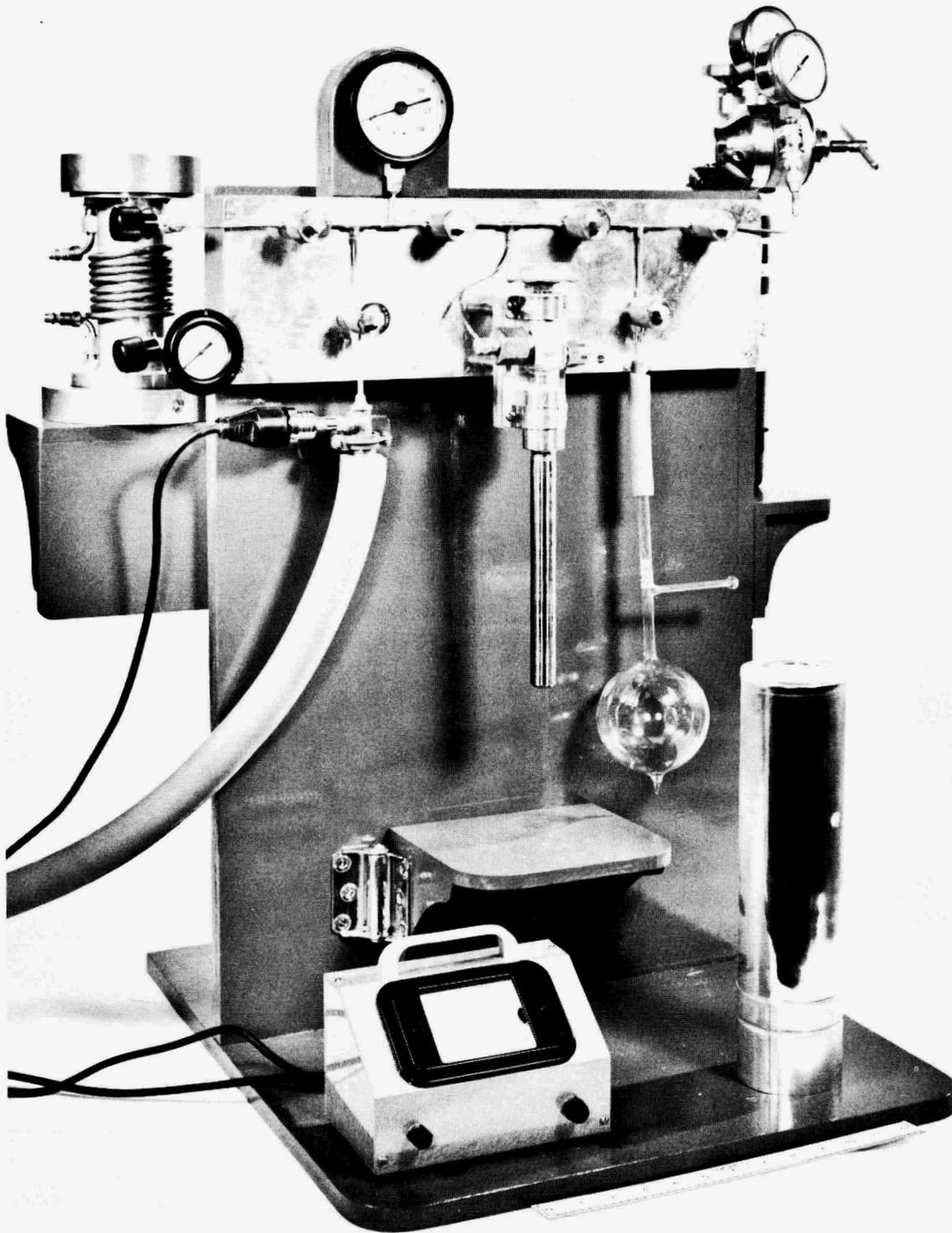
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Fig. 2.1.2. Krypton Gas Target Filling and Storage System for ORNL 86-Inch Cyclotron.

2.1.1. Isotopic Composition of Krypton Gas Target
Used in the Production of Rubidium-84

Isotope	Normal Abundance %	Kr ⁸⁶ Depleted %
Kr ⁷⁸	0.35	0.41
Kr ⁸⁰	2.27	2.44
Kr ⁸²	11.56	12.28
Kr ⁸³	11.55	12.17
Kr ⁸⁴	56.90	60.17
Kr ⁸⁶	17.37	12.54

Table 2.1.2. Rubidium-83, -84, and -86 Yields From
the Proton Irradiation of Krypton

Isotope	Half-life (days)	Average Yield Beam Current ~15 μ a (mc/hr)
Rb ⁸³	83	0.23
Rb ⁸⁴	33	1.1
Rb ⁸⁶	18.6	0.5

Nuclear Reaction Cross Section

An estimate of the upper limit of the nuclear reaction cross sections may be made from the thick target yields. It is probably correct to assume that the contribution to Rb⁸³ production by the Kr⁸⁴(p,2n)Rb⁸³ reaction is negligible on the basis that the Q value is equal to -11.54 Mev (calculated from the semi-empirical mass tabulations of Cameron⁹). If the yield per incident proton (Y/I) at 13.9 Mev is not more than an order of magnitude different than Y/I at 11.9 Mev (which is a reasonable overestimate in this energy range), the cross section ($\bar{\sigma}$) at 12.9 Mev for the reaction may be estimated by inserting the stopping power ($\frac{1}{\rho} \frac{dE}{dx}$) of krypton for protons in the equation given below:

$$\bar{\sigma} = \frac{(Y)}{(I)} \frac{MW}{N_0} \left(\frac{1}{\rho} \frac{dE}{dx} \right) \frac{1}{f},$$

⁹A. G. W. Cameron, Atomic Energy of Canada Limited Report, AECL-433 (1957).

where

MW = molecular weight,
 N_0 = Avogadro's number,
 f = isotopic abundance,
 ρ = density.

The stopping power of krypton for protons was interpolated from range energy relations given by Sternheimer.¹⁰ These estimated cross sections (calculated in the manner described above) are shown in Table 2.1.3.

Table 2.1.3. Upper Limits of Cross Section for the Production of Rubidium-83, -84, and -86 by Proton Bombardment of Krypton

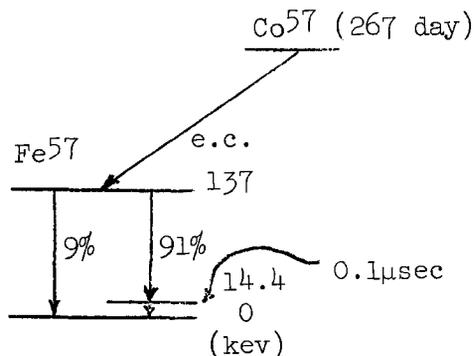
Target	Reaction	Product	Q (Mev)	Y/I	Cross Section ($\bar{\sigma}$) (mb)
Kr ⁸³	p,n	Rb ⁸³	- 1.47 ^a	205x10 ⁻⁶	≤ 4300
Kr ⁸⁴	p,2n	Rb ⁸³	-11.54 ^a	- -	- -
Kr ⁸⁴	p,n	Rb ⁸⁴	- 3.43	445x10 ⁻⁶	≤ 1900
Kr ⁸⁶	p,n	Rb ⁸⁶	- 1.07 ^a	138x10 ⁻⁶	≤ 2200

^aCalculated from the semi-empirical mass tabulations of Cameron.⁹

Additional irradiations are planned which will utilize ~12-Mev incident proton beam. Hence, the assumptions pertaining to the yield at 11.9 Mev will not be necessary, and a more exact value of the cross sections will be known.

2.2. Production of Cobalt-57

Cobalt-57 is very useful as a standard in gamma ray spectrometry and in labeling radiocyanocobalamin (vitamin B-12). The 267-day half-life Co⁵⁷ nucleus decays by electron capture which is followed by the emission of a 122-kev gamma ray to the 0.10 μ sec Fe⁵⁷ level which emits a 14.4-kev gamma. Iron-57 is a particularly good source for Mössbauer studies (i.e., recoilless emission and absorption of gamma rays).

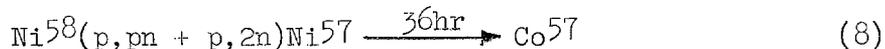


(Decay scheme after Strominger, Hollander, and Seaborg³)

¹⁰R. M. Sternheimer, Phys. Rev. 115, 137 (1959).

Nuclear Reactions and Nuclear Reaction Cross Sections

Cobalt-57 can be produced in nickel by the following nuclear reactions:



The primary production method is the (p,2p) reaction (9) and the secondary method, the (p,pn + p,2n) reaction (8). The cross sections¹¹ of these reactions at 21.5 Mev are 680 mb for reaction (9) and 240 mb for reaction (8). The cross section for the (p,pn + p,2n) reaction agrees with the measured excitation function.¹² The cross sections for the other three reactions are orders of magnitude less than for reactions (8) and (9). A thin plating (0.005 to 0.006 in.) of nickel absorbs the high energy portion of the proton beam.

The target is stored for 2 weeks after irradiation to allow the Co⁵⁷ to grow by the decay of Ni⁵⁷. The decay of Co⁵⁷ during the 2 weeks is negligible when compared to the 35% increase in the available Co⁵⁷.

Target Preparation

A convenient form of high purity, low-cobalt nickel was available as foils; 3 x 4 x 0.010 in. foils were soldered in a recess atop a copper flat plate target.¹³ The target foils were either soft soldered, silver soldered, or gold soldered under various atmospheres. The targets were ultrasonically tested for bonding and were x-rayed for voids. Targets which exhibited minimum voids or non-bonding surfaces were selected for proton irradiation. The power input for routine production runs amounts to ~35 kw, 90% of which covers a zone ~1 x 3 in. Such targets were not usually successful, since the portion of the foil which received most of the beam was invariably damaged so that considerable losses of activity were incurred.

The lack of success with soldered targets suggested the advisability of investigating the purification of nickel in order to obtain low-cobalt content nickel suitable for electroplating. Nickel, plated by conven-

¹¹B. L. Cohen, E. Newman, and T. H. Handley, Phys. Rev. 99, 723 (1955).

¹²J. J. Pinajian, J. L. Need, and W. H. Webb, Bull. Am. Phys. Soc. 5, 267 (1960).

¹³T. A. Butler, Reactor- and Cyclotron-Produced Isotopes, July-October, 1962, ORNL-TM-463.

tional techniques, has a cobalt content in the range of 1000 to 2000 ppm. Pure plated targets were prepared by using nickel salts which had been purified by ion-exchange (Dowex 1-X10) and an extremely high purity, low-cobalt, nickel anode.¹⁴ A lucite plating chamber, shown in Fig. 2.2.1, was prepared so that the 5-5/8- x 6- x 1/2-in. copper flat plate formed one side and the high purity nickel briquette formed the other side. The nickel anode (i.e., the briquette) was mounted in the face of a lucite slab with epoxy resin.

The copper flat plate was degreased and a fresh surface was exposed by treatment with acid. A Watts-type bath was prepared using the following formulation and conditions:

Nickel sulfate, NiSO ₄	99.0 g
Nickel chloride, NiCl ₂ ·6H ₂ O	26.9 g
Boric acid	17.9 g
Sodium lauryl sulfate	2.25 g
Sodium carbonate, Na ₂ CO ₃	3.0 g
Solution volume	610 ml
pH	3 to 5
Temperature (room)	22 to 25°C
Current density	4.16 amp/sq ft

A Constant Current Regatron¹⁵ was used for the power source. Nickel plates were prepared in thicknesses ranging from 0.005 to 0.018 in.

Target Irradiation

The targets were mounted as described in an earlier report¹³ and installed in the cyclotron at such a radius as to give the maximum proton energy consistent with stable operation. The latter is dependent on the cyclotron operating condition and will vary from time to time. It has appeared from past experience that, although this type of nickel target is capable of withstanding a beam current of > 1750 μ a (when 80 gal/min of cooling water is used instead of the standard 56 gal/min), there is no resultant increase in the production yield. A possible explanation of this is that surface temperatures are sufficiently high at these beam currents to evaporate the surface material. This explanation is substantiated by the presence of large amounts of activity on the cyclotron dees after an extremely high beam current irradiation of this sort. A beam current of \sim 1250 μ a appears to be most satisfactory. With such a beam and under optimum operating conditions, \sim 24 mc/hr Co⁵⁷ yield was realized.

¹⁴Obtained from Mond Nickel Co., Ltd., England, and supplied by Dr. A. Edson, The International Nickel Company, Inc., New Jersey.

¹⁵Electronic Measurements Company, Inc., Eatontown, New Jersey.

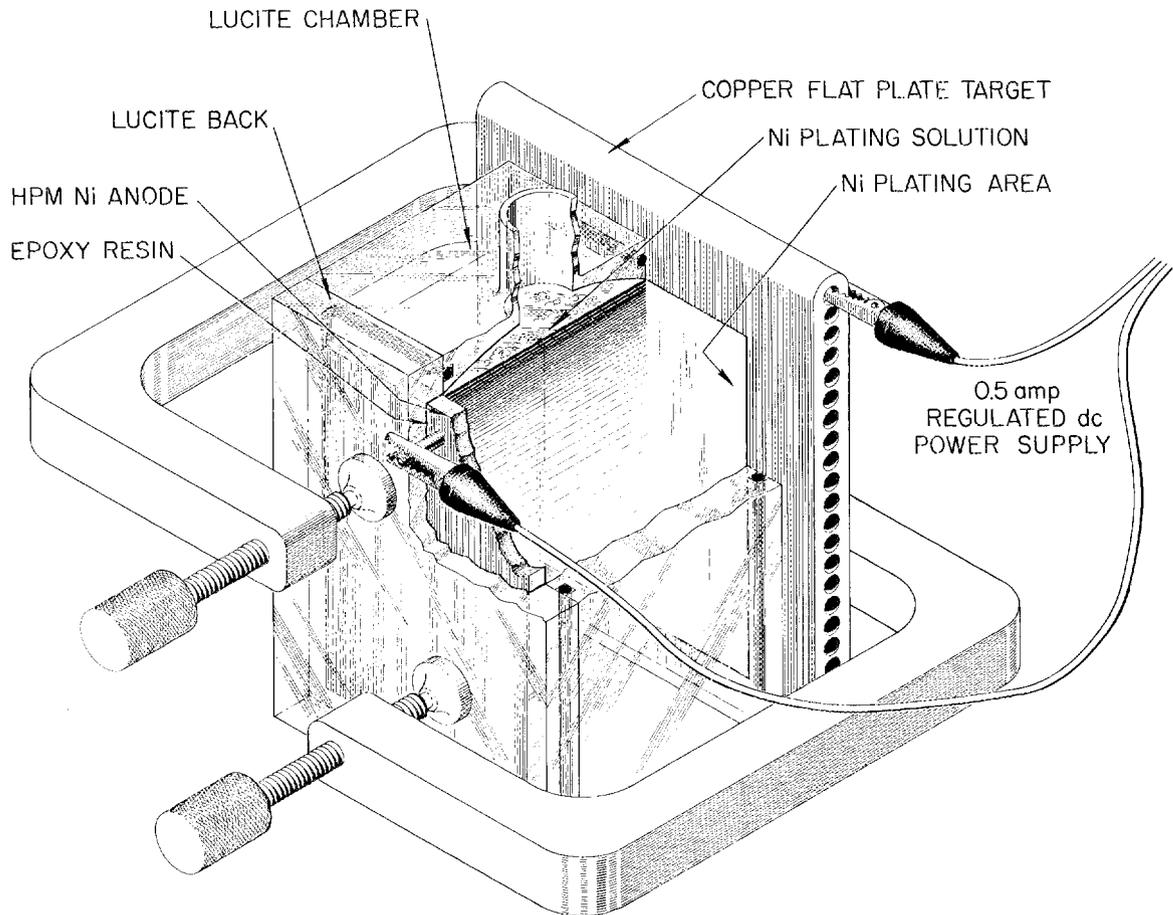
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Fig. 2.2.1. Nickel Plating Chamber.

Target Processing

The aged irradiated target was disassembled and placed in the aluminum base plate of the electrostripping apparatus shown in Fig. 2.2.2. Electrostripping of the target using 0.5 N HCl, a platinum electrode ~1 in. from the nickel surface, and a direct current of 2.0 amp, removed ~65% of the total activity in 30 min, 97% in 150 min, and 99% in 180 min. Because of experimental variations in target thickness as well as beam current differences, the 0.010- to 0.020-in. thick platings required additional HCl and longer stripping periods. Thin targets (0.006 to 0.008 in.) required ~2 hr for removal of > 95% of the total activity. Individual targets were electrostripped stepwise so that the penetration of the activity could be studied. The results are shown in Fig. 2.2.3. It is clear that a target thickness of 0.002 in. is the minimum thickness required for the optimum production of Co⁵⁷.

Specific Activity Determination

The crude product solution was treated with H₂S to remove the copper. The normality of the filtrate was adjusted to ~9 N HCl, and the same Dowex 1-X10 anion exchange resin system, described earlier, was used for the separation of the Co⁵⁷ activity. The effluent was evaporated to dryness and treated with HNO₃ to destroy organic material. The Co⁵⁷ activity was dissolved in dilute HCl, water was added, and 5- to 10-ml aliquots were used for spectrophotometric analysis.

The initial targets demonstrated a specific activity of 4.2 mc Co⁵⁷ per µg of Co, and later targets had specific activities of ~6 mc Co⁵⁷ per µg Co. More recent targets have shown yields of ≥ 7.2 mc/µg. The specific activity of isotopically pure Co⁵⁷ would be 8.4 mc/µg. Estimates show that stable Co⁵⁹ may be produced from the Ni⁶⁰(p,2p)Co⁵⁹ reaction in sufficient quantity to account for the difference between the experimental specific activity and the calculated specific activity.

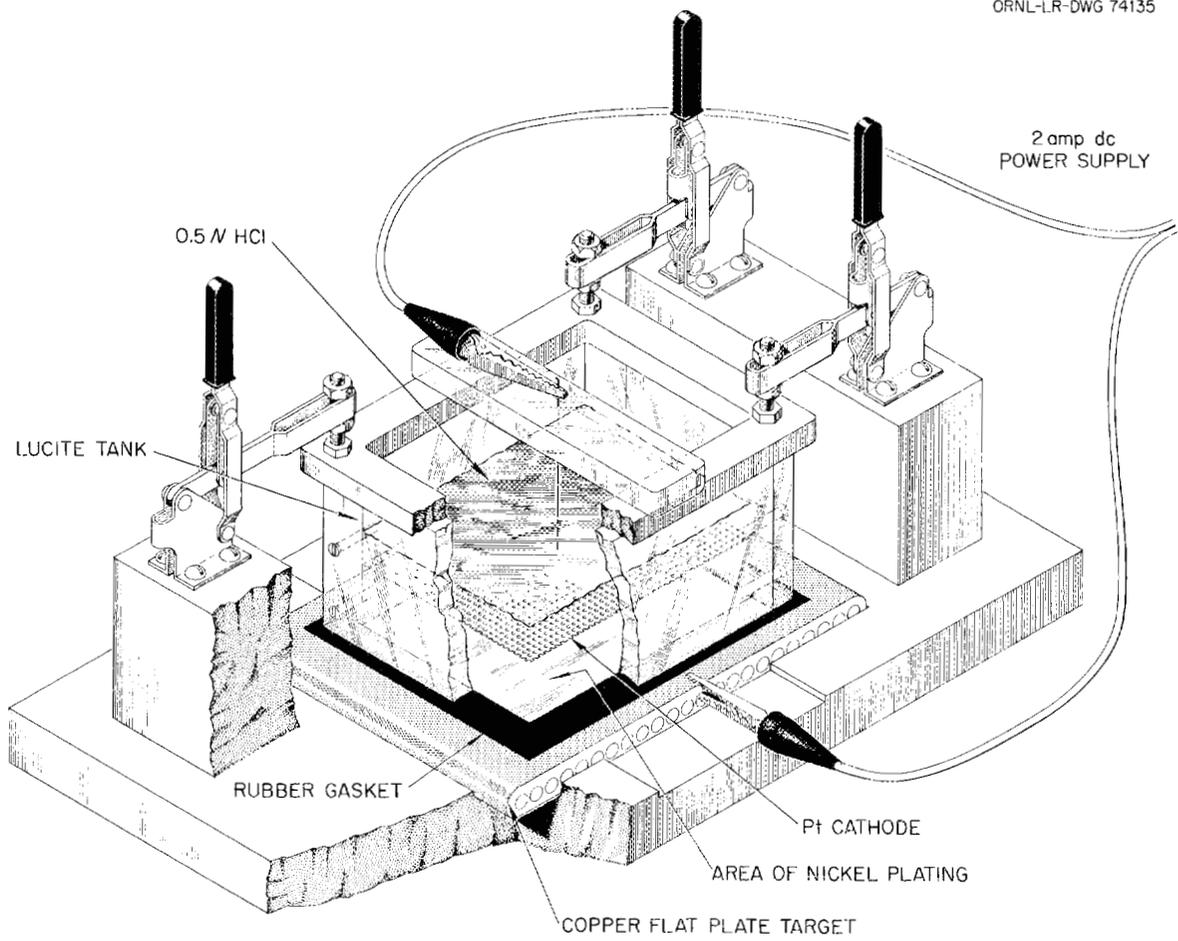
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Fig. 2.2.2. Nickel Electrostripping Apparatus.

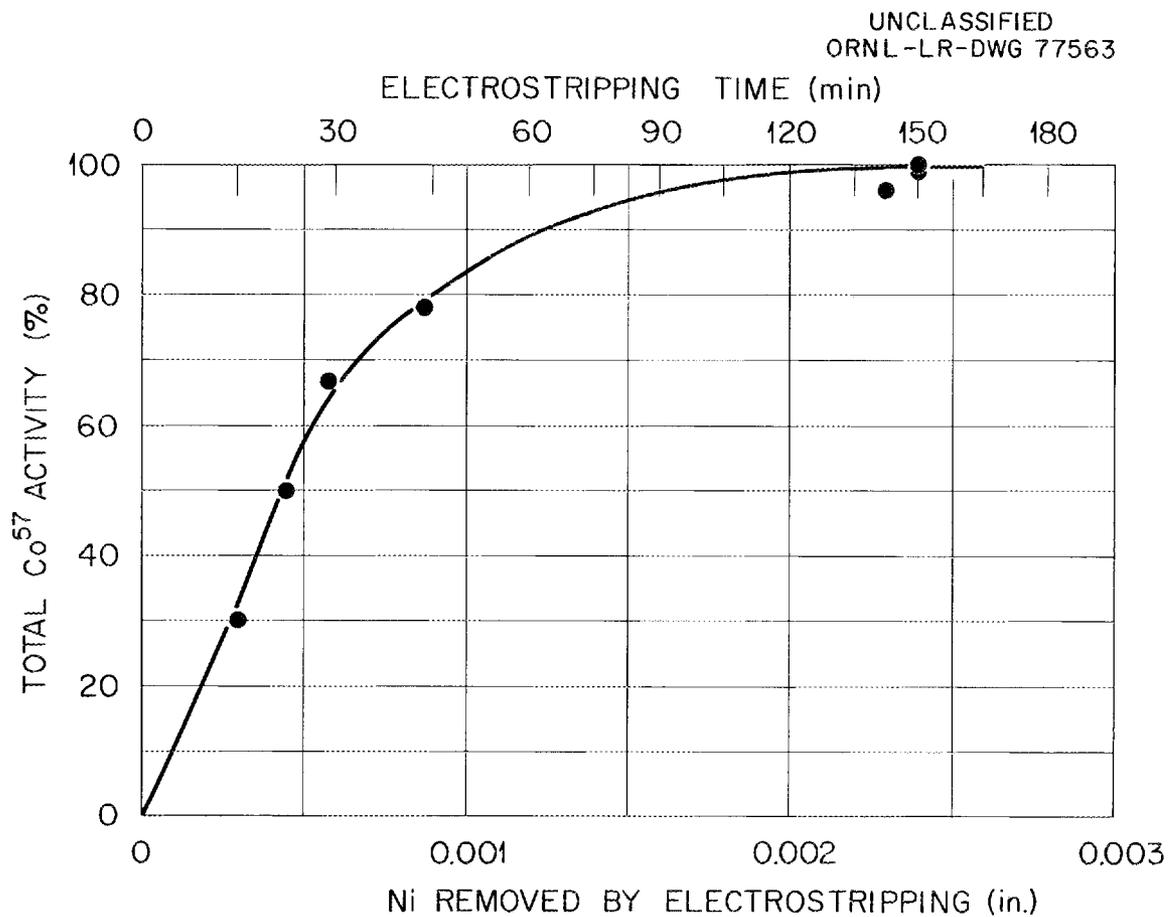


Fig. 2.2.3. Cobalt-57 Removed from Nickel Target by Electrostripping.

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