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MACRORETICULAR ION EXCHANGE RESIN CLEANUP OF PUREX PROCESS TBP SOLVENT

By

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MACRORETICULAR ION EXCHANGE RESIN CLEANUP
OF PUREX PROCESS TBP SOLVENT

By

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August 1, 1970

ATLANTIC RICHFIELD HANFORD COMPANY
RICHLAND, WASHINGTON

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ABSTRACT

Strong base macroreticular ion exchange resins effectively remove both fission products and diluent and TBP degradation products from used Purex process solvent. Application of such resins in routine cleanup of Purex process extractant is potentially attractive to eliminate the large volumes of radioactive waste generated by presently-used solvent wash procedures.

The capacity of macroreticular resins for adsorbing extractant impurities is very high judging from batch and column data. Over 240 bed volumes of unwashed Hanford Purex plant first cycle solvent were passed downflow (at 40 C and 4 bed volumes/hr) through a 59-ml bed of 14 to 50 mesh resin without any detectable breakthrough of impurities. All the effluent solvent was water-white as opposed to the faint-yellow color of the feed; its fission product content and plutonium retention number were both substantially lower than typical washed (alkaline permanganate) plant solvent. Elution of loaded resin with a few bed volumes of $3\underline{M}$ HNO_3-$0.05\underline{M}$ HF and $4\underline{M}$ NaOH removes all the adsorbed yellow color and ^{95}Zr-^{95}Nb activity but only 55 to 65 percent of the ^{106}Ru-^{106}Rh load.

MACRORETICULAR ION EXCHANGE RESIN CLEANUP
OF PUREX PROCESS TBP SOLVENT

The classic Purex Process[1] or some modification thereof is now universally used to reprocess all kinds of irradiated nuclear reactor fuel. All Purex plants routinely perform "solvent treatment" operations to maintain the quality of the tri-n-butyl phosphate (TBP) extractant. Solvent treatment conventionally involves washing the used extractant with aqueous alkaline, alkaline permanganate, or acid solutions to remove dibutyl phosphoric acid (HDBP), residual fission products, and, at least partially, diluent degradation products. [A typical wash sequence is that used to treat Hanford Purex plant first cycle solvent and discussed in the APPENDIX.] Chemical and radiolytic degradation of TBP extractants and solvent treatment procedures have been reviewed by several authors.[2-5]

Sustained, satisfactory operation of various Purex plants in the United States and elsewhere attests to the efficiency of present-day solvent washing techniques. A major disadvantage of these methods, however, is that they generate large volumes of radioactive aqueous waste which must be stored or otherwise treated as high-level waste. At Hanford, for example, large volumes of solvent wash waste are generated each year. Development of an alternative solvent treatment procedure which does not generate such wastes is both economically and environmentally desirable.

Very promising results have been obtained in this direction utilizing the properties of macroreticular ion exchange resins. Such resins are identical to their conventional microreticular counterparts except for having much larger pore diameters. These large pores do not disappear when the swelling solvent,

water, is removed; hence, macroreticular resins are especially suited for use with non-aqueous, even non-polar, solutions.[6, 7]

Japanese workers[8, 9] previously applied microreticular ion exchange resins for removal of various acidic components from degraded TBP-diluent solutions. Our work extends the concept to the more suitable macroreticular resins and to experiments with actual Purex plant solvent.

The Hanford Purex plant recently changed from Soltrol-170, a mixture of 100 percent branched paraffins, to NPH, a mixture of C_{10} to C_{14} normal paraffins, as a diluent for TBP. [Other Purex plants have made similar diluent changes.] Subsequently, overall plant decontamination performance improved markedly; and, especially important to this work, the concentration of fission products in the unwashed first cycle solvent decreased 20- to 40-fold. The concentration of nitroalkanes in the recycled solvent also decreased noticeably. These favorable changes in solvent quality and stability significantly enhance applicability of ion exchange solvent cleanup procedures.

EXPERIMENTAL DETAILS

MATERIALS

Rohm and Haas Company macroreticular (*Amberlyst*) resins were used throughout. Amberlyst-15 (cation exchange) and Amberlyst A-21 (weak base anion exchange) resins were used in the as-received (H^+- and OH^--forms, respectively) condition. Amberlyst A-26 and A-29 (strong base anion exchange) resins were converted from the as-received chloride- to the hydroxide-form by exhaustive washing with 4$\underline{M}$ NaOH. Air-dried resins were screened (U.S. Standard Sieve series) to obtain fractions encompassing desired particle sizes for use in batch equilibration tests.

As-received TBP (Commercial Solvents Corporation) was
diluted with NPH (South Hampton Company) to prepare 30 per-
cent TBP solvent; HDBP (Eastman, purified from monobutyl-
phosphoric acid) was added in some cases to a concentration of
0.054$\underline{M}$.

Unwashed Hanford Purex plant first cycle solvent (1CW
solution) was used in fission product retention studies. This
pale-yellow material contained, nominally, 30 percent TBP, and,
depending on plant performance, from 18 to 350 µCi/liter
^{95}Zr-^{95}Nb (*ca.* 50 percent ^{95}Zr) and from 75 to 350 µCi/liter
^{106}Ru-^{106}Rh. Small amounts of ^{103}Ru and HNO$_3$ ($\sim$0.005$\underline{M}$) were
also present; the concentration of HDBP in plant solvent was
not measured. Plant 1CW solution was obtained fresh as needed
and was not allowed to age more than about 7 days before use.
For comparative purposes, representative wash (*cf.* APPENDIX)
Hanford Purex plant first cycle solvent (1OO solution) was also
procured--composition and properties of this solvent are listed
later.

All other chemicals were of reagent grade quality.

DISTRIBUTION RATIO TESTS

Two-gram portions of screened, air-dried resin were con-
tacted (30 min, 25 C; mechanical stirring) twice with fresh
10-ml portions of 30 percent TBP-NPH and then (at various times
and temperatures) with 10 ml of either plant 1CW or laboratory-
prepared 30 percent TBP-0.054$\underline{M}$ DBP-NPH. [All liquid-solid
separations were by centrifugation.] Initial and final liquid
phases from the last contact were analyzed either for fission
product content [gamma energy pulse height analyses with NaI
or Ge(Li) detectors] or for DBP [Beckman Automatic Titrator;
derivative mode titration with alcoholic KOH]. Distribution
ratios (K$_d$) for the loading step were calculated as

$$K_d = \frac{\text{amount of material on resin per gram of air-dried resin}}{\text{amount of material in solution per milliliter of solution}}.$$

Batch fission product elution tests were performed with two-gram amounts of 14- to 20-mesh A-29 resin previously equilibrated twice with 30 percent TBP-NPH and once (30 min at 40 C) with plant 1CW. The resulting resin was washed twice at 40 C with 10-ml portions of NPH and then contacted 15 min at either 25 or 40 C with 10 ml of eluent solution.

COLUMN RUNS

Jacketed glass columns (1.88 cm ID) were filled with 25 ml (*ca.* 17 g) of air-dried 14- to 50-mesh A-26 resin. After classification by upflow of water, the bed height was about 21 cm corresponding to a bed volume of 58 ml. Successive 4 bed volume portions of 4$\underline{M}$ NaOH and laboratory-prepared 30 percent TBP-NPH were passed downflow at 25 C and at a rate of 4 bed volumes/hr. No change in bed volume occurred during these latter treatments.

All column runs with 1CW solution were made with downflow loading at 40 C. Three runs were made at flow rates of 1.1, 4.3, and 8.6 bed volumes/hr, respectively, to determine the effects of flow rate upon fission product retention. In each test 50 to 55 bed volumes of 1CW were loaded.

Additional tests were made to establish A-26 resin capacity and behavior during consecutive load and elution cycles. Initially, 245 bed volumes of 1CW solution (taken at various times during plant operation) were loaded onto a fresh resin bed at a rate of 4 bed volumes/hr. Consecutive portions of water, 3$\underline{M}$ HNO$_3$-0.05$\underline{M}$ HF, and 4$\underline{M}$ NaOH (4, 16, and 12 bed volumes, respectively) were used to wash, elute, and regenerate the resin. Elution was done upflow at 25 C and at a rate of 4 bed volumes/hr. Following reclassification of the eluted bed with

water, an additional 107 bed volumes of 1CW solution were loaded. Except that it was done at 40 C and at 2 bed volumes/hr, the second elution cycle was identical to the first.

SOLVENT QUALITY TESTS

Various physical and chemical properties of plant solvent after ion exchange cleanup were measured. Similar measurements were made with washed and unwashed plant solvents. Tri-n-butyl phosphate concentrations were determined by gas-liquid chromato-graphy.[10] The apparatus of Mendel and Moore[11] served to measure disengaging times when a TBP solvent was mixed at 25 C with an equal volume of $1.84\underline{M}$ $UO_2(NO_3)_2$-$0.5\underline{M}$ HNO_3 solution; uranium extraction distribution ratios (E_a^o) were calculated from uranium concentrations of the resulting phases.

Plutonium retention tests involved contacting the TBP phase (5 min, 25 C) with one-fifth volume of $3\underline{M}$ HNO_3-$0.01\underline{M}$ $Pu(NO_3)_4$ solution; the resulting organic phase was scrubbed three times with fresh double-volume portions of $0.01\underline{M}$ HNO_3. The Pu retention number was calculated by multiplying the molarity of plutonium in the final organic phase by 10^9.

<div align="center">RESULTS AND DISCUSSION</div>

BATCH TESTS

Loading Tests

Results of various batch loading tests with macroreticular resins are presented in Figures 1 through 5, pages 15 through 19. Data from these tests provided valuable guidance for selection of optimum column operating conditions.

The two strong base exchangers, A-26 and A-29 resins, exhibit about the same affinity for adsorbing fission products from unwashed Purex process solvent. For this purpose, both

A-26 and A-29 resins are superior to either A-21 (weak base exchanger) or Amberlyst-15 (cation exchanger) resins. This same resin ranking order was also observed with smaller resin particles than the 14 to 20 mesh fractions used to obtain the data shown in Figures 1 and 2, pages 15 and 16. Affinity of the Amberlyst-15 resin for ^{95}Zr-^{95}Nb was greater than that of A-21 resin, while just the opposite was true for adsorption of radioruthenium.

Kinetics of adsorption of fission products from used Purex extractant by macroreticular ion exchange resins are significantly faster at 40 C than at 25 C (Figures 3 and 4, pages 21 and 22; to take advantage of this fact, subsequent column runs were made at 40 C. These column runs were made with A-26 resin since manufacturer's literature indicates thermal stability of A-26 resin is slightly greater than that of the A-29 resin.[7]

Kinetics of fission product uptake by the macroreticular resins also varies with resin particle size. As expected, the smaller particles absorb activity faster than large beads at both 25 and 40 C. At long contact times the particle size effect disappears and the equilibrium distribution for a particular resin is attained. Column runs were made with as-supplied 14 to 50 mesh A-26 resin; classification into smaller particle size fractions is considered impractical for projected plant-scale application.

Characteristics of the strong adsorption of HDBP by strong base macroreticular anion exchange resins are shown in Figure 5, page 19. Kinetic effects operative in this case and their variation with temperature and particle size are similar to those noted for uptake of fission products. In the Japanese work cited earlier, in addition to HDBP various other acidic components (e.g., monobutylphosphoric acid and carboxylic acids) present in degraded TBP solutions also reported strongly to the resin phase. Indirect evidence for adsorption of acidic

components other than HDBP and fission products from Purex 1CW
solution was obtained in column runs; this evidence is con-
sidered later.

Elution Tests

Various reagents were screened on a batch basis to deter-
mine their ability to elute fission product activity from
loaded macroreticular resin. Results of some of these tests
are listed in Table I.

TABLE I

FISSION PRODUCT ELUTION-BATCH TESTS

Two grams 14-20 mesh A-29 resin containing 12 μCi ^{95}Zr-^{95}Nb
and 1.6 μCi ^{106}Ru-^{106}Rh contacted 15 min at 25 C with 10 ml
eluent

	Percent Eluted	
Eluent Composition	^{95}Zr-^{95}Nb	^{106}Ru-^{106}Rh
1M HNO$_3$	52.9	55.9
3M HNO$_3$	67.9	53.6
5M HNO$_3$	60.3	55.4
1M NaOH	27.6	37.2
1M NaHCO$_3$	10.9	17.1
1M Na$_2$S$_2$O$_3$	2.8	12.0
1M sodium citrate	14.8	22.0
3M HNO$_3$-0.05M HF[a]		
Contact 1	79.7	66.2
Contact 2	18.3[b]	10.1[b]
Contact 3	4.7[b]	6.0[b]
3M NaOH[a]		
Contact 1	64.3	63.4
Contact 2	11.1[b]	14.8[b]
Contact 3	2.8[b]	7.9[b]

(a) Three successive contacts with fresh 10-ml portions
     eluent.
(b) Percent of total activity on initial 2 g resin.

Nitric acid solutions containing small concentrations of fluoride are highly effective for eluting $^{95}Zr-^{95}Nb$; as shown by the data in Table I, three successive contacts with 3$\underline{M}$ IINO$_3$-0.05$\underline{M}$ HF solution eluted all the $^{95}Zr-^{95}Nb$ from a batch of resin. [Beneficial effects of fluoride ion in removing $^{95}Zr-^{95}Nb$ from anion exchange resins have been noted earlier.[12]] Conversely, no completely satisfactory elutriant for removing $^{106}Ru-^{106}Rh$ activity from the loaded macroreticular resin has yet been found. Of the reagents tested for this purpose, NaOH and HNO$_3$-HF solutions appear best and were used in column tests.

COLUMN TESTS

Column tests with A-26 resin extended the new solvent treatment scheme to a dynamic system; data obtained in these runs generally substantiated effects noted in the batch work.

Flow Rate Effects

Illustrative of the kinetic aspects of the new solvent treatment process is the way fission product absorption increases with decreasing feed flow rate (Figures 6 and 7, pages 20 and 21). For the conditions used, over 97 percent of both $^{106}Ru-^{106}Rh$ and $^{95}Zr-^{95}Nb$ were retained by the resin bed at a flow rate as high as 4 bed volumes/hr. At all flow rates, column effluent fission product concentrations remained essentially constant over the entire 50 to 55 column volumes, thus evidencing the great capacity of the A-26 resin.

Cyclic Load-Elution Tests

Spectacular confirmation of the ability of the A-26 resin to clean up large quantities of used Purex solvent was provided by cyclic load and elution tests. Results of the loading portion of these runs are plotted in Figures 8 and 9, pages 22 and 23. Throughout the first loading cycle (245 column volumes),

the effluent fission product content remained at a very low
level with no indication that breakthrough was approaching.
The first loading cycle was terminated and the bed eluted
at this point only because 1CW feedstock was temporarily
unavailable. Effluent product obtained in a second loading
cycle (107 bed volumes) was comparable in all respects to that
produced in the first cycle and again there was no evidence
for any breakthrough.

Throughout both loading cycles, the fission product con-
tent of the organic effluent remained approximately constant.
As a consequence, the fraction (C/C_O) of each fission product
reporting to the effluent stream varied with its concentration
in the influent 1CW feed. This effect is very evident in the
^{95}Zr-^{95}Nb results shown in Figure 9, page 23; and, to a lesser
extent, also in the ^{106}Ru-^{106}Rh data plotted in Figure 8,
page 22. Such behavior emphasizes the kinetic aspects of the
absorption process; operation at a flow rate lower than 4 bed
volumes/hr would have reduced C/C_O values correspondingly.

A significant feature of the column tests was the removal
of the yellow color from the degraded feed solvent. All the
effluent including that obtained after the first elution cycle
was water-white just like virgin solvent! During both loading
cycles, a yellow color-front slowly moved down the resin bed.
The identity of the yellow material has not yet been estab-
lished, but it presumably represents the small amount of
nitrated diluent present in the 1CW solution. The first
portions of 4M NaOH eluent appeared to remove all the yellow
color from the bed in both elution cycles.

Essentially all the ^{95}Zr-^{95}Nb but only about 64 percent of
the ^{106}Ru-^{106}Rh present on the loaded resin bed were removed
in the first elution cycle. The second elution cycle made at
40 C was not as effective as the first (made at 25 C) in that
only 71 percent of the ^{95}Zr-^{95}Nb and 55 percent of the
^{106}Ru-^{106}Rh activity on the bed were eluted. [The latter

percentage takes into account ^{106}Ru-^{106}Rh left on the bed after
the first elution cycle.] Of the ^{95}Zr-^{95}Nb and ^{106}Ru-^{106}Rh eluted
in each cycle, 97 percent and 80 percent, respectively, were
removed by the HNO$_3$-HF eluent. Poorer ^{95}Zr-^{95}Nb elution ob-
served in the second elution cycle agrees with temperature effects
noted in batch work (Table I, page 7).

The elution cycle results point up the relative ineffective-
ness of HNO$_3$-HF and NaOH solutions for removing radioruthenium
from loaded macroreticular anion exchange resin. In plant
applications, however, exhausted beds might be simply discarded
as solid waste without any need for regeneration. This favor-
able position could exist if both resin capacity and resin
stability were sufficiently great to offset economic penalties
involved in resin replacement.

SOLVENT QUALITY

New procedures proposed for cleaning up Purex process
solvent must do at least as good a job as the aqueous wash
schemes presently in use--and preferably better. Against this
standard, the merits of the macroreticular ion exchange treat-
ment stand out clearly (Table II, page 11). Particularly
noticeable are the low fission product content and Pu reten-
tion number of the resin-treated extractant; both values are
substantially lower than those for alkaline permanganate-washed
plant solvent. The Pu retention number like the analogous "Z"
and "H"[13] numbers has traditionally been considered a sensi-
tive measure of the presence of deleterious diluent and/or TBP
degradation products in used Purex process solvent. The color-
less appearance of the resin-treated TBP extractant and its
very low Pu retention number are convincing evidence that the
ion exchange procedure effectively removes these degradation

TABLE II

PROPERTIES OF ION EXCHANGE-TREATED PUREX SOLVENT

Test/Property	Plant 1CW*	Plant 100	Ion Exchange-Treated Bed Volumes[†]		Lab-Prepared Solvent
			119	195	
TBP, vol %	29.6	29.2	28.8	29.9	30.0
Color	Yellow	Yellow	Colorless	Colorless	Colorless
Density, g/ml	0.8111	0.8122	0.8108	0.8114	0.8126
Fission Product Content, Ci/liter					
^{95}Zr	90.	3.4	0.62	0.35	–
^{95}Nb	98.	2.1	0.69	0.54	–
^{106}Ru	170.	9.	1.2	3.0	–
Disengaging Time, sec	37	61	28	50	67
U Extraction, E_a^o	0.318	0.326	0.318	0.334	0.387
Pu Retention Number	2070.	50.	6.	9.	23.

* Typical plant material.
† From Run described in Figures 1 and 2.

products. It is truly a "solvent cleanup" method [in the sense defined by Blake et al.[4]] and not just a mechanism for removing radioactivity.

The other properties listed in Table II (TBP concentration, density, etc.) all confirm that ion exchange treatment neither removes nor adds components to the Purex solvent which affect its hydraulic and chemical performance as an extractant for uranium and plutonium. [Variation of a factor if two in disengaging time with the apparatus used is not regarded as significant.]

CONCLUSIONS

Application of macroreticular ion exchange resins to cleanup of used Purex process solvent has been successfully demonstrated on a laboratory-scale. A primary advantage of the ion exchange method is that it eliminates the large volumes of radioactive waste generated by present-day aqueous wash schemes. Also, quality of the product obtained by the ion exchange procedure is equal or superior to that of solvent washed with conventional alkaline permanganate solutions.

Further laboratory and/or pilot plant-scale work are needed to accurately establish resin capacity for removing fission products and various degradation products from used TBP solvent. Improved techniques for eluting radioruthenium from the loaded resin are also needed. Assessment of the economic potential of the ion exchange solvent treatment procedure is also in order.

ACKNOWLEDGMENTS

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APPENDIX

APPENDIX

A simplified version of the flowsheet used to wash the organic waste stream (1CW) from the First Decontamination and Partition Cycle is shown in Figure 10, page 24. The 1CW stream is initially given a semi-batch contact with an Na_2CO_3-$KMnO_4$ solution to remove fission products, traces, if any, of plutonium and uranium; and, to some extent, solvent degradation products. Subsequently, the solvent stream is pumped to the 10 column where contact with dilute HNO_3 removes entrained carbonate and MnO_2. From the 10 column, the organic overflows to a Turbomixer tank where it is given a final wash with dilute NaOH before routing to the 100 Pump Tank. Entrained NaOH is continuously neutralized in the 100 Pump Tank by addition of a small flow of HNO_3. The aqueous wash solutions are charged out on a scheduled basis--the frequency depending on the quality of the clean solvent.
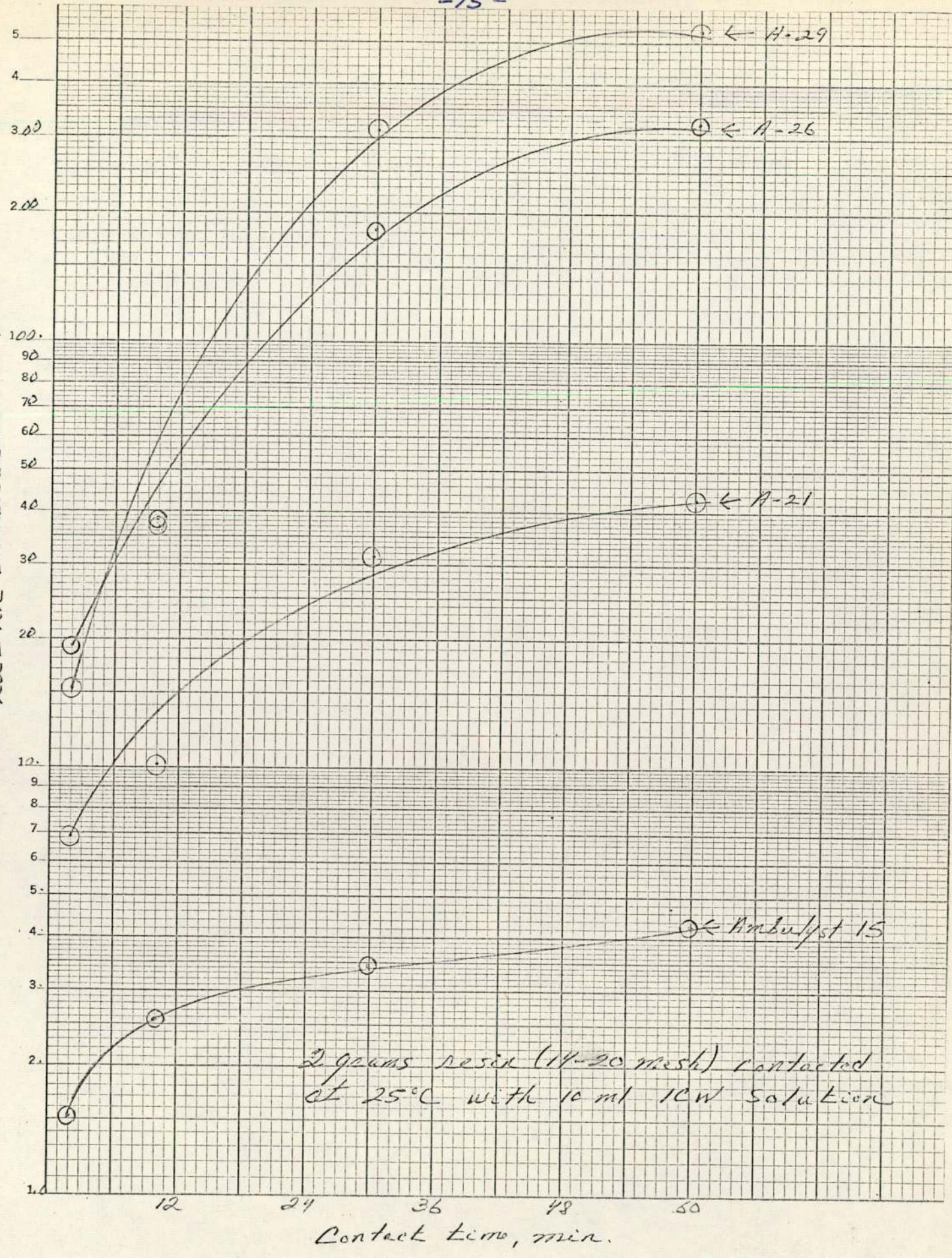
Figure 1. $Ru^{106} - Rh^{106}$ Loading Distribution Ratios - Variation With Resin Type
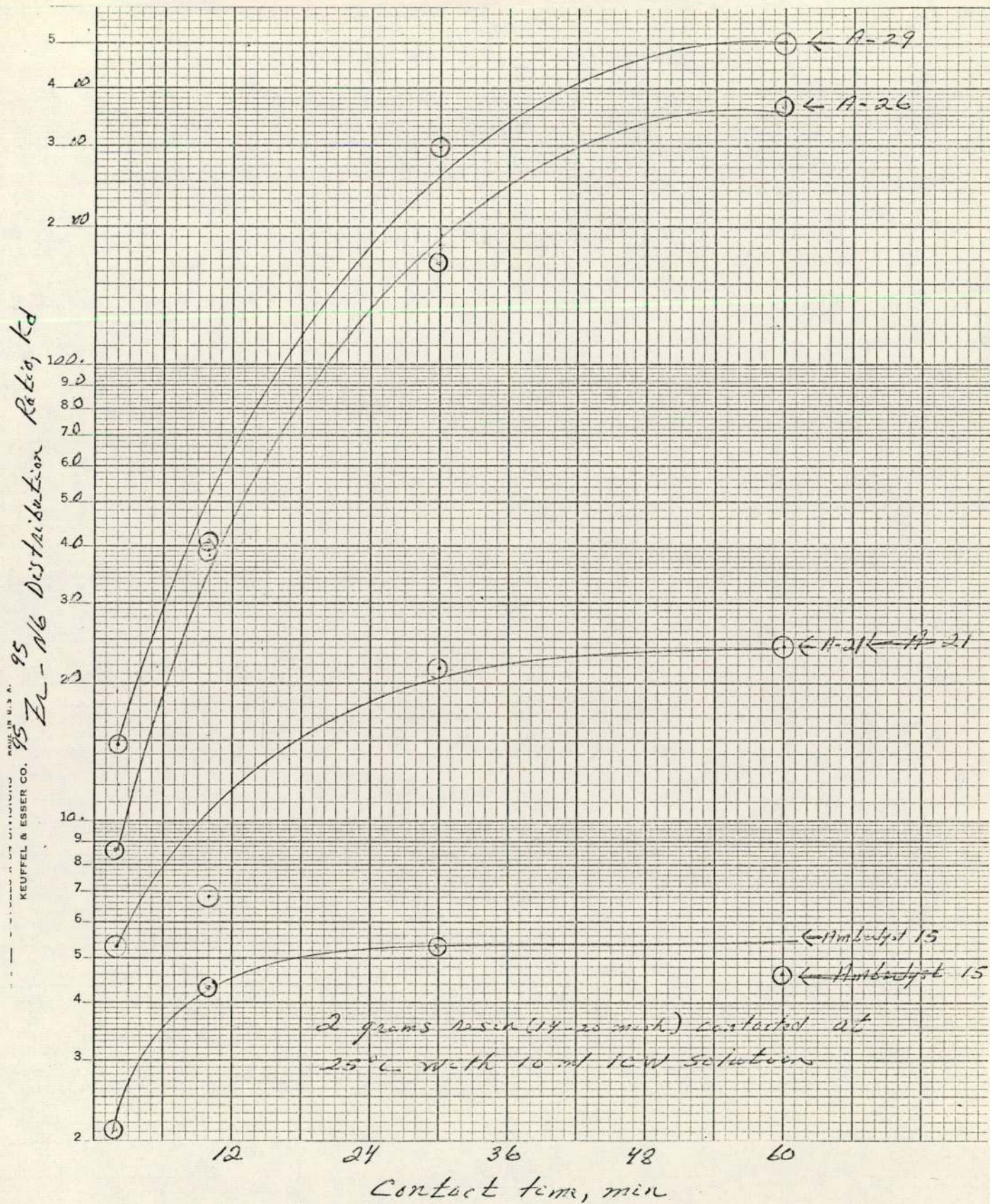
Figure 2. $^{95}Zn - ^{95}Nb$ Loading Distribution Ratios -
Variation with Resin Type

Ru — Rh Distribution Ratio, Kd

106 106 KEUFFEL & ESSER CO.

22-35 mesh, 40 °C

22-35 mesh, 25 °C

14-20 mesh, 40 °C

14-20 mesh, 25 °C

2 gram portions air-dried resin
(H⁺ form) contacted with 10 ml
10 M solution.
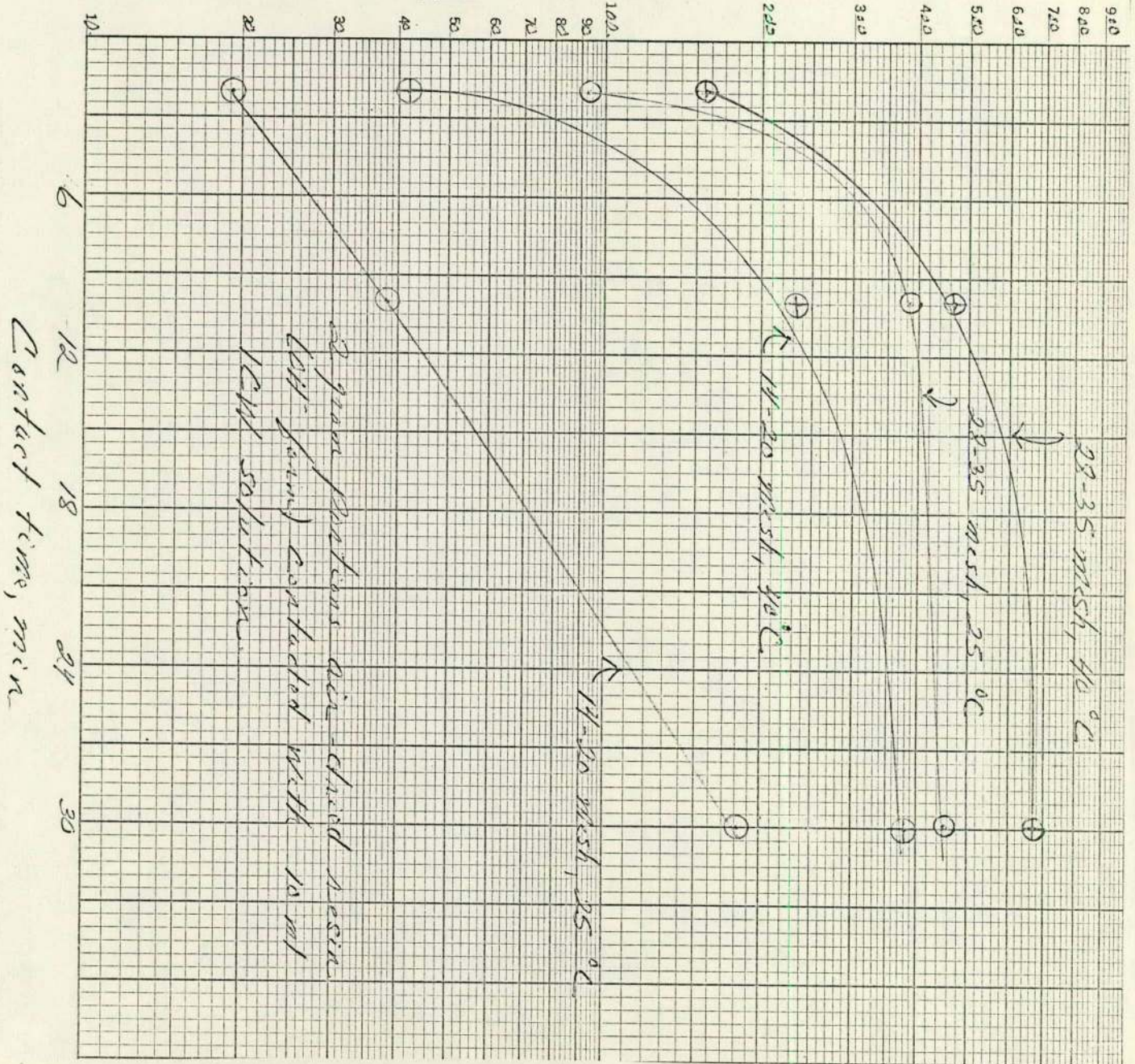
Contact time, min
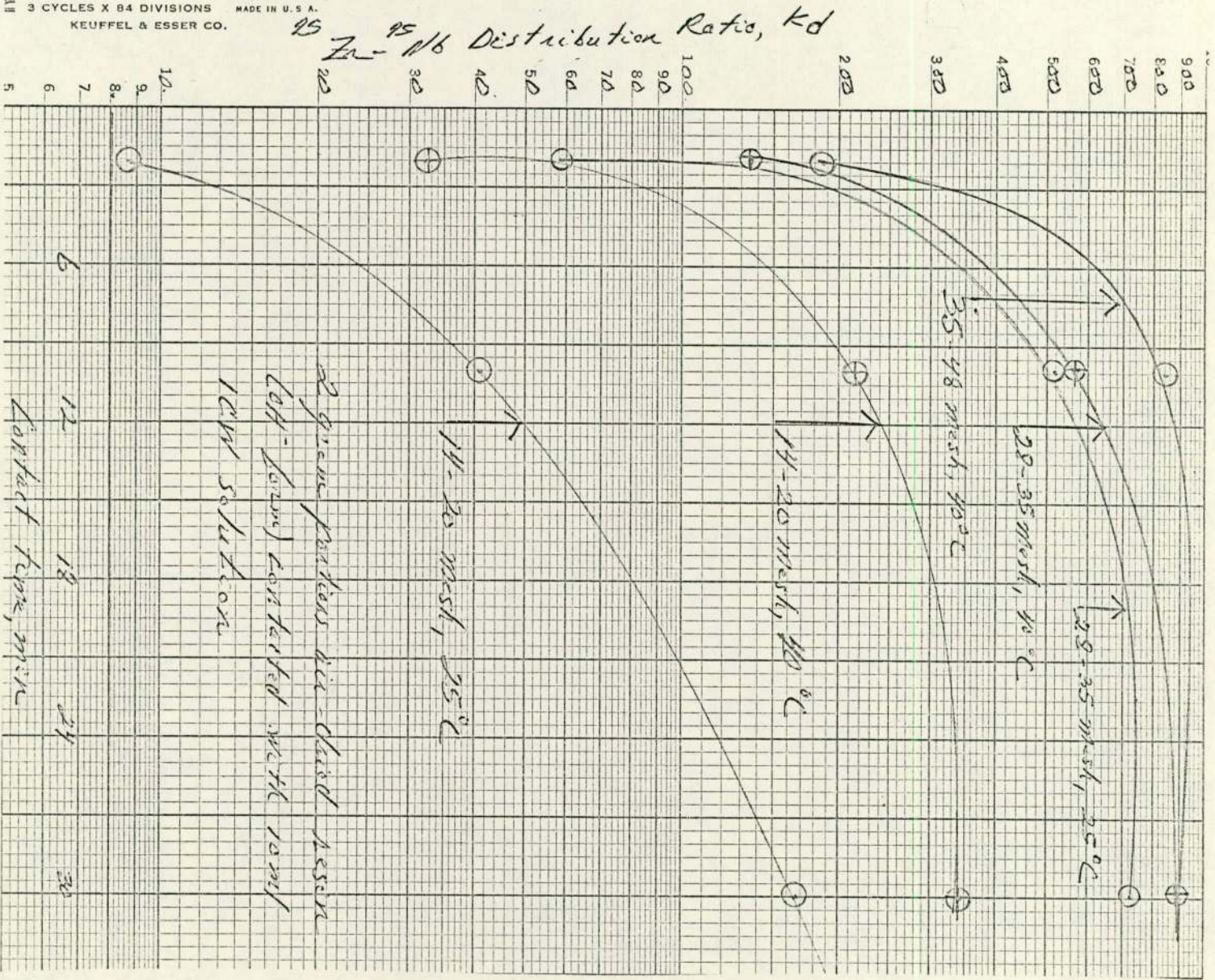
Figure 3. Loading of Ru-Rh onto Amberlyst A-26 Resin

-17-

Figure 4. Loading of $^{95}Zr - ^{95}Nb$ onto Amberlyst A-26, Resin
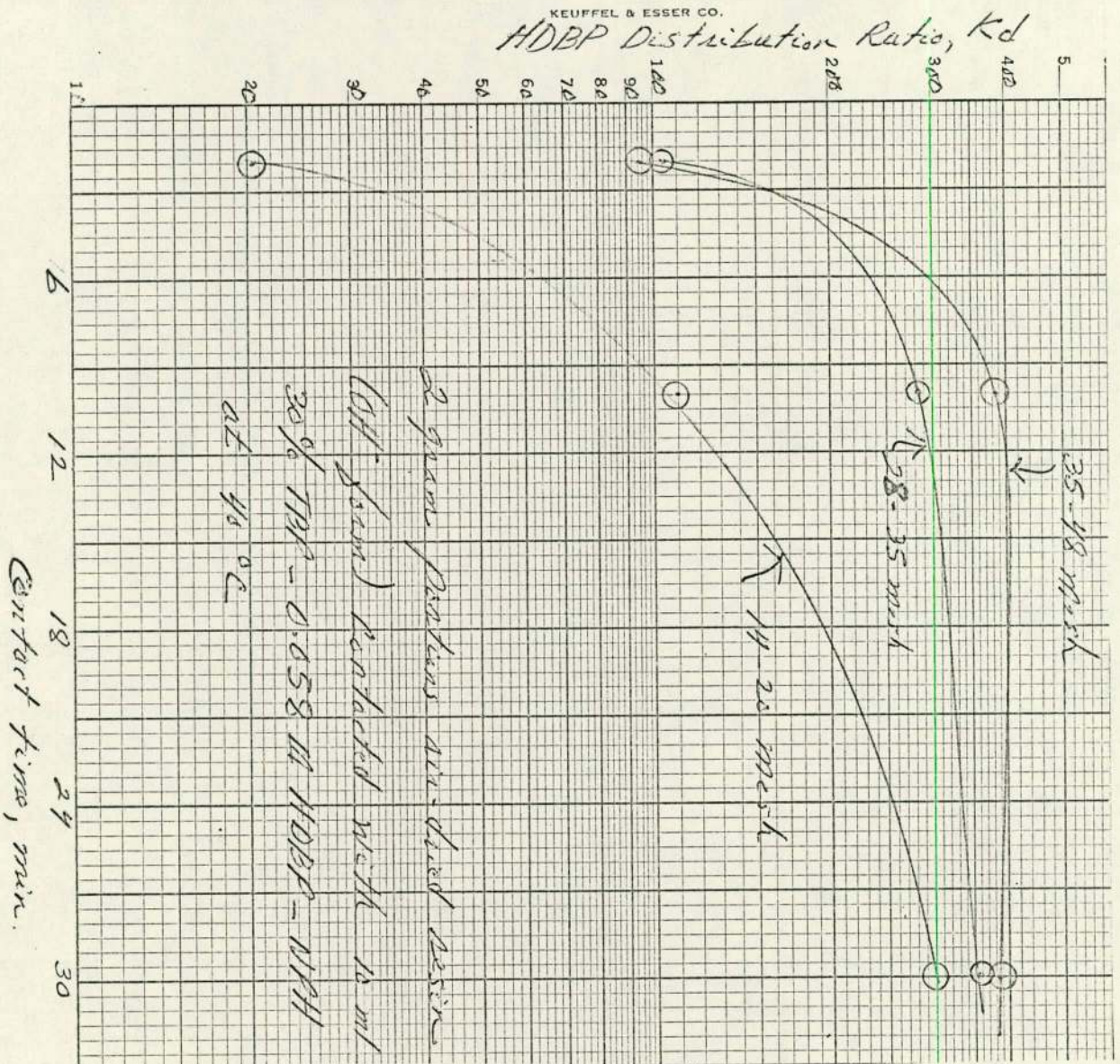
Figure 5. Loading of HDBP onto Amberlyst A-26 Resin
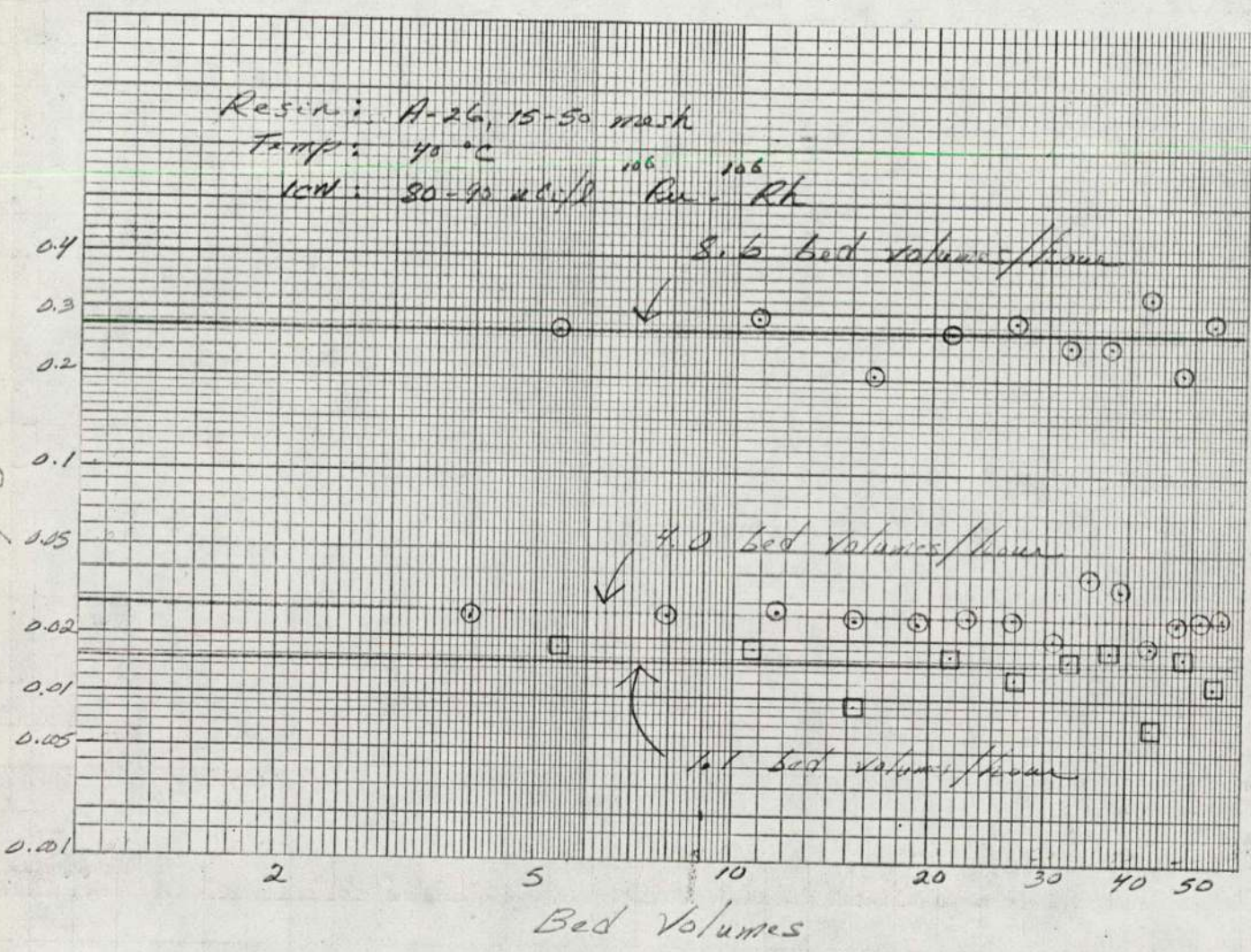
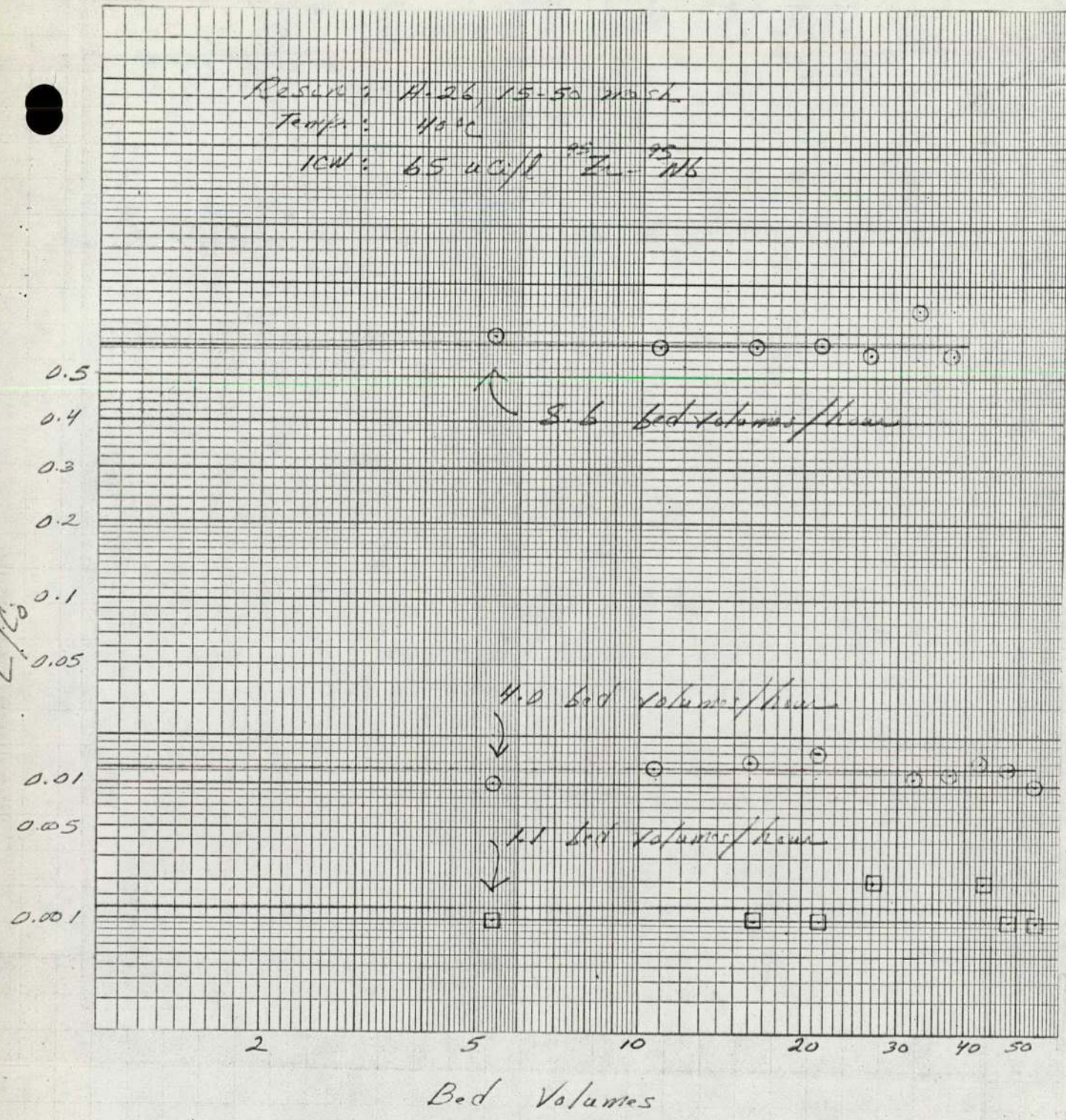Figure 6. Variation of [106]Ru Loading with Flowrate

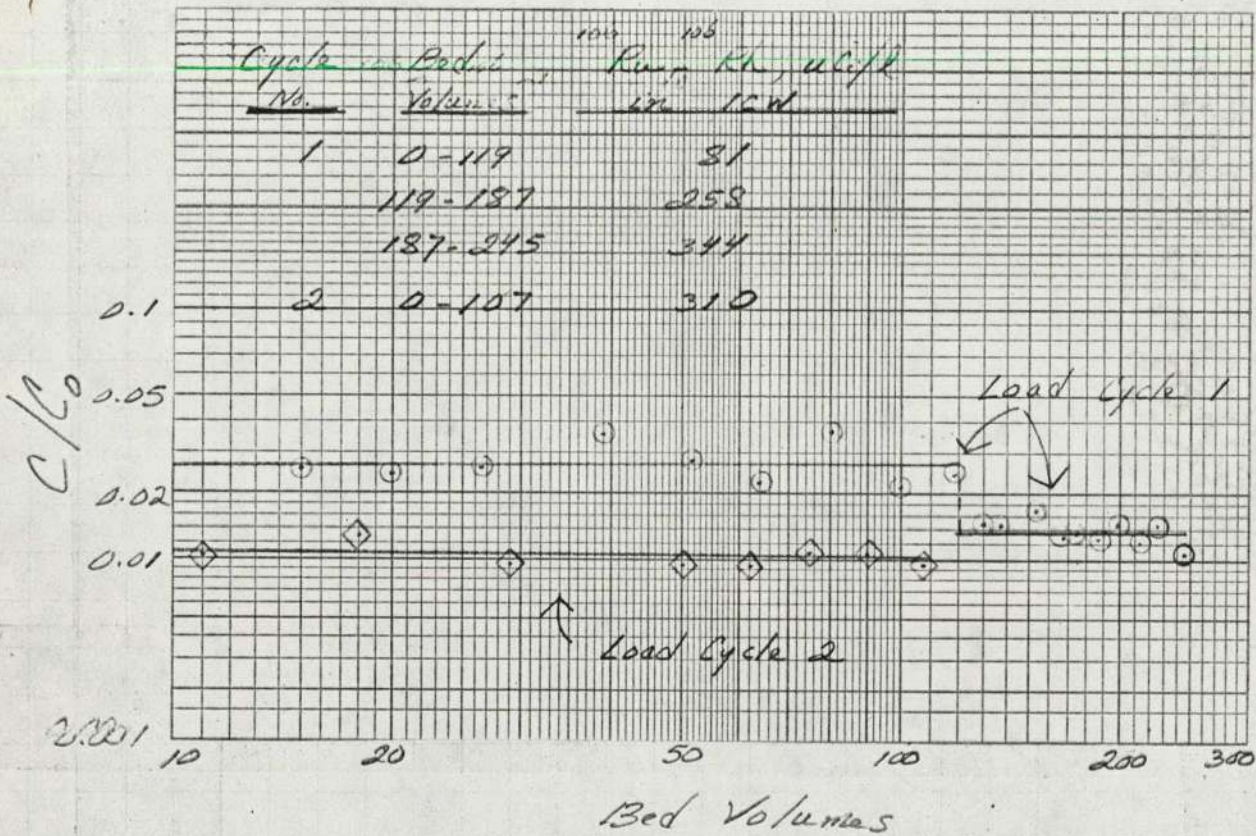Figure 7. Variation of $^{95}Zr - ^{95}Nb$ Loading With Flowrate.

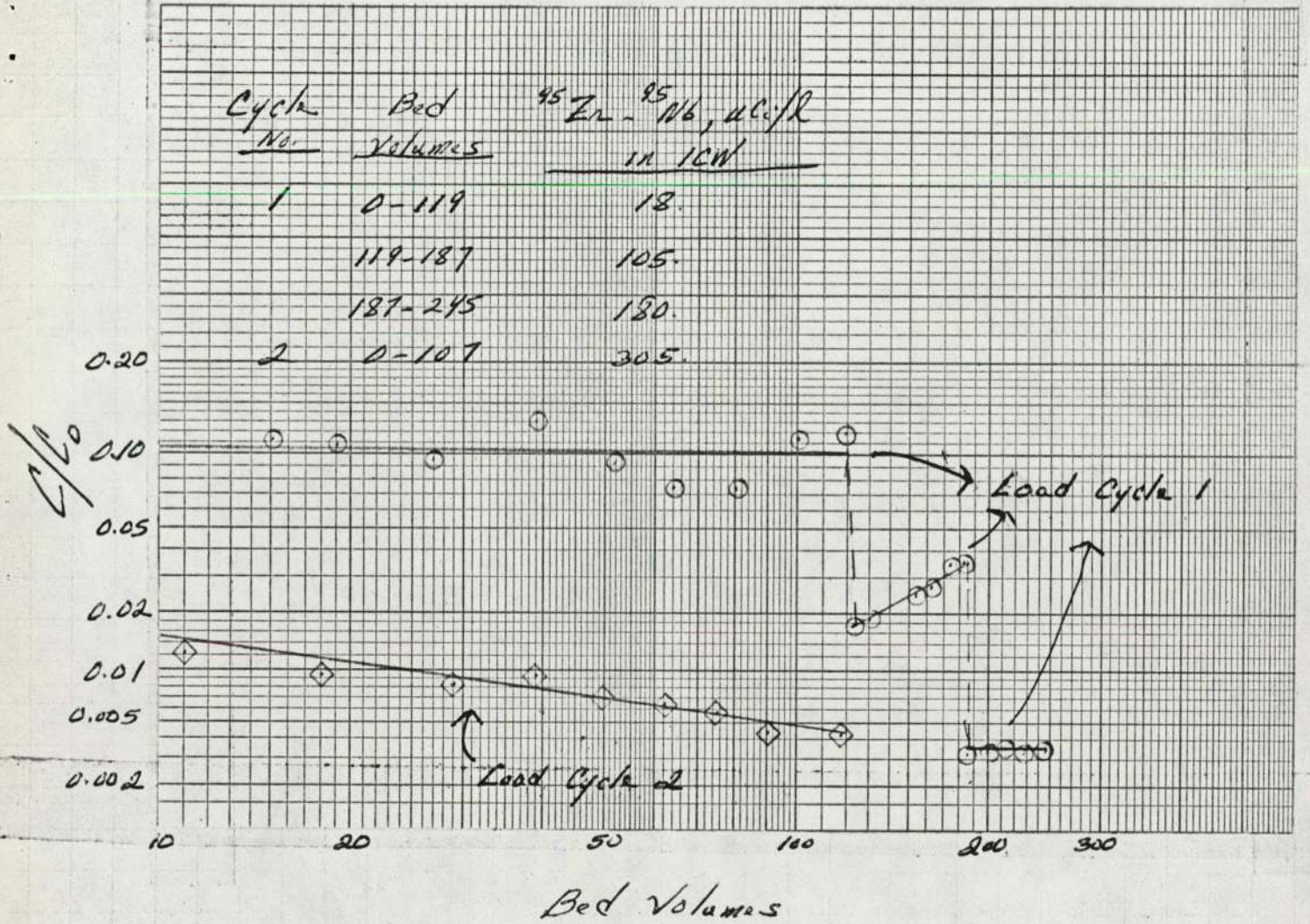Figure 8. Ru Behavior in Cyclic Loading Tests (14-50 mesh, A-26 resin (OH⁻ form) at 40°C).

Figure 9. $^{95}Zr - ^{95}Nb$ Behavior in Cyclic Loading Tests.
(14-50 mesh, A-26 resin (OH⁻ form) at 40°C)

10B - Na₂CO₃
Na_2CO_3 0.24 M
$KMnO_4$ 0.024 M
Flow 0.012

1CW
Temp. ~55°C
HNO_3 ~0.003 M
Flow 1.0

10S HNO₃
HNO_3 12.2 M
Flow ~10⁻⁴

10C
$NaOH$ 0.25 M
Flow ~0.02

100 - HNO₃
HNO_3 12.2 M
Flow ~0.001

10B'
Temp. 50°C
Composition varies
between 10B &
10BW

SOLVENT WASH TANK

10 Column

10SR
Temp. ~50°C
HNO_3 0.2 M
Flow

TURBO-MIXER SOLVENT WASHER

ORGANIC

ORGANIC

10BW
$NaNO_3$ 0.15 M
Na_2CO_3 0.13
$KMnO_4$ ~0.012
MnO_2 ~0.012
Flow 0.016

10F
Temp. 50°C
Na_2CO_3 Trace
Flow 1.0

ORGANIC

HOLD TANK

10SW
Temp. 50°C
HNO_3 ~0.3 M
Flow ~0.01

10D
$NaOH$ ~0.2 M
Flow ~0.02

WASHED SOLVENT
100
HNO_3 ~0.01 M
$^{95}Zr-Nb$ ~5 uc/l
$^{106}Ru-Rh$ ~15 uc/l
Flow 1.0

AQUEOUS WASH WASTES

10W
$NaNO_3$ 0.1 M
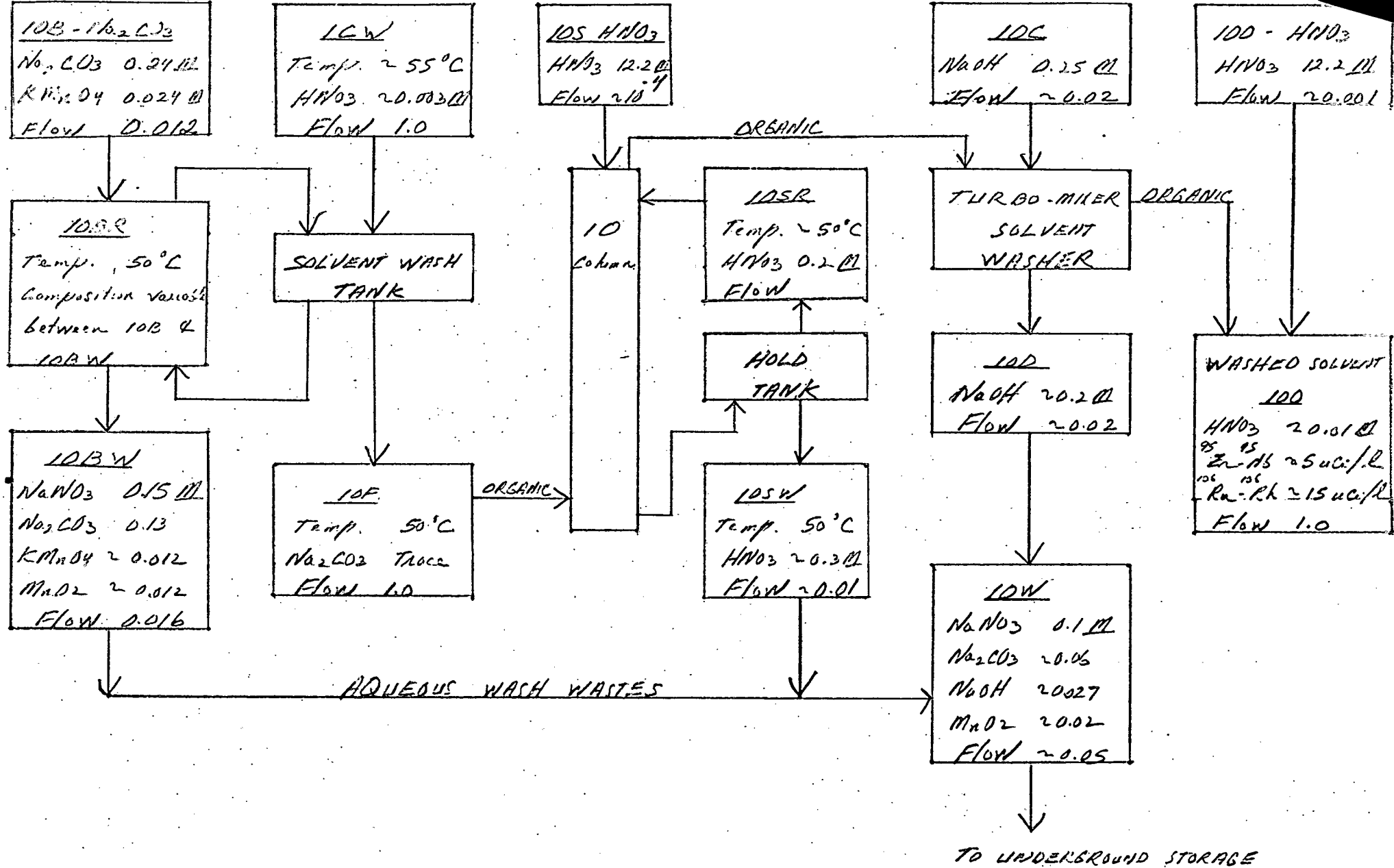Na_2CO_3 ~0.06
$NaOH$ ~0.027
MnO_2 ~0.02
Flow ~0.05

TO UNDERGROUND STORAGE

Figure 10.   HANFORD PUREX FIRST CYCLE SOLVENT WASH SCHEME

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