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PUREX PULSE COLUMN STUDIES - 1960

by

G. Jansen and G. L. Richardson

Chemical Engineering Development
Chemical Development Operation
Chemical Research and Development
HANFORD LABORATORIES OPERATION

February 22, 1961

HANFORD ATOMIC PRODUCTS OPERATION

RICHLAND, WASHINGTON

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INTRODUCTION

A series of pilot plant runs was made in the 321-Building Demonstration Unit during the year to define new cartridges which would increase capacity in the Purex 1BX, 2A, 1C and 2E Columns and eliminate plastic cartridge failures in the HA Column scrub section, the HS Column and the 2A Column. Assistance was given in determining the cause of unusually low instability thresholds in the 1C and 2E Columns.

SUMMARY

The following cartridge designs are recommended as those which appear most favorable:

Columns	Continuous Phase	Plate Type	Hole Diam., In.	% Free Area	Plate Spacing, In.	Nozzles Point	Remarks
HA Scrub	Organic	Nozzle	0.13	6	3	Down	23% free area nozzle plates with 3/16-inch holes would be recommended for a unidiameter HA Column.
HS	Organic	Nozzle	0.125	10	2	Down	
1BX	Aqueous	Nozzle	0.188	23	4	Up	No pronounced advantage over present 33% free area sieve plate cartridge.
1C, 2E	Aqueous	Nozzle	0.188	23	2 top half 4 bottom half	Up	
2A	Organic	Nozzle	0.188	23	3 Scrub 2 Extraction	Down	

At this date, the recommended HS, 1C and 2E cartridges are being Plant tested and firm plans have been made for installation of the 2A[redacted] cartridge[redacted]. The recommended 1BX cartridge offers only a rather nebulous advantage over the currently-installed cartridge, and no plans have been made to change this cartridge. Its advantage lies in the superior ability of nozzle plates to redisperse the organic phase if the plates become preferentially organic-wet.

During the year, low flooding thresholds were noted in the Purex Plant 1C and 2E Columns. Pilot plant studies definitely traced their cause to the water from the cation exchangers of the water demineralizer. The precise impurity was not identified, but circumstantial evidence points to the cation resin itself. Addition of pulverized resin beads to the pilot plant 1CX definitely lowered the flooding threshold, and in the Purex Plant the instability tendency became invariably more pronounced within hours after the cation exchanger had been regenerated. Replacement of the former resin (Nalcite HCR) with more stable Nalcite HCR-W has apparently decreased the instability problem.

EQUIPMENT AND FLOWSHEET

The pilot plant studies were carried out in conventional 3 and 4-inch-diameter Pyrex glass columns using the available demonstration unit auxiliary equipment. Cartridge heights were usually 9 feet but ranged from 6 feet for the 2A[redacted] scrub section[redacted] to 12 feet for the 2A extraction section and 15 feet for the final 1BX Column.

Flowsheet conditions approximated those presented in HW-64450[1]. Significant variations are noted in the text and tables.

EXPERIMENTAL RESULTS

HA AND HS SCRUB SECTION STUDIES

The presently-installed "zebra" cartridges have been demonstrated to have a useful life of only 8 to 12 months in HA Column service because of a combination of radiation and flexure failure. Consequently, pilot plant efforts were devoted to seeking either a plastic-free cartridge or a cartridge with the plastic in a more stable form, such as Raschig rings.

Three types of cartridges were tested: (1) the present Plant zebra cartridge, (2) a modified zebra cartridge composed of stainless steel sieve plates with 6-inch to 12-inch-high void spaces every 2 to 3 feet for insertion of plastic packing, and (3) nozzle plate cartridges. They were evaluated by comparing their flooding frequencies with those of the extraction section at the same uranium throughput

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(1) R. G. Geier and J. P. Duckworth, Purex Plant Flowsheet for High Capacity Study, March 28, 1960.

rates, their abilities to remove MnO_2 from the solvent phase (formed in situ at the feed point by injection of a saturated $KMnO_4$ solution), and their general appearance at optimum extraction section frequencies. Most of the runs were made with a dual-diameter dual-purpose column (9-feet-high, 4-inch-diameter scrub section; 6-feet-high, 3-inch-diameter extraction section), but several were made with a unidiameter 3-inch-column. The organic phase was continuous.

The [redacted] and HAO streams were omitted from the flowsheet for simplicity, and the organic rate was adjusted to produce an HAP containing 0.29 M uranium at a scrub A/O flow ratio of 0.15. Mistron* and bentonite were added separately to the scrub in several runs to test their effect on flooding and MnO_2 scrubbing.

Details of the cartridges and the results of the runs are given in Table I. In summary, none of the other cartridges tested provided as satisfactory a match for the extraction section cartridge as the Plant zebra cartridge in a dual-diameter column. The limited number of runs made with the modified zebra cartridges in which the plastic plates were replaced by short void sections, polythene Tellerettes**, or fluorothene Raschig rings indicated that such cartridges could not be made to operate with a stable zebra emulsion. The plastic zones were about 2 feet apart, however, and closer spacing might have produced a better zebra. The scrubbing effectiveness in removing MnO_2 also appeared inferior with these cartridges.

Both 6 and 10 percent free area nozzle plate cartridges (1/8-inch holes) appeared to be suitable replacements for the zebra cartridge. Both provided high capacity and good MnO_2 scrubbing at frequencies within the range of the Plant pulsers. The flooding frequencies of both cartridges were greater than the corresponding flooding frequency of the extraction section (dual-diameter column), but the operating range of the 6 percent free area cartridge for good MnO_2 scrubbing fell within the normally-used frequency range of the extraction section. The quality of MnO_2 scrubbing with the 10 percent free area cartridge was borderline at the maximum stable extraction section frequency. For this reason, the 6 percent free area cartridge is recommended for the HA scrub section while the 10 percent free area cartridge, with its lower pressure drop and more stable operating characteristics, is recommended for the HS Column. Flooding curves for both cartridges are presented in Fig. 1.

Although both cartridges were tested with 2-inch plate spacing, 3-inch spacing is recommended for the 6 percent free area cartridge to reduce the pressure drop. The HA Column with this cartridge should operate without cavitation at 95 cycles/minute.

The effect of Mistron and bentonite addition to the HAS was determined during the tests with zebra cartridges. The additives generally improved the column operating stability, primarily by preventing the development of organic-in-aqueous dispersions. Either additive at about 20 ppm in the HAS destroyed the zebra

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* Trademark: Sierra Talc and Clay Company
** Trademark: Harshaw Chemical Company

dispersion within minutes. Surprisingly, the ability of the zebra cartridge to remove MnO_2 from the solvent seemed to be enhanced by Mistron addition. The apparent improvement may have been due to the elimination of the alternate aqueous-continuous layers which tended to accumulate MnO_2 and facilitate its backmixing up the column. More detailed results of the Mistron tests are presented in HW-63791.[2]

1BX COLUMN STUDIES

The throughput capability of a 4-inch-spaced 23 percent free area nozzle plate cartridge (3/16-inch holes) was compared with that of the present Plant cartridge, which uses 4-inch-spaced 33 percent free area stainless steel sieve plates with 3/16-inch holes. Both cartridges were 15 feet high and contained 28 percent free area louver plates at 4, 8, and 12 feet from the bottom. The aqueous phase was continuous.

There was very little difference between the cartridges using a rough simulation of the 1BX feed. The sieve-plate cartridge had a slightly greater range of operable frequencies at any given throughput, but the maximum capacities were virtually the same. Details of the runs are given in Table 2 and Figure 2.

Both cartridges were found to have a few organic-wet plates. The effect of these plates was to coalesce the organic emulsion to form large drops which would channel through several more plates before becoming redispersed. These plates appeared to act as safety valves to prevent the buildup of heavy, unstable emulsions. However, if a majority of the plates were to become organic-wet, the extraction efficiency would undoubtedly suffer. For this reason, nozzle plates with the nozzles pointed up would be slightly preferable to sieve plates because of their demonstrated ability to redisperse either phase in HA and 1C service. This ability was observable to a small degree in the 1BX Column in that the coalesced organic globules were somewhat smaller than those produced by the organic-wet sieve plates.

1C COLUMN STUDIES

The instability of Purex Plant 1C and 2E Columns at frequencies and flow rates much lower than had been anticipated from previous pilot plant studies led to an attempt to develop 1C Column cartridges operable at high flow rates with maximum stability of operation and yet having good extraction efficiency at both low and high flow rates. A complicating factor in the cartridge evaluation was the poor quality of the Purex Plant water with respect to solvent extraction column performance. Several nozzle plate cartridges and sandwich cartridges (Figures 3 and 4) were tested in an aqueous continuous, 3-inch-diameter column with 321 Building steam condensate. Results shown in Table 3 and Figures 5 through 9 indicated that the N-4 cartridge at 1.0 inch pulse amplitude was the most suitable cartridge tested from an overall standpoint. Its maximum operating capacity was 2000 gph/ft^2 at 60 C.

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(2) G. L. Richardson, 'Crud' Scrubbing with a Purex-Type Zebra Cartridge, February 4, 1960.

Observation of an apparent cycle in the performance of the Purex Plant 1C Column indicated that the poor performance might be related to regeneration of the cation bed in the water demineralizer unit. To confirm this, water from several sources was tested in the pilot plant at 1270 gph/ft^2 and 60 C. Performance of the Plant cartridge at 0.5-inch amplitude was very poor with water from the demineralizer unit after cation bed regeneration. Tests with the N-4 cartridge indicated an instability threshold (defined as the point where tight emulsion began to form in the bottom half of the column) of about 32 cycles with this water compared with 55 to 60 cycles with 321 Building steam condensate and Purex sanitary water before demineralization. Completely deionized water from the Plant weak base anion bed performed very poorly after cation bed regeneration and improved gradually until the next regeneration. Further tests indicated that finely ground resin, 5 to 20 ppm silica, and the technical grade sulfuric acid used in the plant for regenerating the cation resin all had significant adverse effects on the instability point, the greatest effect being obtained with the resin fines. Analyses of water from the plant revealed that there was some weak acid in the water leaving the demineralizer, a large part of which was CO_2. No other impurities above 1 ppm were identified. Replacement of both the cation and anion beds in the demineralizer resulted in improved operation in both the pilot plant and Purex Plant.

Plant Cartridge

The Plant cartridge consisted of 4-inch-spaced, 10 percent free area stainless steel nozzle plates with 1/8-inch holes, nozzles pointing down. Tests of this cartridge at 0.5-inch amplitude and 60 C gave a maximum capacity of about 2000 gph/ft^2 with relatively good recovery from column upsets (Figure 5). However, there was a tendency toward cyclic instability at high frequencies and flow rates above about 1600 gph/ft^2. This tendency was observed at even lower frequencies and flow rates in the Purex Plant. Operation at 1.0-inch amplitude gave about the same ultimate capacity, though at lower frequencies. However, cycling occurred over most of the region of operation, with stable operation possible only at flow rates below 1250 gph/ft^2.

Graded Nozzle Plate Cartridges

A survey of previous work(3) revealed that a nozzle plate cartridge with 1/8-inch holes, 23 percent free area, nozzles down, and two-inch plate spacing had promise as a high capacity cartridge. A similar cartridge (N-1) with 3/16-inch holes was tested at 1-inch amplitude and 60 C (Figure 6). The capacity was about 1700 gph/ft^2; however, cycling began in the lower half of the column at lower flow rates. It was felt that the emulsion in this part of the column was too tight, so the cartridge was modified by increasing the plate spacing to 4 inches in the bottom half of the cartridge. Cycling with this cartridge (N-2) was decreased substantially at both 0.7-inch and 1.0-inch amplitudes but some local cycling still occurred at the plate spacing discontinuity at high flow rates. The column capacity did not increase significantly. Cartridge N-3 with 4-inch plate spacing in the lower two-thirds of the column did not exhibit improved cycling or capacity performance over cartridge N-2.

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(3) Nicholson, G. A., Purex Pulse Column Studies with Hydrocarbon Diluent, HW-40550, Volume 2, Figure 18c, Page 39, July 24, 1956 (SECRET)

A tendency for nozzle plates to become organic wet had been suspected in the Purex Plant. Pointing the nozzles up might decrease the influence of this on column operation, since the organic droplets wetting the upper surface of the plate would not pass through the nozzle and during the upward pulse the organic would not be able to cling to the sharp nozzle edge but would be dispersed by jetting action to a region away from the plate. Cartridge N-4 (similar to cartridge N-3 but with nozzles turned up) operated at 2000 gph/ft^2 with top interface and 1.0-inch amplitude at 60 C (Figure 7). Although the efficiency and the maximum capacity of the N-4 cartridge were about the same as for the Plant cartridge, the range of stable operating frequencies was much wider, with only a narrow instability range. It was easier to start up and more stable toward upsets than the Plant cartridge. In overall performance this was the most suitable cartridge tested.

Sandwich Cartridge

Sandwich cartridges are composed of closely-packed-plate configurations between which are large spaces. Each large space is bisected by a sieve plate. The close-packed configurations or "sandwiches" contain several plastic plates between two nozzle plates which point away from the plastic. These cartridges tend to form much coarser emulsions than ordinary sieve plate or nozzle plate cartridges because the sandwich, wetted by the dispersed organic phase, promotes coagulation of the small organic droplets which are often the capacity limiting factor. Consequently they may be useful in situations where higher capacity is desired and in which the column efficiency has been more than adequate.

The S-2 cartridge previously reported in HW-56450[4] was tested at 50 C and 0.7 inch amplitude with a sandwich at the bottom of the column. In several tries it could not be operated above 850 gph/ft^2. The sandwich prevented dispersion of the organic phase at the bottom of the column, creating a phase inversion which hindered phase counterflow. When the cartridge was shifted so that a sieve plate was at the bottom, the cartridge operated stably to a maximum flow rate of 1350 gph/ft^2. It was felt that non-alignment of holes in the organic plates of the sandwich caused high resistance to phase counterflow, so cartridge S-5 with the holes aligned was tried. There was no detected difference in column efficiency, flooding frequency or column capacity. The HTU for both cartridges was less than 1.5 feet at operable frequencies.

Since the 23 percent free area sieve plate with 1/8-inch holes appeared to be promoting phase dispersion too well, it was replaced in cartridge S-6 by a 33 percent free area sieve plate with 3/16-inch holes. Simultaneously the pulse amplitude was rasied to 1.0-inch. This resulted in an increase in column capacity to about 1700 gph/ft^2 at 50 C. Raising the temperature to 60 C increased the capacity to 2100 gph/ft^2 (Figure 8). Unlike most other cartridges, the lower flooding frequency is higher than that determined from the pulsed volume. Excluding the solution trapped between the organic plates of the sandwich at the end of each pulse from the effective pulse volume would move the pulse volume line to closer agreement with the data.

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(4) DeMier, W. V., Purex 1C-Column Studies Part II, Cartridge Modification, October 6, 1958. (SECRET)

Since a sandwich might be supplied as a single, very thick plastic plate, the spaces between the plastic plates were removed in cartridge S-7 (Figure 9). At a pulse amplitude of 1.0 inch and 60 C this cartridge operated at 2300 gph/ft^2, the highest capacity obtained with any cartridge tested in this series. HTU's were less than 1.5 feet with both S-6 and S-7 cartridges. With all the sandwich cartridges tested, there was very little difference between the frequency at which cycling began to occur and the flooding frequency.

A major disadvantage of sandwich cartridges is their poor reaction to column upsets. Flow rates and frequencies must be changed in small increments to prevent local flooding when approaching steady state operating conditions. Once a local phase inversion has been formed near the flooding frequency it is usually necessary to lower the frequency considerably to break it, rather than allowing it to work itself out.

Double Pulser Studies

A brief test was made to determine the effect of using a low frequency-high amplitude pulse for "pumping" action and a high frequency-low amplitude pulse for efficiency. The test was made in a 1C Column using cartridge N-4 with a 3.5-inch amplitude at 60 C. At flow rates of 850, 1250 and 1575 gph/ft^2, the flooding frequencies were 31.5, 21.5 and 19.5 cycles per minute, respectively. The column could not be operated at 1900 gph/ft^2. Superimposition of a 121 cycle, 5/16-inch amplitude pulse had little effect on column efficiency, capacity or flooding frequency. Further studies with a superimposed pulse of much higher frequency and lower amplitude are suggested.

2A COLUMN STUDIES

A limited number of 2A Column runs were made to confirm the suitability of a nozzle plate cartridge for replacement of the fluorothene Raschig rings in this organic continuous column. The cartridge studied contained 23 percent free area, 3/16-inch-hole nozzle plates on 2-inch-spacing in the 12-foot-high extraction section and 3-inch-spacing in the 6-foot-high scrub section.

The flooding data are presented in Table 4 and Figure 10. The data agree fairly well with those obtained with a similar cartridge described in HW-40550[3]. Dispersion characteristics in both the scrub and extraction sections indicated that satisfactory extraction and scrubbing should be obtained with this cartridge in a unidiameter column. The capacity greatly exceeds Purex Plant requirements.

(3) Nicholson, G. A. Purex Pulse Column Studies with Hydrocarbon Diluent, HW-40550, July 24, 1956 (SECRET)

TABLE 1

HA COLUMN SCRUB SECTION STUDIES

COLUMN: Dual-Purpose with 6-ft-high, 3-inch diameter extraction section; 9-ft-high, 3 or 4-inch-diameter scrub section (identified by run code number). Bottom interface.

FLOWSHEET: HAF = 1.4 M U, 1.7 M HNO_3; HAS = 2.5 M HNO_3; HAX = 30% TBP; Flow Ratio HAF/HAS/HAX = 96/70/450. 25°C.

Run No.	Cartridge	Vol. Vel., Gph/ft^2	Pulse Ampl. In.	Flooding Freq. Cyc./Min.	Remarks
4"-70-ASH	Plant zebra	410	0.6	95 ± 5	
4"-71-ASH	Plant zebra	650	0.6	75 ± 5	
4"-72-ASH	Plant zebra	1070	0.6	67 ± 7	
4"-73-ASH	SS-Tellerettes	580	0.6	95 ± 5	No true zebra dispersion.
4"-74-ASH	" "	1040	0.6	$\geq$70	Severe to moderate cycling.
4"-75-ASH	SS-Void	1060	0.6	50 ±10	No good dispersion observed.
4"-76-ASH	" "	530	0.6	75 ± 5	Operated stable at 80 cyc./min. with bentonite added to HAS.
4"-77-ASH	" "	530	0.6	$\geq$100	Mistron was added at 0.08 g/l in HAS. Poor scrubbing of MnO_2, produced *in situ* at feed point.
4"-78-ASH	" "	1060	0.6	65 ± 5	Stable operation obtained at 80 cyc./min. with Mistron added. Poor MnO_2 scrubbing.
4"-79-ASH	" "	1060	0.6	70 ±10	One foot of 1-in. fluorothene Raschig rings added above top sieve plate. Had minor effect on flooding.

TABLE 1 CONTINUED

Run No.	Cartridge	Vol. Vel., Gph/ft^2	Pulse Ampl. In.	Flooding Freq. Cyc./Min.	Remarks
4"-80-ASH	SS Void	530	0.6	90 ± 5	Same as above.
4"-81-ASH	SS-Fluor R/R	530	0.6	94 ± 4 }	Plastic rings improved stability,
4"-82-ASH	" " "	1060	0.6	>60 }	but zebra effect was poor.
4"-83-ASH to	4"-86-ASH: Measured chloride D.F.'s using the zebra cartridge. Results were inconclusive.				
4"-87-ASH	10% FA Noz.Pl.	530	0.6	>100	Good MnO_2 scrubbing.
4"-88-ASH	" " "	1060	0.6	>80	Only fair dispersion at 70 cyc./min.
3"-336-ASH	" " "	1870	1.1	55 ± 3	
3"-337-ASH	" " "	940	1.1	72 ± 4	
3"-338-ASH	" " "	940	0.6	116 ± 4	Better MnO_2 scrubbing with 0.6-inch amplitude.
3"-339-ASH	" " "	1870	0.6	95 ± 3	
3"-343-ASH	" " "	740	0.6	114 ± 11	Ca.5% aqueous holdup at 80 cyc./min.
3"-340-ASH	6% FA Noz Pl.	940	0.6	95 ± 5	Flood difficult to break.
3"-341-ASH	" " "	1870	0.6	72 ± 4	
3"-342-ASH	" " "	500	0.6	125 ± 3	

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* Cartridge Details: Plant zebra: 1-in. spaced repeating units of four 0.085-inch-hole, 21% free area stainless steel sieve plates, two 0.188-inch-hole, 23% free area linear polyethylene plates.

SS-Telleretter: 1-inch spaced 0.085-inch hole 21% free area stainless steel sieve plates with 6-inch-high open zones containing polyethylene Tellerettes every 3 feet.

SS-void: Same as above except open zones are 6 to 12-inch-high and are empty.

SS-Fluor. R/R: Same as above but with open zones filled with 1-inch-high Raschig rings.

10% FA Noz. Pl.: 2-inch-spaced 10% free area nozzle plates with 1/8-inch holes.

6% FA Noz. Pl.: 2-inch-spaced 6% free area nozzle plates with 1/8-inch holes.

TABLE 2

1BX COLUMN STUDIES

COLUMN: 15-foot-high, 3-inch-diameter cartridge of 4-inch-spaced plates with 28% free area louver plates at 4, 8, and 12 feet from bottom. Top interface. Pulse amplitude = 0.85 inch.

FLOWSHEET: 1BXF No. 1 = 0.26 M UNH, 0.28 M HNO_3
1BXF No. 2 = 0.17 M UNH, 0.07 M HNO_3
1BX = 0.3 to 0.4 M HNO_3
Flow ratio 1BX/1BXF = 0.045

Run No.	Temperature, C	Vol. Vel., Gph/ft^2.	Instability Threshold Freq., Cyc./Min.	1BXF No.	Remarks
Cartridge: 23% free area nozzle plates, 3/16-inch holes; nozzles point up					
3"-76-1BXH	45	630	70 ± 8	1	
3"-77-1BXH	45	970	57 ± 6	1	
3"-78a-1BXH	42	≤1320	41 to 51	1	Mildly unstable at all frequencies.
3"-78b-1BXH	53	1320	43	1	Probably at or near instability threshold.
3"-79-1BXH	50	970	77 ± 7	2	
3"-80-1BXH	50	1320	60 ± 3	2	
3"-81-1BXH	56	1520 ± 100	51	2	
Cartridge: 33% free area stainless steel sieve plates, 3/16-inch holes					
3"-82-1BXH	45	970	89 ± 5	2	
3"-83-1BXH	45	1320	66 ± 4	2	
3"-84-1BXH	50	1520 ± 100	51	2	
3"-85-1BXH	51	≤1320	46	1	
3"-86-1BXH	51	970	60 ± 3	1	

TABLE 3

1C COLUMN STUDIES

COLUMN: 9-foot-high cartridge, 3-inch-diameter. Top interface.

FLOWSHEET: 1CF = 0.29 M U, 0.11 M HNO_3 in 30% TBP; 1CX = 0.02 M HNO_3.

Run No.	Temp. C	Volume Velocity gph/ft^2	A/O	Pulse Ampl. In.	FREQUENCY, Cycles/Min* Lower Flooding	Instability Threshold	Upper Flooding	HTU Measured at	HTU, Ft.	Color Line Position, Ft.
Plant Plate Nozzle Cartridge										**
3"-181-1CH	60	1040	1.03	0.7	-	85	103	80	1.29	4.5-9+
3"-182-1CH	60	1250	1.24	0.7	-	76	89	-	-	5-9
3"-183-1CH	60	1610	1.06	0.7	-	<66 [redacted]	-	-	-	8
3"-184-1CH	60	1650	1.07	0.5	-	<75 [redacted]	-	-	-	6.5-8.5
3"-185a-1CH	60	915	1.25	0.5	25-30	-	-	-	-	-
3"-185b-1CH	60	1575	0.87	0.5	40-42	-	-	-	-	-
3"-185c-1CH	60	1330	1.05	0.5	32-34	-	-	-	-	6-8
3"-186-1CH	60	788	1.09	0.5	20	-	-	-	-	-
3"-187-1CH	60	1042	0.99	0.5	24	-	-	-	-	-
3"-188-1CH	60	1220	1.08	0.5	30	-	-	-	-	-
3"-189-1CH	60	1340	1.05	0.5	33	84	98	-	-	6-8
3"-190-1CH	60	1860	1.15	0.5	47	70	79	-	-	6.5-7
3"-191-1CH	60	824	1.03	0.5	-	104	114	-	-	6.5-8
3"-192-1CH	60	1850	0.98	0.5	51	ALL	70	-	-	-
3"-193-1CH	60	2090	1.01	0.5	60	ALL	79	-	-	8-9+
3"-194-1CH	60	855	1.08	1.0	12	47	79	-	-	5-7
3"-195-1CH	60	985	1.05	1.0	17	50	70	-	-	7
3"-196-1CH	60	2010	1.15	1.0	38	ALL	52	-	-	7

TABLE 3 CONTINUED

Run No.	Temp. C	Volume Velocity gph/ft^2	A/O	Pulse Ampl. In.	FREQUENCY, Cycles/Min* Lower Flooding	Instability Threshold	Upper Flooding	HTU Measured at	HTU, Ft.	Color Line Position, Ft.
N-1 Cartridge										
3"-166-1CH	60	1675	1.08	1.0	-	ALL	-	52	1.18	6-8
3"-167-1CH	60	1060	1.16	1.0	-	73	76	62	1.13	-
3"-168-1CH	60	1315	1.09	1.0	-	66	73	62	1.07	4.5-8
3"-169-1CH	60	1635	1.09	1.0	-	48	52	48	1.25	6.5-8
3"-170-1CH	60	1021	1.02	1.0	-	80	89	-	-	4.5-5
3"-171-1CH	60	1250	1.10	1.0	-	73	-	-	-	-
3"-180-1CH	60	1620	1.03	0.7	-	ALL	71	62	1.33	7.5-8
N-2 Cartridge										
3"-172-1CH	60	1690	1.08	1.0	-	-	71	52	1.48	6-8
3"-173-1CH	60	988	1.02	1.0	-	90	103	76	1.03	5.5-7.5
3"-174-1CH	60	1010	1.07	0.7	-	94	99	80	1.16	7-8
3"-175-1CH	60	1575	1.07	0.7	-	62	-	57	1.17	7-8
3"-176-1CH	60	1585	1.11	0.7	-	67	76	62	1.23	7-8
3"-177-1CH	60	1630	1.10	0.7	-	<66	-	-	-	8-5
3"-177a-1CH	60	1770	1.1	0.7	-	<66	-	-	-	-
N-3 Cartridge										
3"-178-1CH	60	1240	1.20	0.7	-	82	85	71	1.28	5.5-6.5
3"-179-1CH	60	1600	1.08	0.7	-	64	71	62	1.23	7-8
N-4 Cartridge										
3"-197-1CH	60	1710	1.07	1.0	22	74	79	-	-	6-7
3"-198-1CH	60	815	1.05	1.0	12	83	88	-	-	4-5
3"-199-1CH	60	1990	0.97	1.0	38	ALL	55	-	-	9+
3"-200-1CH	60	1280	1.10	1.0	-	74	89	-	-	5-7.5
3"-200a-1CH	60	1580	1.05	1.0	-	74	82	-	-	6.5

TABLE 3 CONTINUED

Run No.	Temp. C	Volume Velocity gph/ft^2	A/O	Pulse Ampl. In.	FREQUENCY, Cycles/Min* Lower Flooding	Instability Threshold	Upper Flooding	HTU Measured at	HTU, Ft.	Color Line Position, Ft.
S-5 Cartridge									***	
3"-154-1CH	50	940	0.95	0.7	-	-	100	82	0.80	7.5-8.5
3"-155-1CH	50	1120	0.95	0.7	-	-	82	75	0.61	8-8.5
3"-156-1CH	50	805	0.94	0.7	-	108	110	94	1.19	8-9+
3"-157-1CH	60	960	1.09	1.0	-	-	-	57	0.75	7
S-6 Cartridge										
3"-158-1CH	60	1620	1.03	1.0	-	>48	-	48	1.18	8
3"-158a-1CH	60	2120	1.00	1.0	-	-	-	48	1.15	-
3"-159-1CH	50	1650	1.00	1.0	-	-	76	70	1.11	7-9+
3"-160-1CH	50	1050	1.0	1.0	-	-	94	-	-	6-7
3"-161-1CH	50	1305	1.0	1.0	-	-	80	-	-	7
3"-162-1CH	50	1620	1.092	1.0	-	-	-	52	1.61	7.5-9+
3"-163-1CH	60	1750	1.092	1.0	-	-	-	51	1.64	8-9+
3"-164-1CH	60	952	1.21	1.0	-	-	80	76	1.26	5.5-8
3"-165-1CH	60	1225	1.12	1.0	-	-	84	62	1.20	6.5-8
3"-201-1CH	60	2000	1.11	1.0	45	ALL	65	-	-	6.5-8
3"-202-1CH	60	1540	1.08	1.0	34	70>	74	-	-	5
S-7 Cartridge										
3"-203-1CH	60	874	1.28	1.0	-	93	97	-	-	2.5
3"-204-1CH	60	1069	1.01	1.0	-	-	81	-	-	3-4
3"-205-1CH	60	1339	1.00	1.0	-	81>	84	-	-	4-5.5
3"-206-1CH	60	1634	1.00	1.0	29	79	81	-	-	6-6.5
3"-207-1CH	60	2193	1.02	1.0	40	62	70	-	-	6.5-8

* All frequencies are ± 5%.

** Color line position, measured from the bottom of the column, gives only a qualitative picture of column efficiency. It is also a strong function of the aqueous to organic flow ratio.

*** HTU values calculated at 50 C are doubtful since the column was close to a pinch.

TABLE 4

2A COLUMN STUDIES

CARTRIDGE: 18-foot-high, 3-inch-diameter cartridge with 23% free area nozzle plates with 3/16-inch holes. 2-inch plate spacing in bottom 12 feet, 3-inch in top 6 ft. 2AF introduced either 6 or 12 feet from bottom. Bottom interface. Pulse amplitude = 1.05 inch.

FLOWSHEET: 2AF = 2.87 M HNO_3, 2AS = 0.72 M HNO_3, 2AX = 30% TBP. Flow ratio 2AF/2AS/2AX = 2.5/0.4/1

Run No.	Temp. °C	Vol. Vel., Gph/ft². Extraction	Scrub	Extraction Flooding Freq., Cyc/Min.	Extraction Section Height, Ft.	Remarks
3"-184-2AH	25	740	240	112 ± 4	6	Scrub section near or at flooding at f = 130.
3"-185-2AH	25	1230	500	100 ± 4	6	
3'-186a-2AH	25	1540	560	90 ± 4	6	
3"-186b-2AH	25	1540	560	79 ± 3	12	
3"-187-2AH	60 (2AF)	1540	560	95 ± 5	12	
3"-188-2AH	60 (2AF)	1850	590	85 ± 6	12 }	Difficult to determine which section flooded first.
3"-189-2AH	60 (2AF)	1540	560	95 ± 4	6 }	

FIGURE 1

HA COLUMN SCRUB SECTION FLOODING CHARACTERISTICS

Two-in.-spaced stainless steel
nozzle plates (1/8-in. holes)

Bottom interface

Temperature = 25C

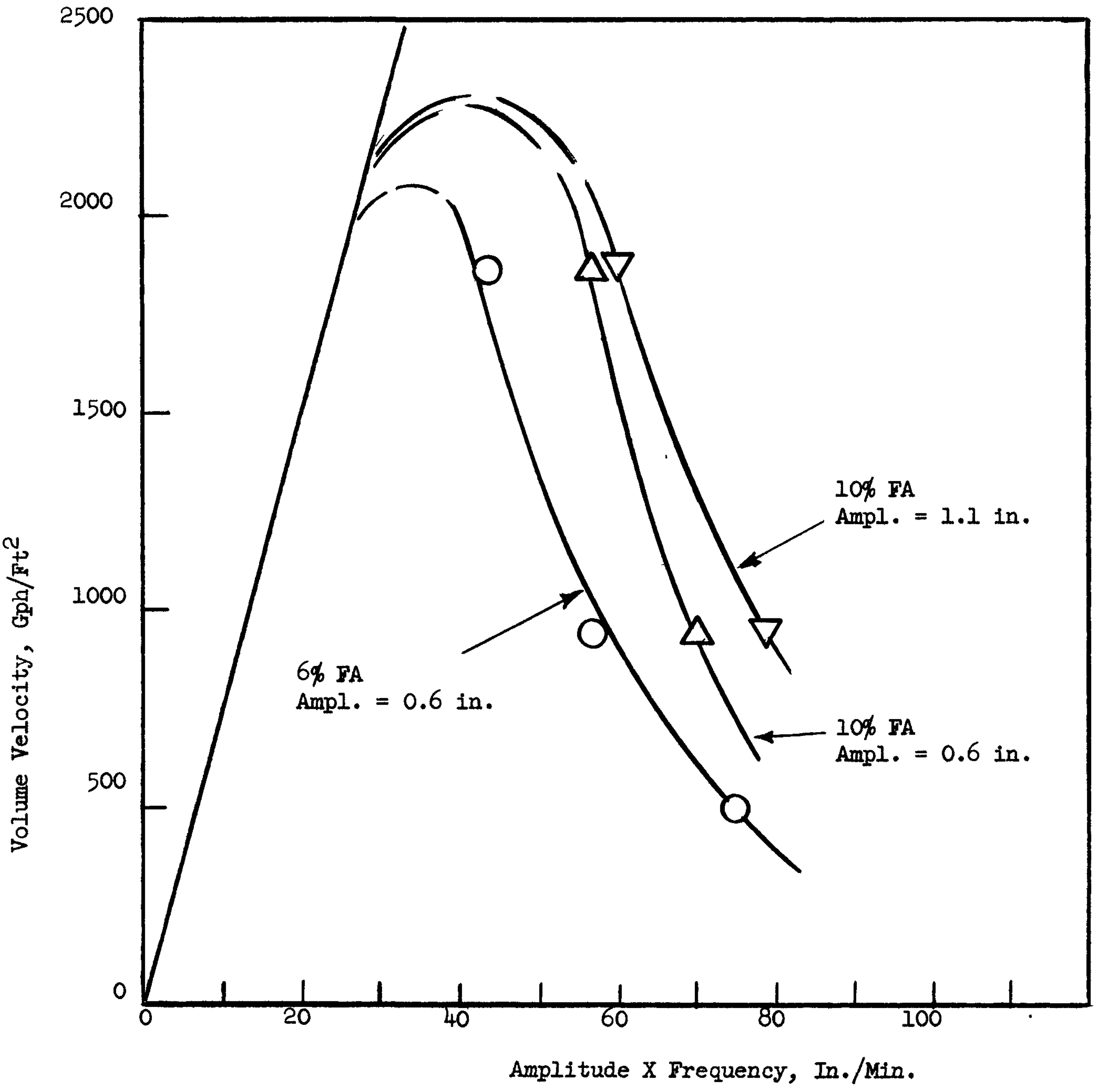


FIGURE 2

1BX COLUMN INSTABILITY THRESHOLDS

Cartridge A = 4-in.-spaced, 23% FA nozzle plates (3/16-in. holes)
Cartridge B = 4-in.-spaced, 33% FA ss sieve plates (3/16-in. holes)

Both cartridges have 28% FA louver plates at 4 ft. intervals

Feed 1 = 0.26M UNH, 0.28M HNO_3
Feed 2 = 0.17M UNH, 0.07M HNO_3

Pulse amplitude = 0.85 in., Top interface

Temperature = 45 to 56 C

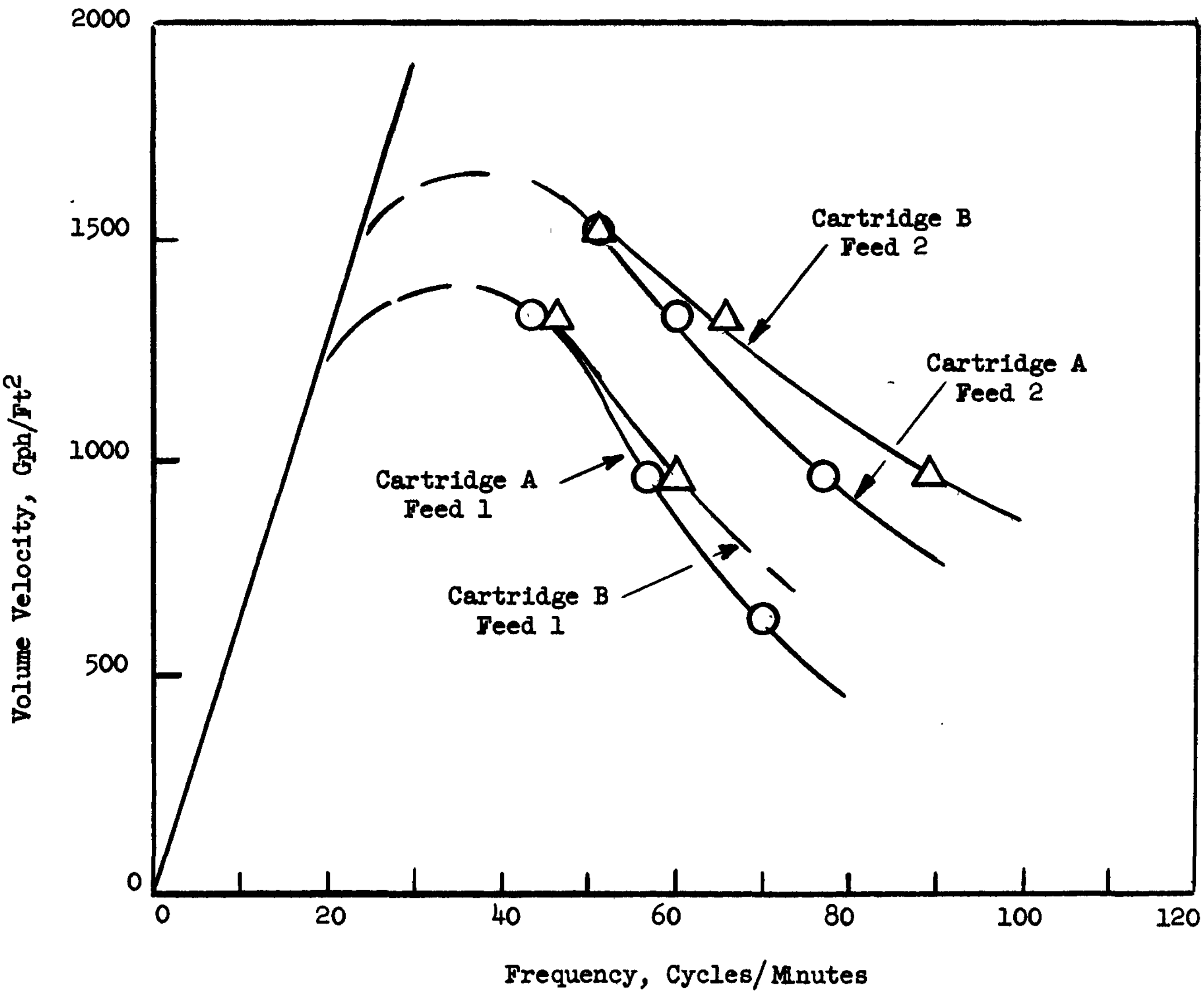
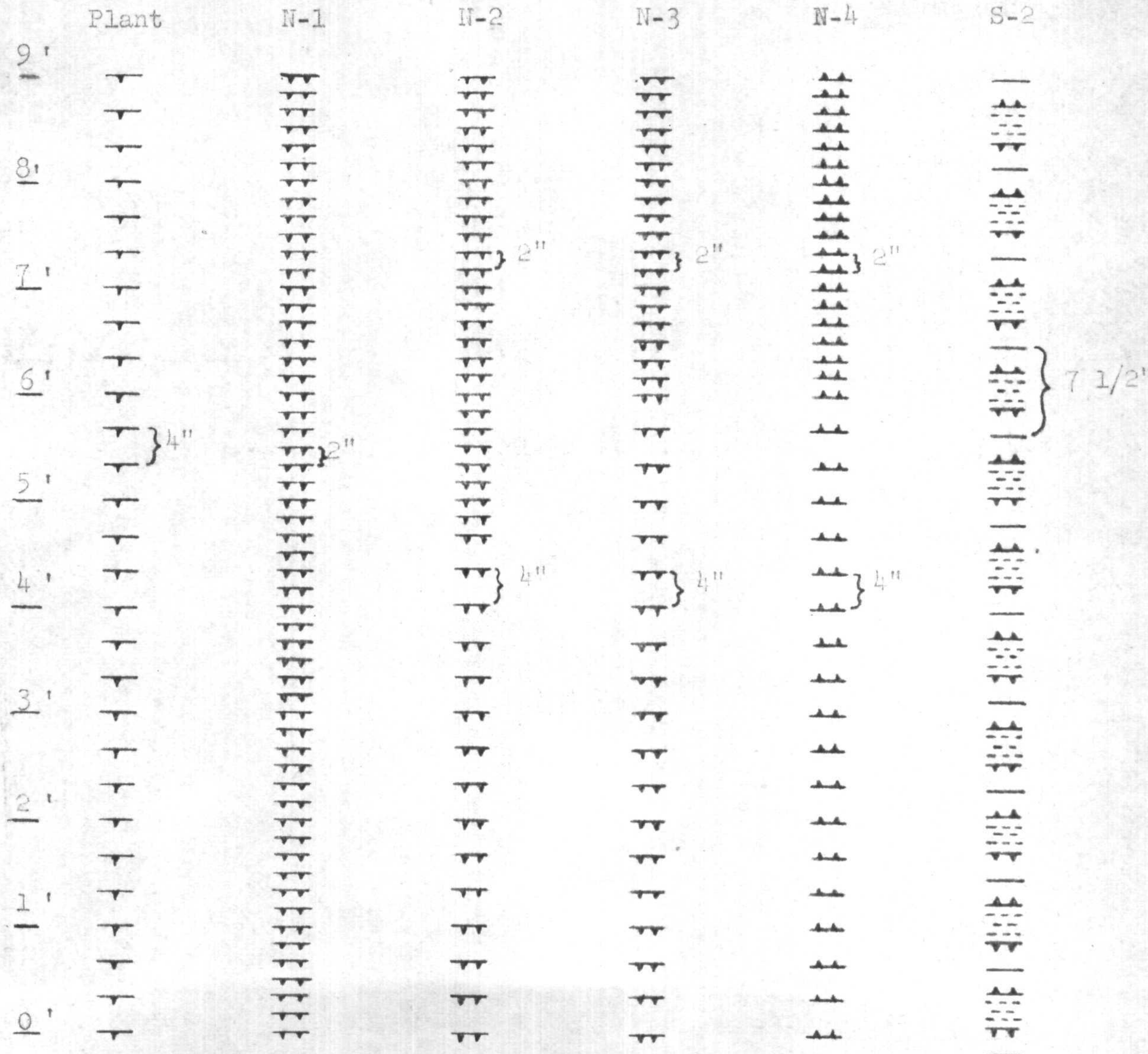


FIGURE 3

1C TEST CARTRIDGE CONFIGURATIONS

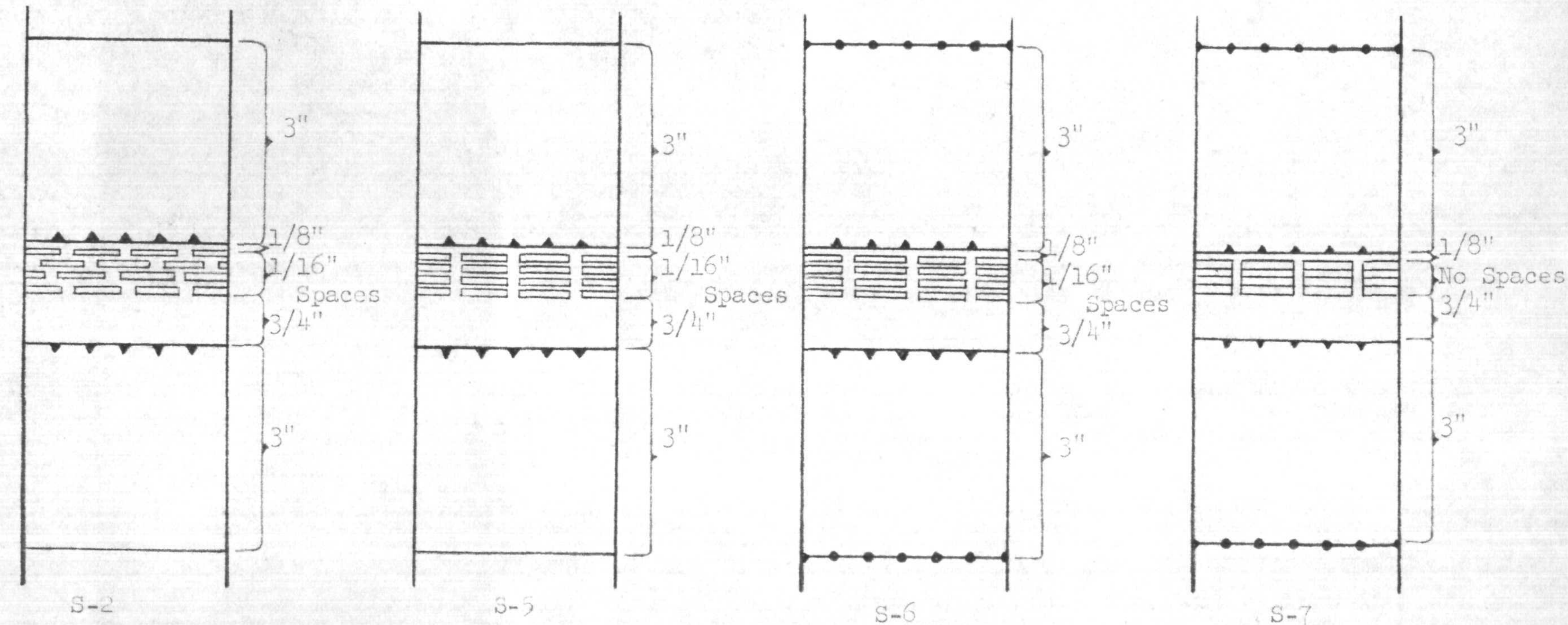


LEGEND

- Stainless steel nozzle plate, nozzles down, 49 mil nozzle depth, 10 percent free area, 1/8-inch hole diameter
- Stainless steel nozzle plate, nozzles down, 42 mil nozzle depth, 23 percent free area, 3/16-inch hole diameter
- Stainless steel nozzle plate, nozzles up, 42 mil nozzle depth, 23 percent free area, 3/16-inch hole diameter
- Stainless steel sieve plate, 23 percent free area, 1/8-inch hole diameter
- --- Linear polyethylene sieve plate, 18 percent free area, 0.18-inch hole diameter

FIGURE 4

TEST SANDWICH CARTRIDGES



LEGEND

- Stainless steel nozzle plate, 42 mil nozzle depth, 23 percent free area, 3/16-inch hole diameter
- Stainless steel sieve plate, 23 percent free area, 1/8-inch hole diameter
- Stainless steel sieve plate, 33 percent free area, 3/16-inch hole diameter
- Linear polyethylene sieve plate, 18 percent free area, 0.18-inch hole diameter, 1/8-inch thick.

FIGURE 5

1C COLUMN INSTABILITY AND FLOODING CHARACTERISTICS

Plant Nozzle Plate Cartridge
Top Interface, Nozzles down
0.5" Pulse Amplitude
Temperature 60°C

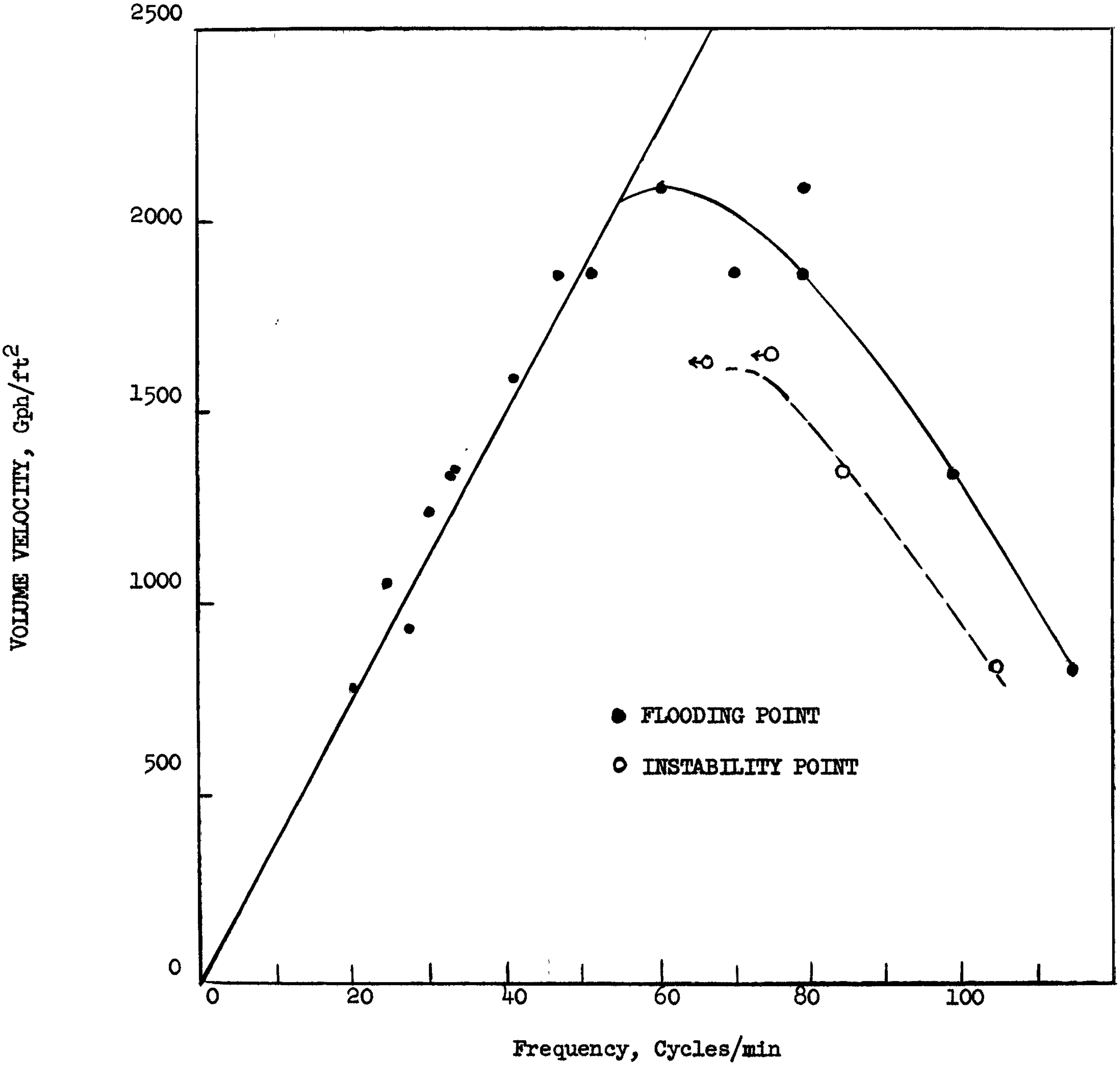


FIGURE 6

1C COLUMN INSTABILITY AND FLOODING CHARACTERISTICS

N-1 Nozzle Plate Cartridge
Top Interface, nozzles down
1.0" Pulse Amplitude
Temperature 60°C

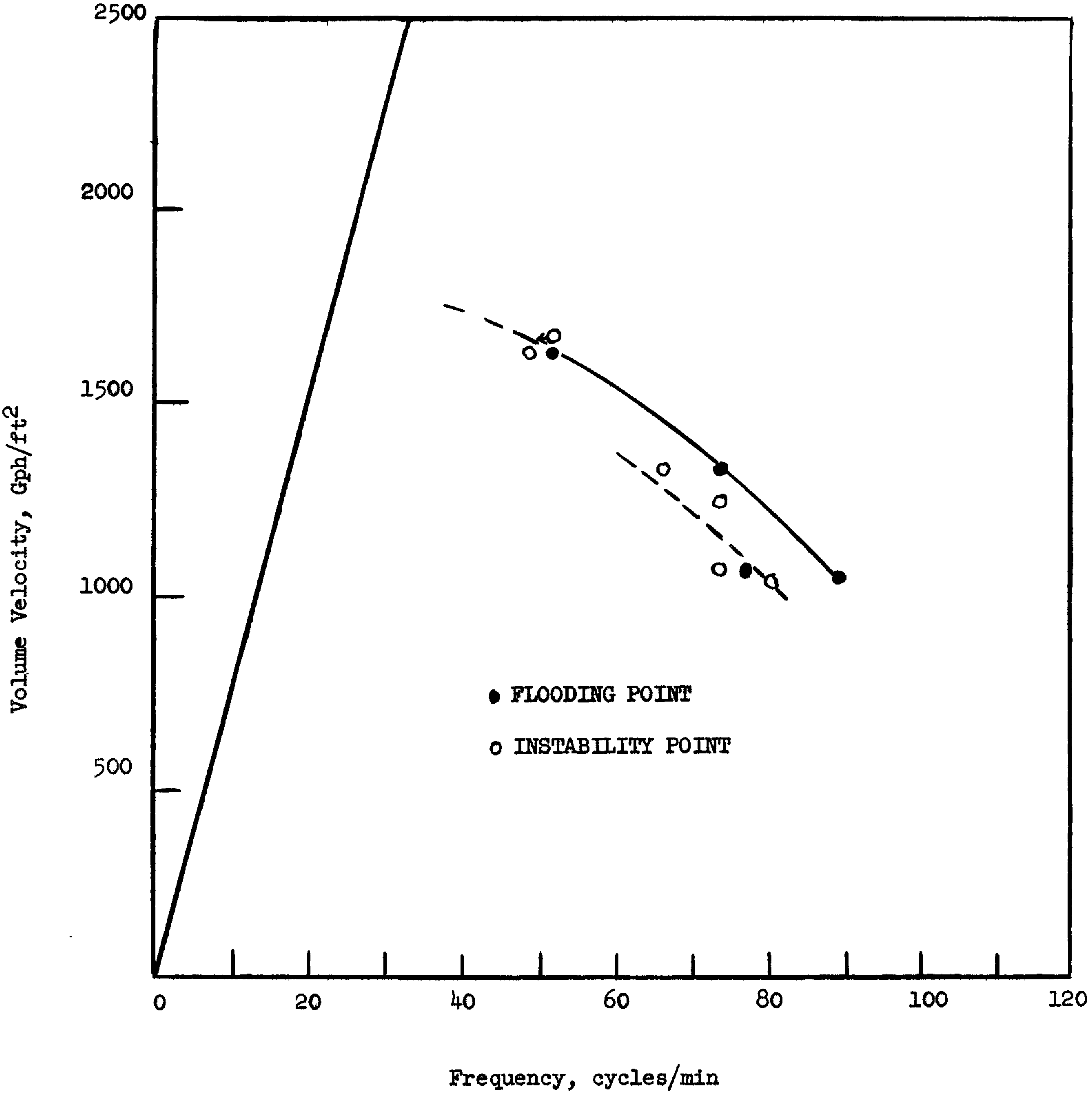


FIGURE 7

<u>1C COLUMN INSTABILITY AND FLOODING CHARACTERISTICS</u>

N-4 Nozzle Plate Cartridge
Top Interface, nozzles up
1.0" Pulse Amplitude
Temperature 60°C

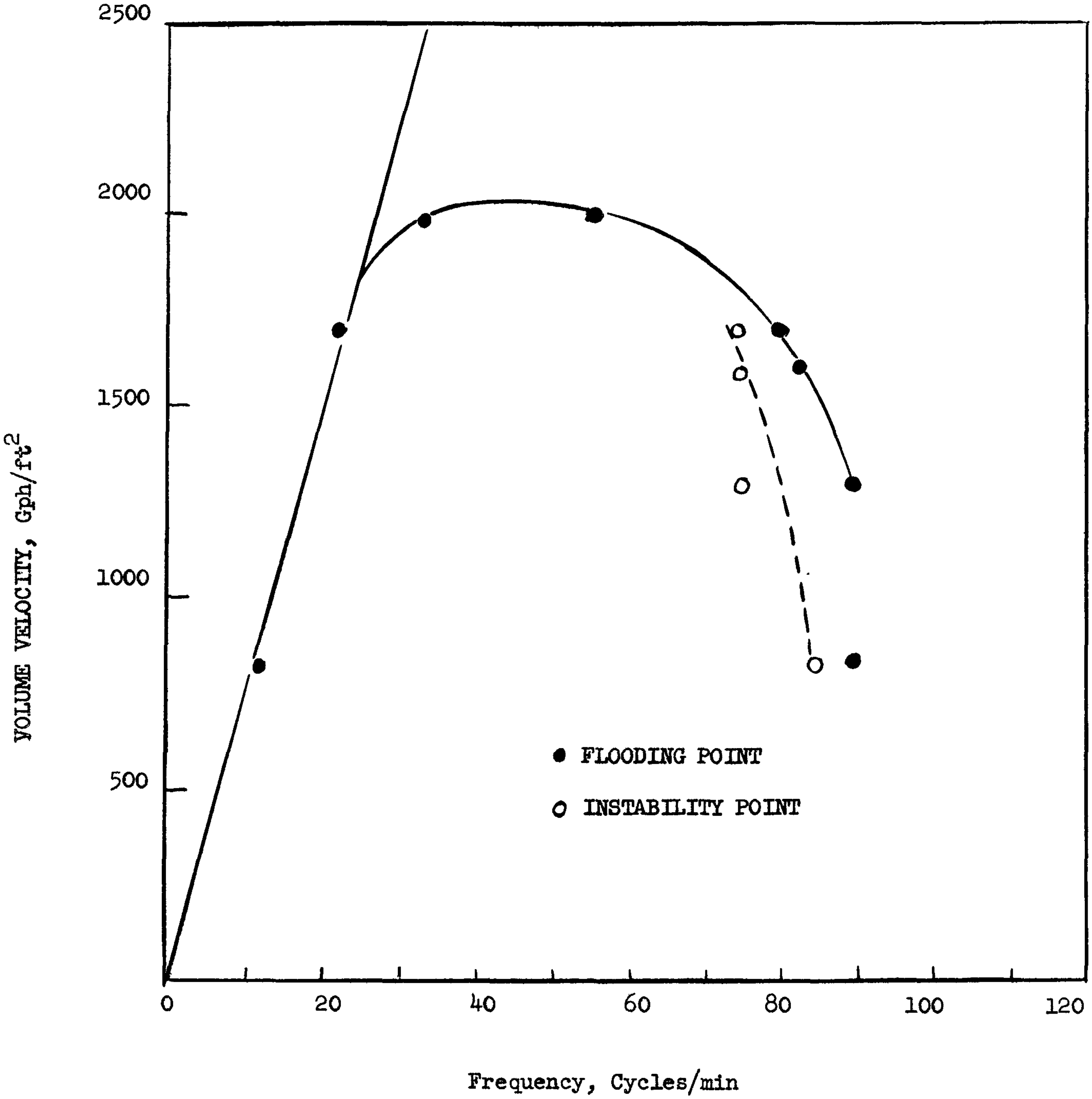


FIGURE 8

1C COLUMN INSTABILITY AND FLOODING CHARACTERISTICS

S-6 Sandwich Cartridge
Top Interface
1.0" Pulse Amplitude
Temperature 60°C

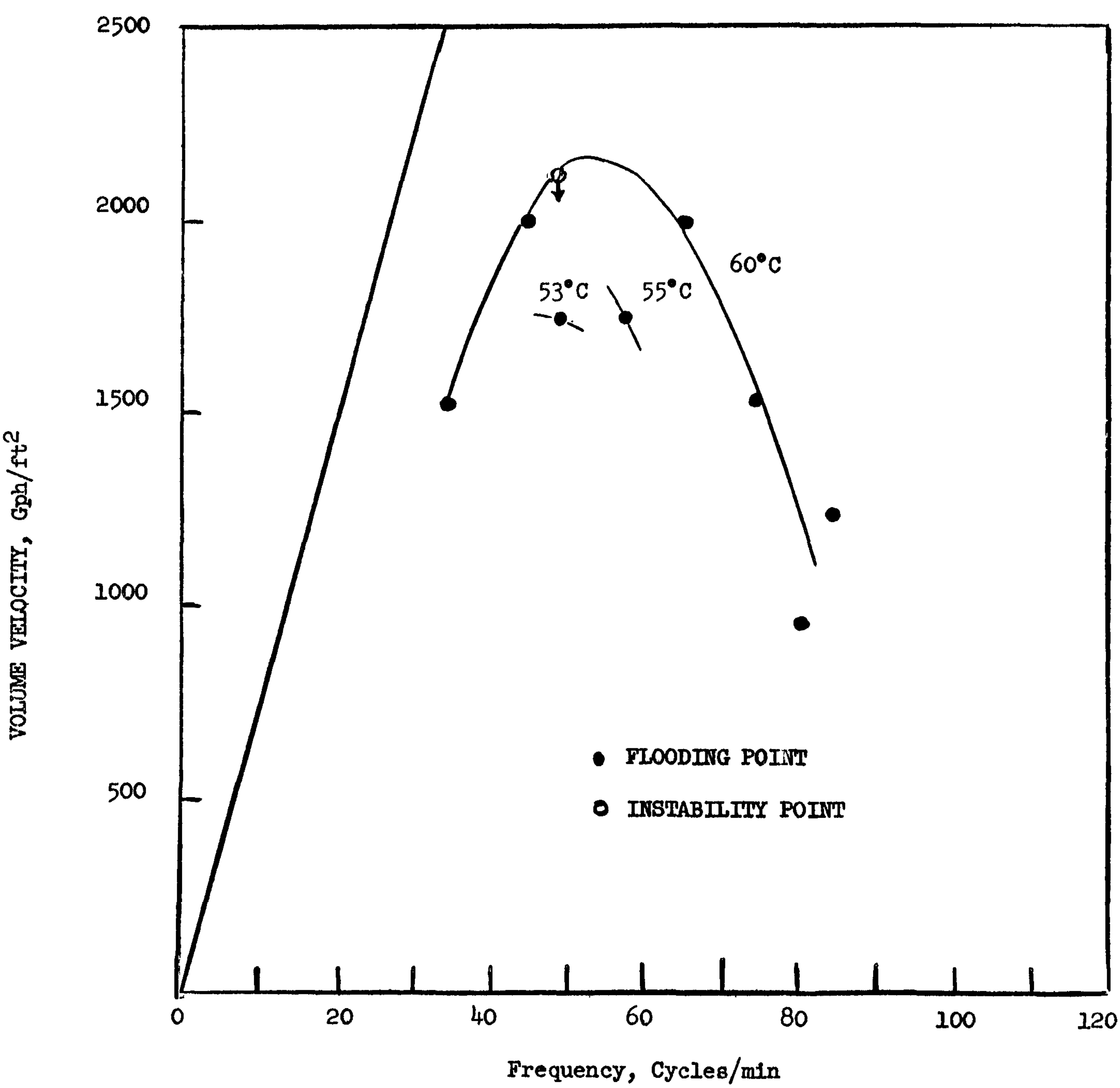


FIGURE 9

1C COLUMN INSTABILITY AND FLOODING CHARACTERISTICS

S-7 Sandwich Cartridge
Top Interface
1.0" Pulse Amplitude
Temperature 60°C

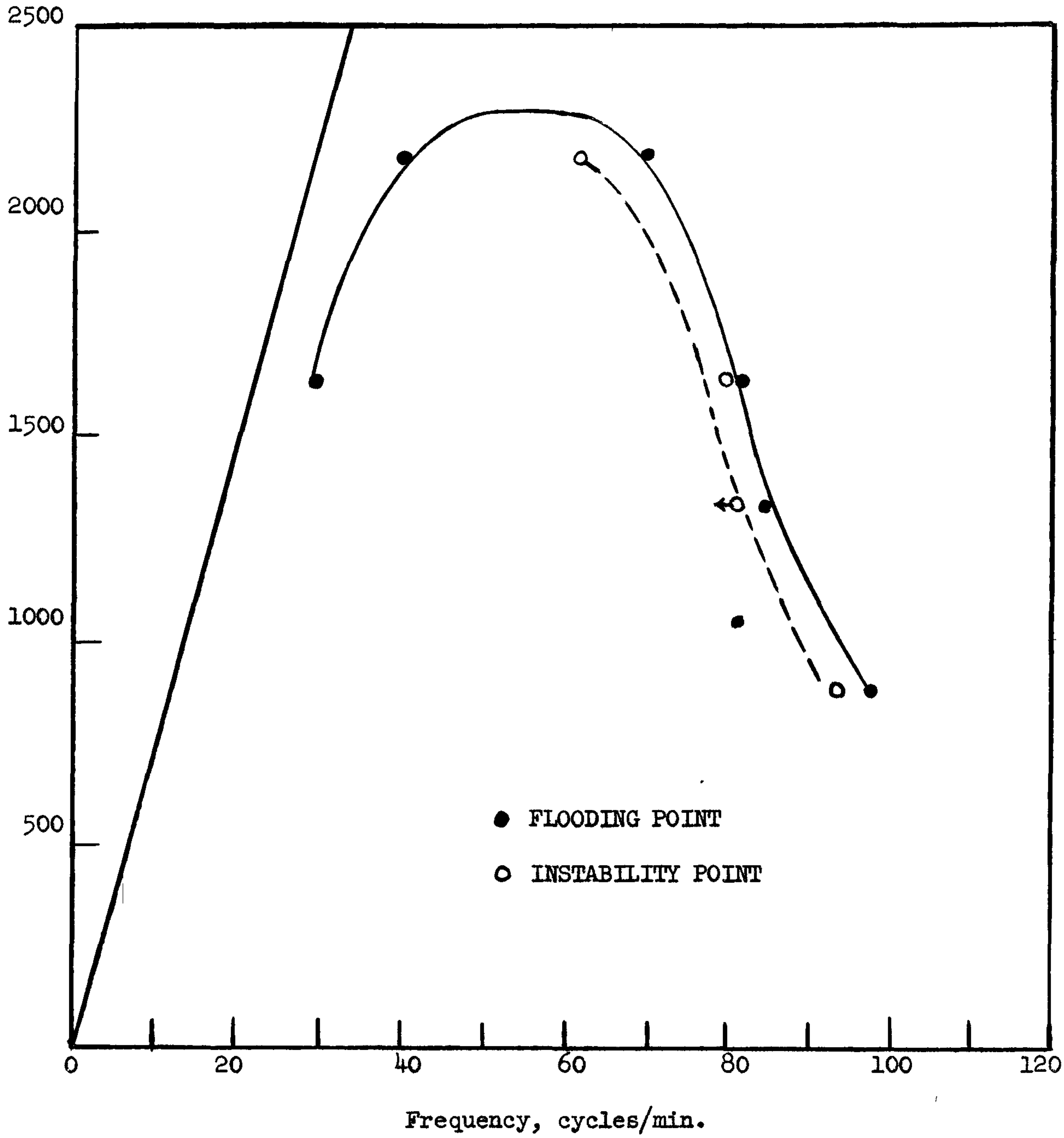


FIGURE 10

2A COLUMN FLOODING CHARACTERISTICS

23% FA nozzle plates (3/16-in. holes)
2-in. spacing in extraction section,
3-inch spacing in scrub section.

Pulse amplitude = 1.05 in.; bottom interface

Temperature 25 to 50 C