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HW-82771 PT1

RECOVERY OF ALUMINUM NITRATE NONAHYDRATE FROM REDOX ACID WASTE PART 1: COMPUTER STUDY

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and
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JULY 1964

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by

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RICHLAND, WASHINGTON

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NOMENCLATURE

B = backwash rate, liters/min
C = noncrystallizing product concentration, g/liter
F = feed rate, liter/min
T = total cycle time, min
t = time, min
V = vessel volume, liter

SUBSCRIPTS

B = backwash half of cycle
F = feed half of cycle
f = conditions at end of each half of cycle
i = initial condition for each half of cycle
n = vessel number (1, 2, 3, . . . n-1, n)
o = outside the system

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INTRODUCTION

Analog computer simulation early in the conceptual and laboratory or bench stages of a new process investigation can eliminate much wasted effort by indicating the importance and effect of process variables. This report describes such an analog investigation of a novel counter-current crystallization process.

Utilization of Hanford's Redox facility as part of a possible thorium irradiation program has been proposed. An important difference between uranium and thorium processing is the latter's requirement for greater amounts of salting agent. In an effort to conserve essential materials and reduce processing costs, it was proposed that the salting agent, aluminum nitrate nonahydrate (ANN), be recovered from the waste stream. Materials and equipment handling problems due to the radioactivity of the process streams preclude the use of conventional crystallization techniques and equipment. It was, therefore, necessary to develop a process more compatible with the "remote" nature of the plant.

Initial laboratory work validated the mechanics and chemistry of the proposed method, and a mathematical model was constructed and programmed on the computer. The model closely represented the physical system. As a result, a great deal of data was rapidly acquired and subsequent laboratory work will be used only to verify the most interesting cases.

SUMMARY AND CONCLUSIONS

A counter-current crystallization process was developed and demonstrated on the laboratory scale for recovering ANN from Redox waste. Equations describing this process were developed and simulated on a conventional analog computer (a Beckman EASE 1132) and an iterative analog computer (a Beckman EASE 2133). The results from both computers checked satisfactorily with laboratory data. Slow "control-relay" operating speeds associated with the EASE 1132 computer caused timing problems;

as a result, reporducible solutions were obtained only by using excessively long computer cycle times. The speed of the control relays on the EASE 2133 computer allowed solution of the problem about 600 times faster.

DISCUSSION

Process Description

The redox acid waste stream contains fission products which must be separated from ANN before the latter's recycle. Crystallization has been determined to be an efficient route for recovery; but materials handling, maintenance, and size considerations preclude the large scale use of standard techniques. The counter-current crystallization process discussed in this report avoids many of these problems. A series of vessels of equal volume equipped with agitators and heating-chilling coils (Figures 1 and 2) is used for the crystallizations. A hot HNO_3 solution made up from Redox waste and containing ANN is fed to the first vessel which overflows into the second and so on through the series. After a prescribed amount of feed has been added, the feed is discontinued and the vessels are chilled to crystallize the ANN. Fresh HNO_3 is then fed into the last vessel which overflows through the series to the first. In this way the mother liguor containing the fission product contamination is flushed from the vessels. Before the second cycle of ANN feed is introduced, the vessels are heated to redissolve the ANN crystals.

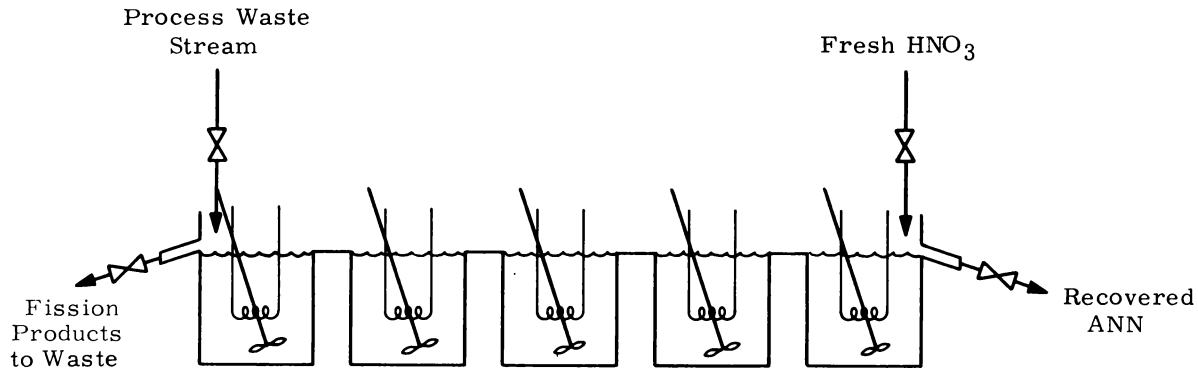

FIGURE 1

ANN Recovery System
Counter Current Crystallization

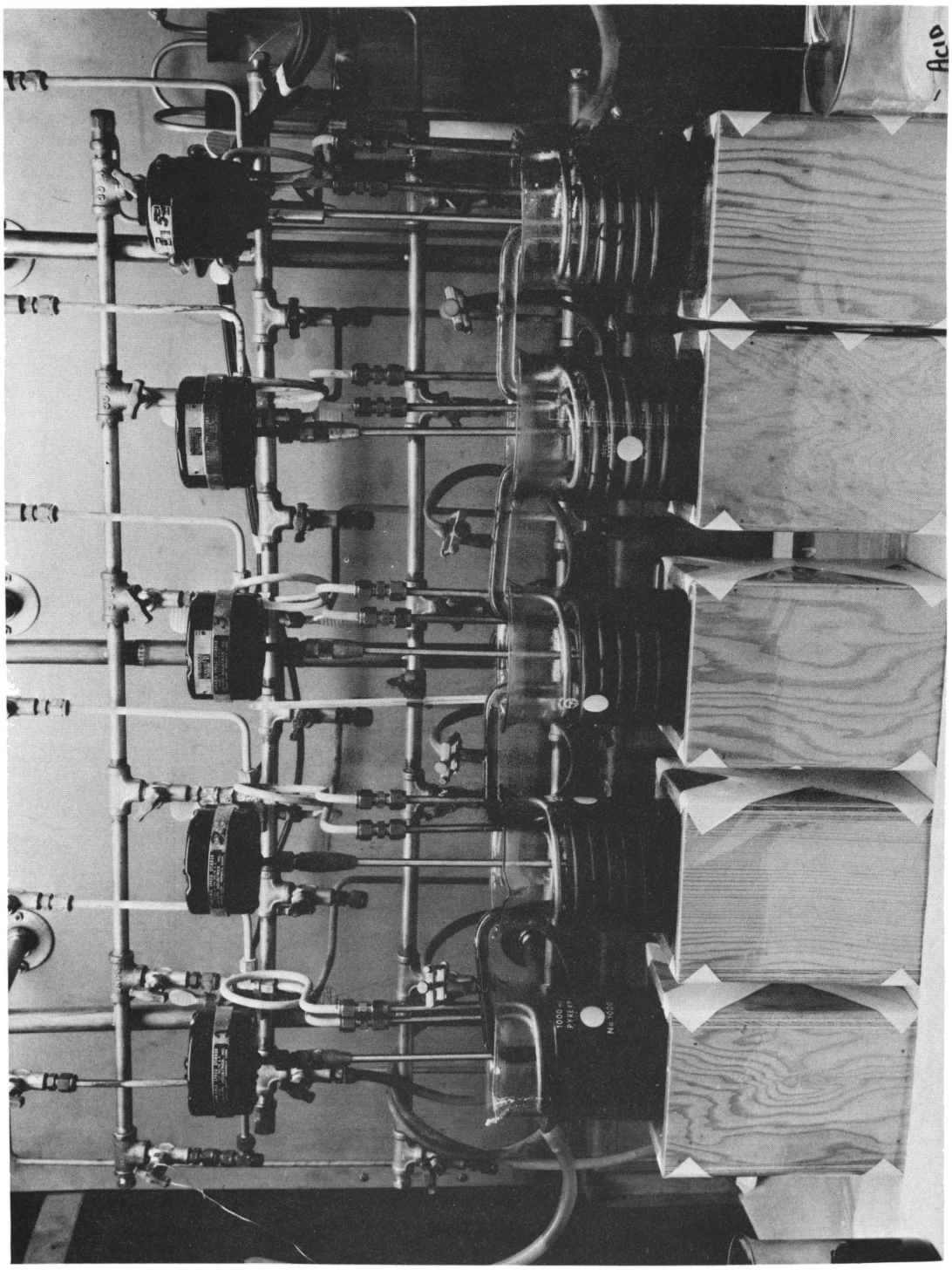

FIGURE 2

Laboratory Scale Counter-Current Crystallization Apparatus

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As the cycle is repeated, the ANN moves toward the downstream end of the series in a pulsating fashion, while the fission products (contained in the mother liquor), are moved toward the head end of the series in an alternating motion.

The basic variables affecting ANN crystallization from HNO_3 have been adequately covered by previous investigators;* however, since the process discussed here does not require the separation, per se, of mother liquor from crystals, additional variables peculiar to the system must be investigated:

- The flow rate of the ANN feed stream
- The flow rate of the backwash stream
- The volume of the vessels
- The number of vessels used.

Mathematical Model

The number of possible combinations of process variables made it impractical to develop the process entirely by laboratory investigation. However, once a mathematical model was developed that represented the process, computer analysis was possible.

The assumptions used in deriving the mathematic model are:

- All stages are of equal volume
- All stages begin at equal concentrations
- The feed rate to the first stage is constant with a mixture of constant composition
- The overflow rate to each vessel is constant and equal to the feed rate
- There is instantaneous perfect mixing in each stage.

When these assumptions are valid, the concentration of noncrystallizing salts in the first vessel may be derived from the material balance for the feed cycle (Figure 3).

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* "Meeting of the AEC Waste Processing Committee - June 26-28, 1950 - Part II - Coprecipitation as a Method of Waste Disposal," edited by J. A. Ayres, KAPL-364 PT2, April 25, 1951.

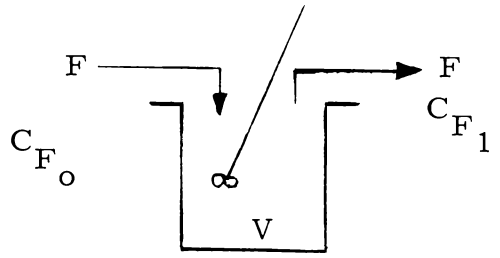

FIGURE 3
Schematic Feed Cycle Diagram

Rate of accumulation equals rate input minus rate output.

$$V \frac{dC_{F_1}}{dt} = FC_{F_o} - FC_{F_1}$$

or

$$\frac{dC_{F_1}}{dt} = \frac{F}{V}(C_{F_o} - C_{F_1}) \quad .$$

The second beaker can likewise be described as:

$$\frac{dC_{F_2}}{dt} = \frac{F}{V}(C_{F_1} - C_{F_2}) \quad .$$

The equation for the backwash cycle also can be determined from a material balance. Consider the fission product change in the last beaker during the first backwash cycle (Figure 4):

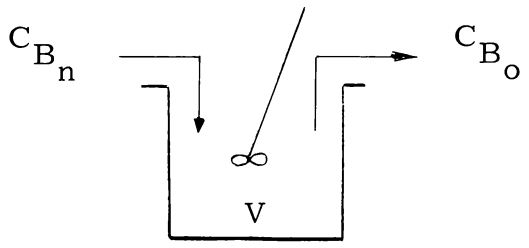

FIGURE 4
Schematic Wash Cycle Diagram

Rate of accumulation equals rate input minut rate output.

$$V \frac{dC_{B_n}}{dt} = BC_{B_o} - BC_{B_n}$$

$$\frac{dC_{B_n}}{dt} = \frac{B}{V}(C_{B_o} - C_{B_n}) .$$

The complete mathematical model for the system for both feed and backwash cycles is summarized below. The equations for the feed cycle are:

Vessel	Equation	Initial Conditions
1	$\frac{dC_{F_1}}{dt} = \frac{F}{V}(C_{F_o} - C_{F_1})$; $C_{F_o} = 100$	$C_{F_{1_i}} = C_{B_{1_f}}$
2	$\frac{dC_{F_2}}{dt} = \frac{F}{V}(C_{F_1} - C_{F_2})$	$C_{F_{2_i}} = C_{B_{2_f}}$
n-1	$\frac{dC_{F_{n-1}}}{dt} = \frac{F}{V}(C_{F_{n-2}} - C_{F_{n-1}})$	$C_{F_{n-1_i}} = C_{B_{n-1_f}}$
n	$\frac{dC_{F_n}}{dt} = \frac{F}{V}(D_{F_{n-1}} - C_{F_n})$	$C_{F_{n_i}} = C_{B_{n_f}}$

The equations for the wash cycle are:

1	$\frac{dC_{B_1}}{dt} = \frac{B}{V}(C_{B_2} - C_{B_1})$	$C_{B_{1_i}} = C_{F_{1_f}}$
2	$\frac{dC_{B_2}}{dt} = \frac{B}{V}(C_{B_3} - C_{B_2})$	$C_{B_{2_i}} = C_{F_{2_f}}$

Vessel	Equation	Initial Conditions
n-1	$\frac{dC_{B_{n-1}}}{dt} = \frac{B}{V}(C_{B_n} - C_{B_{n-1}})$	$C_{B_{n-1_i}} = C_{F_{n-1_f}}$
n	$\frac{dC_{B_n}}{dt} = -\frac{B}{V} C_{B_n}$	$C_{B_{n_i}} = D_{F_{n_f}}$

PROGRAMMING TECHNIQUES

General Remarks

The fact that it is possible to solve an iterative type of problem on a general purpose analog computer is unusual. For this reason both the general purpose analog computer (EASE 1132) and the iterative analog computer (EASE 2133) programs are presented.

General Purpose Analog Computer Program

Hanford Laboratories general purpose analog computer (EASE 1132) has a slight modification in the integrator relay control which allow control of the initial condition (IC) relay externally of the IC control bus. The hold-operate relays have not been altered to allow external control independent of the compute mode; therefore, it is possible to use integrators for track and memory purposes. It is also possible to control some of the integrators so that they do not have an IC mode (the integrators are either in the compute or hold mode).

The computer program for the EASE 1132 computer is shown in Figure 5. Integrators 2, 3, 6, 7, 10, 11, 14, 15, 16, 17, 20, and 21 do not have an IC mode and are used to simulate a system of 12 vessels. The system utilizes two different compute cycles: the first compute period corresponds to the feed cycle and the second to wash cycle. The differential relay contacts at the input of each integrator are used for switching between the feed and wash cycles, while the potentiometers associated with the inputs are for time scaling the problem by a factor of 0.1. The manual switches are for changing the number of vessels, and Amplifiers 26, 27,

IC CONTROL BUS SWITCH POSITIONS

INTEGRATORS	SWITCH POSITIONS
2, 3, 6, 7	UP
10, 11, 14, 15	UP
16, 17, 20, 21	UP
24, 25, 28, 29	UP

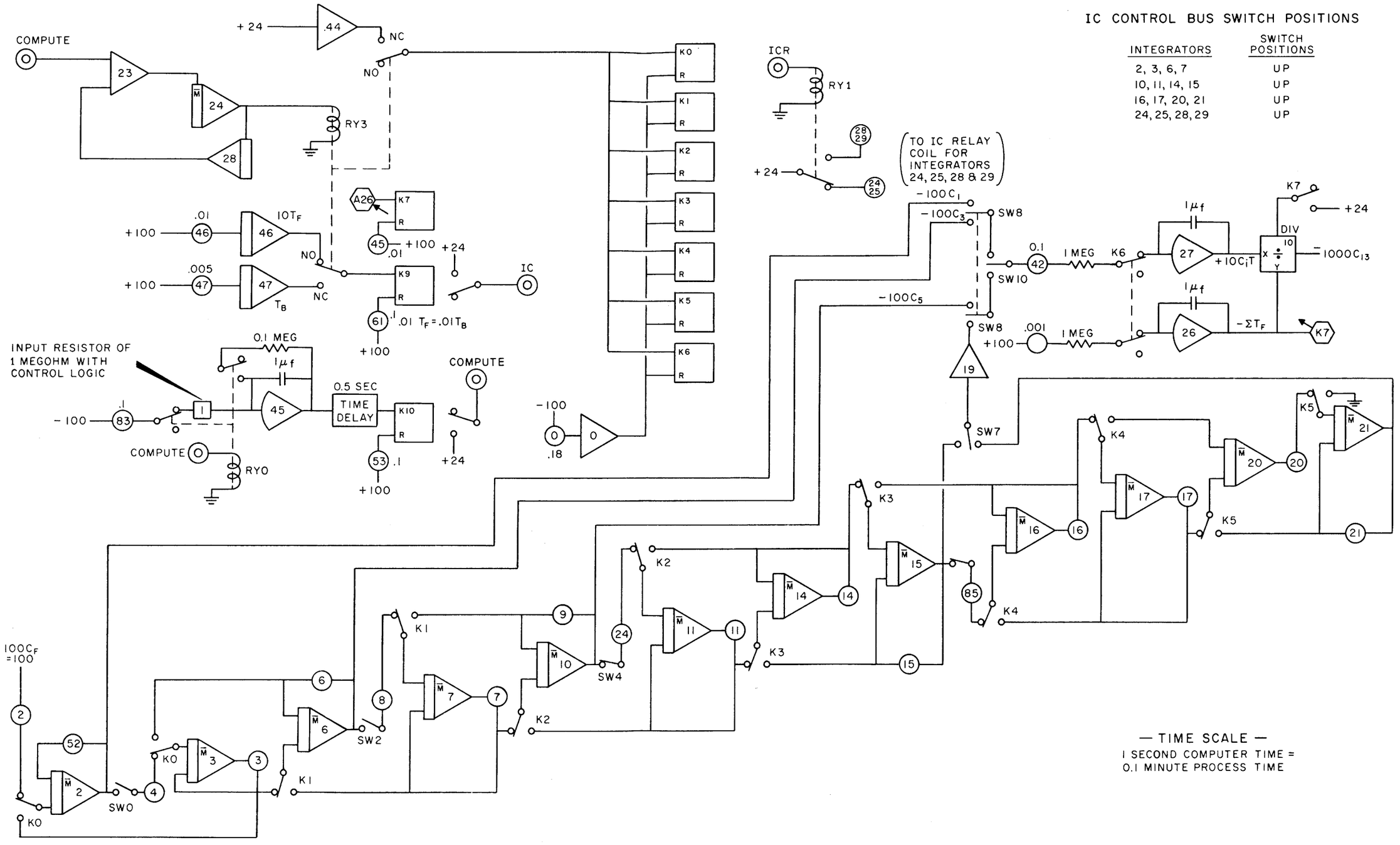

FIGURE 5

EASE 1132 Patching Diagram

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and Divider 10 are used for calculating the concentration of fission products in a collection vessel.

The two different compute cycles are obtained by using the circuitry shown in the upper left corner of Figure 5. Amplifiers 23, 24, and 28 are used to count every other compute cycle. The output of Amplifier 24 is either 0 or +24 v and is used to operate Relay 3 (RY3). Amplifiers 46 and 47 generate the feed and wash cycle time and are connected to one set of contacts from RY3. (The other set of RY3 contacts is used for operating the differential relays that switch the inputs to the simulated vessels.) The switching signal to Differential Relay 9 is taken from RY3. When Relay 9 operates, the computer is triggered into IC. The length of the compute cycle is dependent on the integration rates on Amplifiers 46 and 47, and on the switching position of RY3.

The circuit employing Amplifier 45, the time delay, and K10 is used for adjusting the time the computer is in IC. When K10 operates, the computer is switched back to compute and the cycle is repeated.

The values of F/V and B/V are taken to be unity; therefore, values for the ratio F T_F/V and B T_B/V could be selected by varying the integration rates on Amplifiers 46 and 47.

The answers produced from this simulation were accurate when a computer time scale six times faster than process time was selected. When the computer time scale was 60 times process time, timing problems associated with relay closures caused inaccuracies to occur. Therefore, to solve the problems in a reasonable time, the iterative EASE 2133 analog computer was required.

Iterative Analog Computer Program

Figure 6 presents the EASE 2133 iterative analog computer program for simulating 12 vessels. Each pair of forward (M) and reverse ($\bar{M}$) integrators are used for simulating a vessel. The forward (M) integrator computes during the compute mode (the feed cycle) and tracks during the IC mode (backwash cycle). The reverse memory ($\bar{M}$) integrator tracks during the compute mode and computes during the IC mode. Precise time control

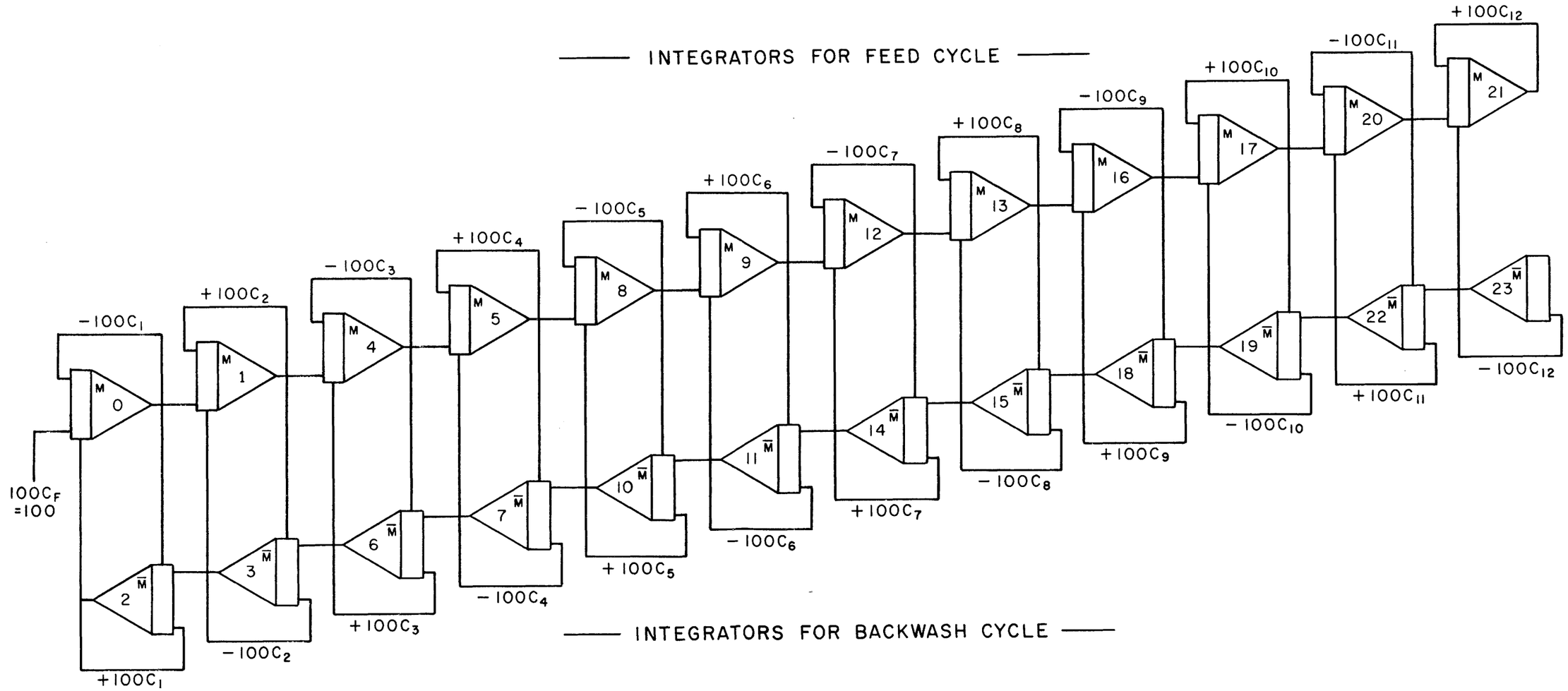

FIGURE 6

EASE 2133 Patching Diagram

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of the compute (feed cycle time, T_F) and IC (backwash cycle time, T_B) modes is accomplished by adjustment of an iterative control unit.

The simulation was time scaled to operate 600 times faster than process time. As before, values of F/V and B/V were taken to be unity.

The variables studied were F T_F/V, B T_B/V, and the number of stages used. Table I shows the data taken for a system of 12 vessels. Systems containing 2, 3, 4, 5, 6, 8, 10, 15, and 20 vessels were also simulated. Calculation for each set of variables was begun with initial conditions of zero, and as many as 30 computer cycles were necessary to achieve a steady state value for the final vessel.

An eight-channel Brush Mark 200 recorder was used for graphic readout; however, in recording the data shown in Table I, a digital voltmeter was used. At times, depending on the feed and wash cycle times, amplitude scale factors of from 10 to 100 were used on parts of the problem to maintain accuracy at noncrystallizing product concentrations.

TABLE I

STEADY-STATE DECONTAMINATION FACTOR OF FISSION PRODUCTS
IN THE LAST VESSEL FOR DIFFERENT FEED AND WASH CYCLE CONDITIONS
12 VESSELS

$\frac{FT_F}{V}$	$\frac{BT_B}{V}$ = 0.25	0.5	1.0	2.0	3.0	4.0
0.05						
0.1	2×10^{-6}					
0.15	8.13×10^{-4}					
0.20		8.0×10^{-6}				
0.25	8.35×10^{-2}	1.3×10				
0.40		1.78×10^{-2}	1.3×10^{-5}			
0.5	5.6×10^{-1}	9.5×10^{-2}	1.8×10^{-4}			
0.75	7.4×10^{-1}	4.1×10^{-1}	1.24×10^{-2}			
0.90				9.5×10^{-5}		
1.0	8.3×10^{-1}	6.1×10^{-1}	1.1×10^{-1}	3.0×10^{-4}	5.0×10^{-6}	
1.4				9.4×10^{-3}		
1.5	9.15×10^{-1}	8.0×10^{-1}	4.7×10^{-1}	1.7×10^{-2}	4.7×10^{-4}	3.5×10^{-5}
2.0			6.9×10^{-1}	1.46×10^{-1}	8.4×10^{-3}	7.4×10^{-4}
2.5			8.0×10^{-1}	3.7×10^{-1}	5.5×10^{-2}	6.5×10^{-3}
3.0			8.7×10^{-1}	5.6×10^{-1}	1.77×10^{-1}	3.1×10^{-2}
3.5				6.9×10^{-1}		
4.0				7.8×10^{-1}	5.0×10^{-1}	2.1×10^{-1}
5.0					7.1×10^{-1}	4.8×10^{-1}
7.0						8.1×10^{-1}

RESULTS

A comparison of laboratory data obtained on the equipment shown in Figure 2 and those obtained from the computers is shown in Figure 7. The experimental and analytical effort required to obtain each datum point dictated that only the minimum number of points required to establish a trend be obtained. Thus the particular case shown was not carried to equilibrium. Figure 7 shows that in spite of the expected (and observed) experimental and analytical variation, the process was very closely duplicated by the analog system.

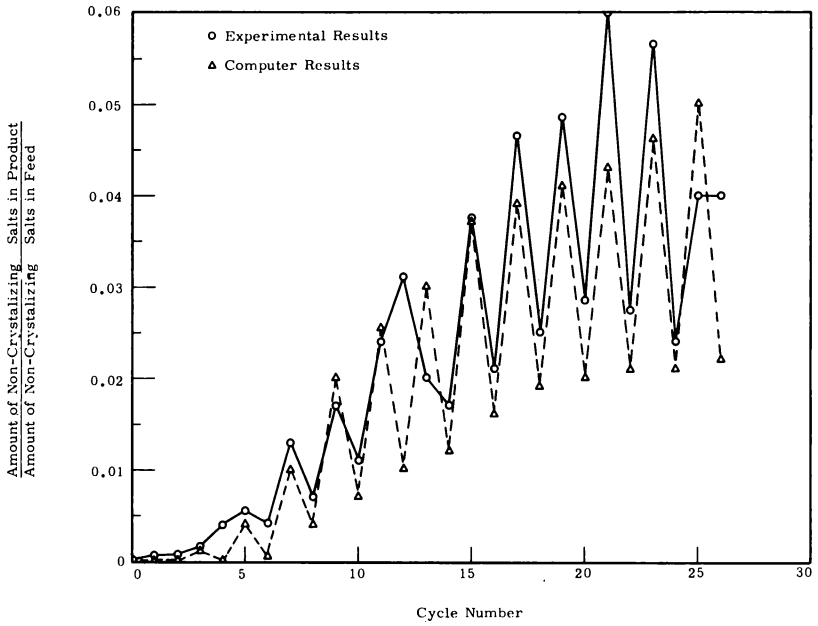

FIGURE 7

Comparison of Computer and Experimental Results
Case for 5 Stages, FT/V = 0.5, BT/V = 0.8

Typical results of the computer investigations are presented in Table I for 12 stages, while a typical plot of the same data is shown in Figure 8. Additional data were obtained for 1, 2, 3, 4, 5, 6, 8, 10, 15, and 20 stages.

ACKNOWLEDGEMENTS

Gratitude is expressed to R. A. Watrous for his assistance in defining the mathematical model for the system, and to G. R. Taylor for his assistance in designing the control circuitry for the EASE 1132 computer program.

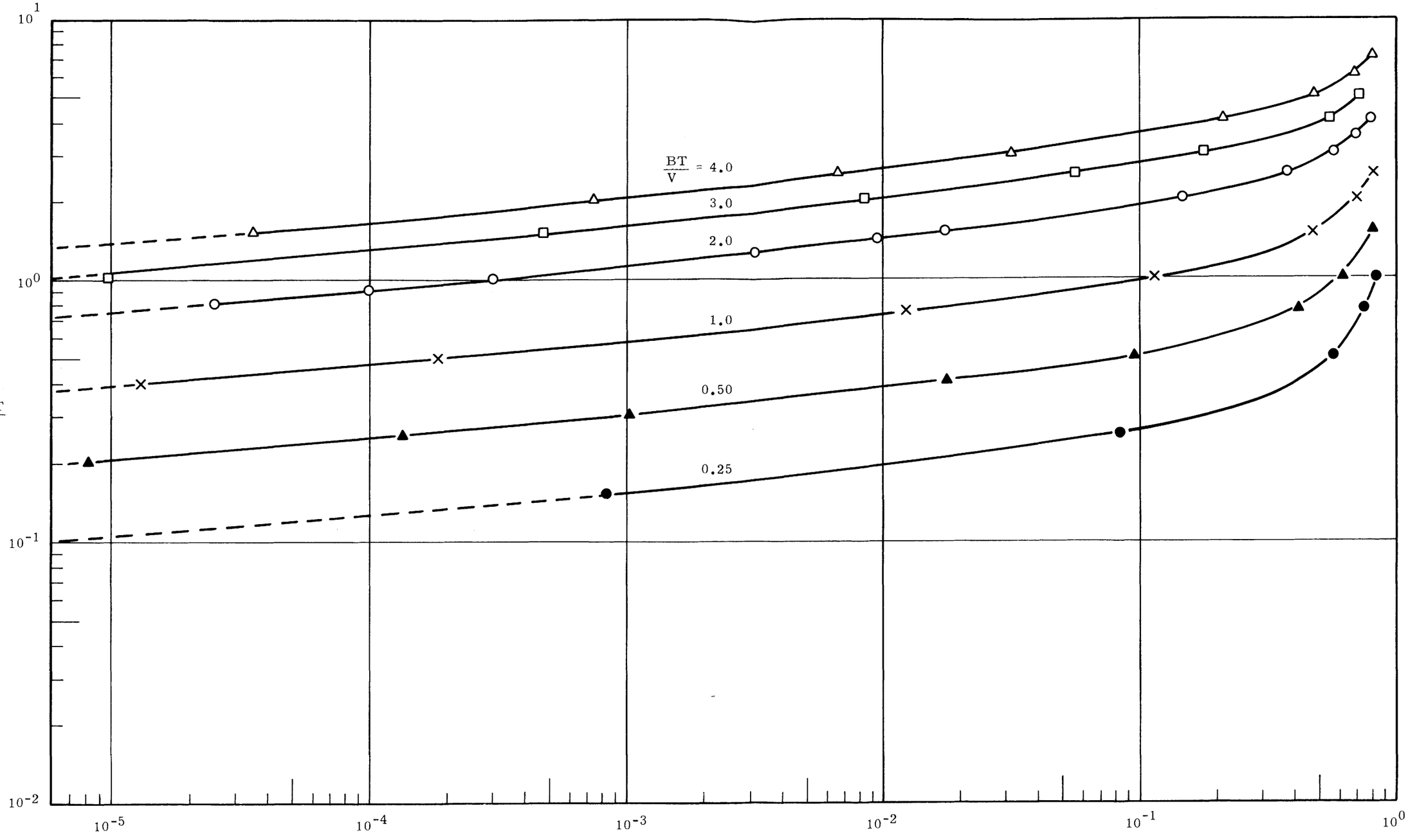

FIGURE 8

ANN Crystallization Parameters for 12 Vessels

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