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EVALUATION OF SIEVE PLATES FOR STEAM STRIPPING OF TBP FROM EVAPORATOR FEED STREAMS

C. V. Chester

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Report Number: ORNL-2339

Contract No. W-7405, eng-26

CHEMICAL TECHNOLOGY DIVISION

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DATE ISSUED:

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1.0 ABSTRACT

Tests of a 6-in.-dia sieve plate column with downcomers on the uranyl nitrate-water-tributyl phosphate system showed average plate efficiencies of 25% for a plate with 2.73% free area over a range of f factors from 0.2 to 0.7 at an L/V of 1.15. The efficiency decreased above f = 0.7. (The f factor is the product of superficial vapor velocity and the square root of vapor density.)

Uranium entrainment in the six-plate column (top two plates dry) was of the order of 50 to 100 μg/liter of condensate at f factors from 0.2 to 0.5. At f = 0.7, entrainment rose to about 300 μg/liter. This entrainment was reduced to less than 100 μg/liter by admitting water as reflux to the sixth plate at a rate equal to 10% of the feed rate. The column flooded at an f factor of about 1.0, and began to weep with f between 0.1 and 0.2.

2.0 INTRODUCTION

Tributyl phosphate is removed from uranyl nitrate solutions prior to concentration by evaporation to prevent the buildup of hydrolysis products in the evaporator. TBP may be steam-stripped from evaporator feed streams by introducing the feed stream through a vapor-liquid contactor, countercurrently to the vapor. In the past, bubble-cap columns were used for this purpose. Sieve-plate columns would be desirable for this service, as sieve plates are cheaper, easier to decontaminate, and may be fabricated from heavy-gage metal to withstand corrosion better.

Since the system is far from ideal, and TBP concentrations are very dilute, experimental data on the performance of sieve plates are needed for an economical design. This report gives the results of a series of tests on a 6-in.-dia sieve plate column.

3.0 APPARATUS

The column (Figs. 1 and 2) was assembled from 12-in.-long sections of 6-in.-dia Pyrex pipe. The six sieve plates are gasketed between the sections. The bottom section of the column is made of stainless steel and carries lines for admission of stripping steam and removal of the bottoms product. The top section of the column contains a 4-in.-long York mesh demister, supported on a plate gasketed between the top section and the one below. The top of the

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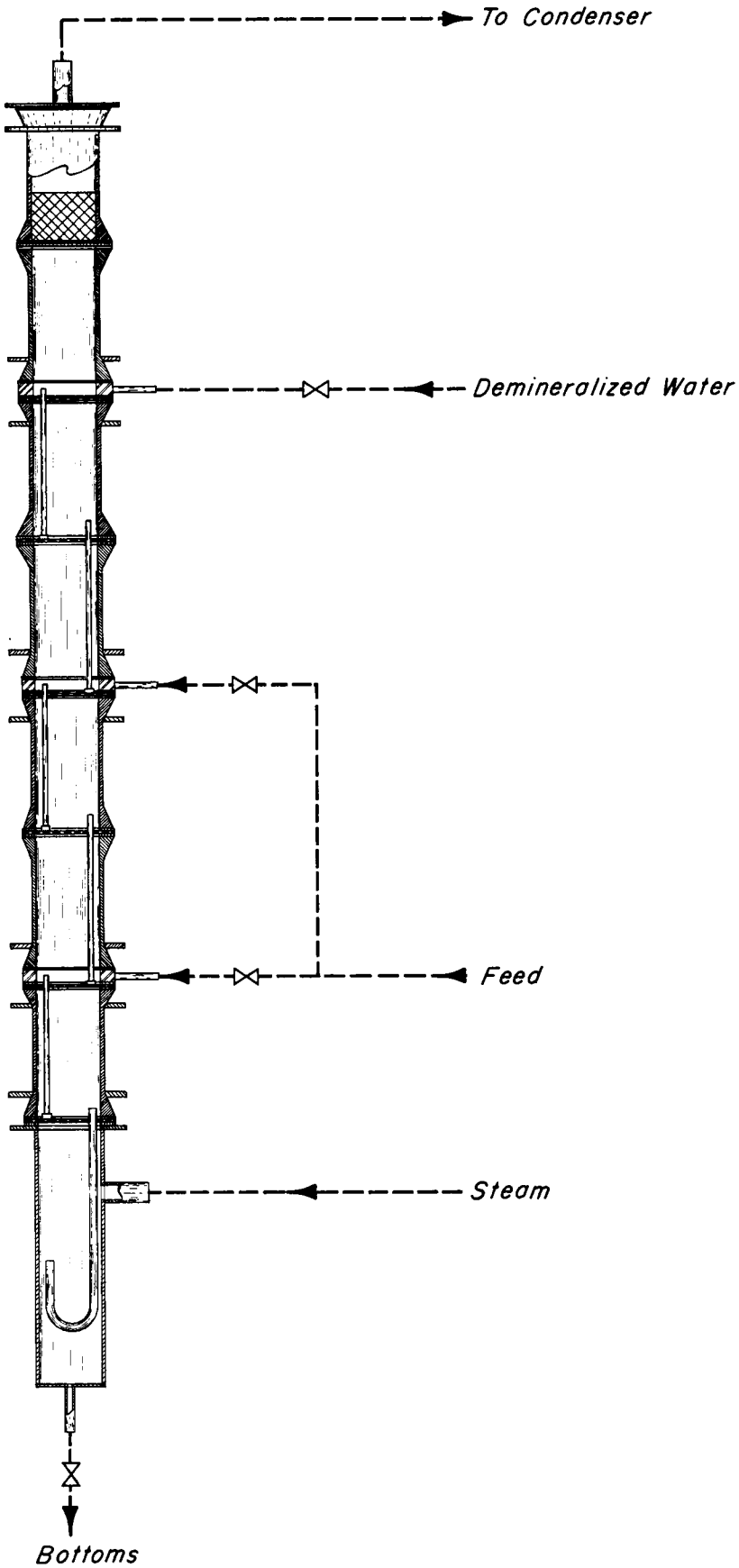

STEAM STRIPPING OF TBP
FIG. 1
GLASS SIEVE PLATE TOWER

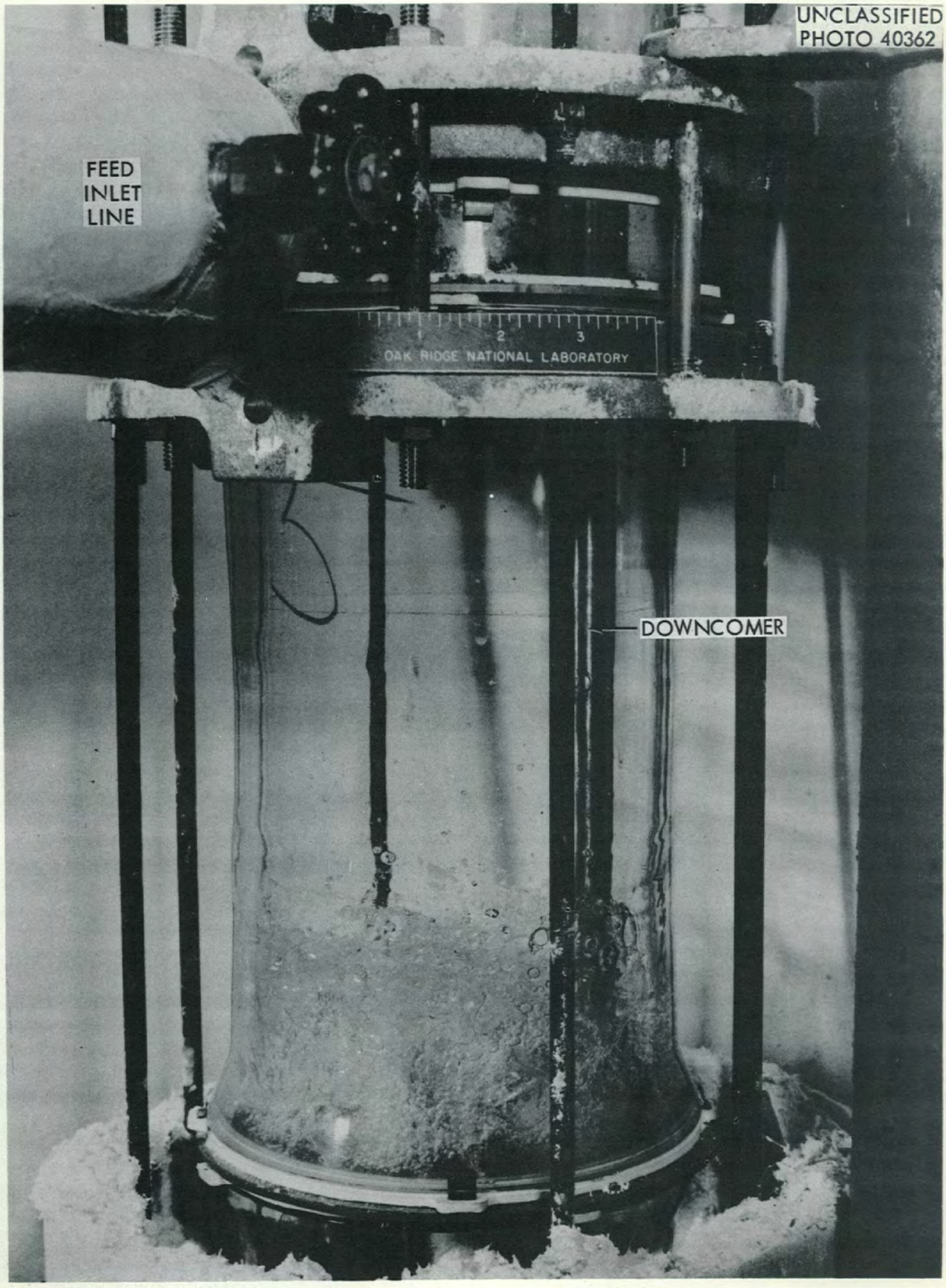

Fig. 2. Closeup of Operating Sieve Plate, Showing Assembly Details.

column is closed by a blind flange carrying a 2-in.-dia vapor outlet line and a thermocouple well. Feed may be admitted to either the second or fourth plate by lines through spacers gasketed between the plate and the column section above. Reflux water may be admitted to the sixth plate via an identical construction.

The sieve plates are made of 1/8-in.-thick stainless steel. Each plate has twenty-eight 3/16-in.-dia holes on 0.8-in. square pitch (Fig. 3). The total area of the holes is 2.73% of the inside column area. In addition, each plate carries a downcomer 2-1/8 in. from the center, which projects up through the plate for 1 in. to serve as its own overflow weir. Opposite the downcomer is a cup which provides a liquid seal for the downcomer from the plate above.

A steam-jacketed kettle serves as the evaporator. The vapor line from the kettle enters the bottom of the sieve plate column. The bottoms line from the column introduces feed near the top of the kettle. The product from the kettle flows by gravity through a control valve to a product storage tank.

Feed is mixed and fed from either of two 50-gal tanks equipped with a circulating pump and is pumped from the tanks through a rotameter and a feed preheater to either the second or fourth plate of the column. Vapor overhead from the column is condensed, and the condensate runs through a rotameter to tanks.

The sieve plate column is an addition to the Badger column equipment installed on the third floor of building 4505 and described in ORNL-CF-57-3-80. The construction details are shown in ORNL drawings D-21430, D-21431 and D-21432. Figure 4 is an overall view of the equipment.

4,0 PROCEDURE

The equipment was not operated as an evaporator since operation as an evaporator would require considerable time for steady state to be reached. Instead, the kettle was kept empty, and process steam was admitted to the bottom of the column via a by-pass line and the kettle. Feed was metered through the feed rotameter. Condensate rate and bottoms rate were measured by the "bucket-and-stopwatch" method (timing the collection of a measured volume).

Feed was made up in 50-gal batches to a uranium concentration of about 47 g/liter, and then saturated with 30% TBP by recirculating the feed with a layer of 30% TBP for 30 min. An additional amount of pure TBP, about 40 g, calculated to saturate the batch of feed, was added during the recirculation.

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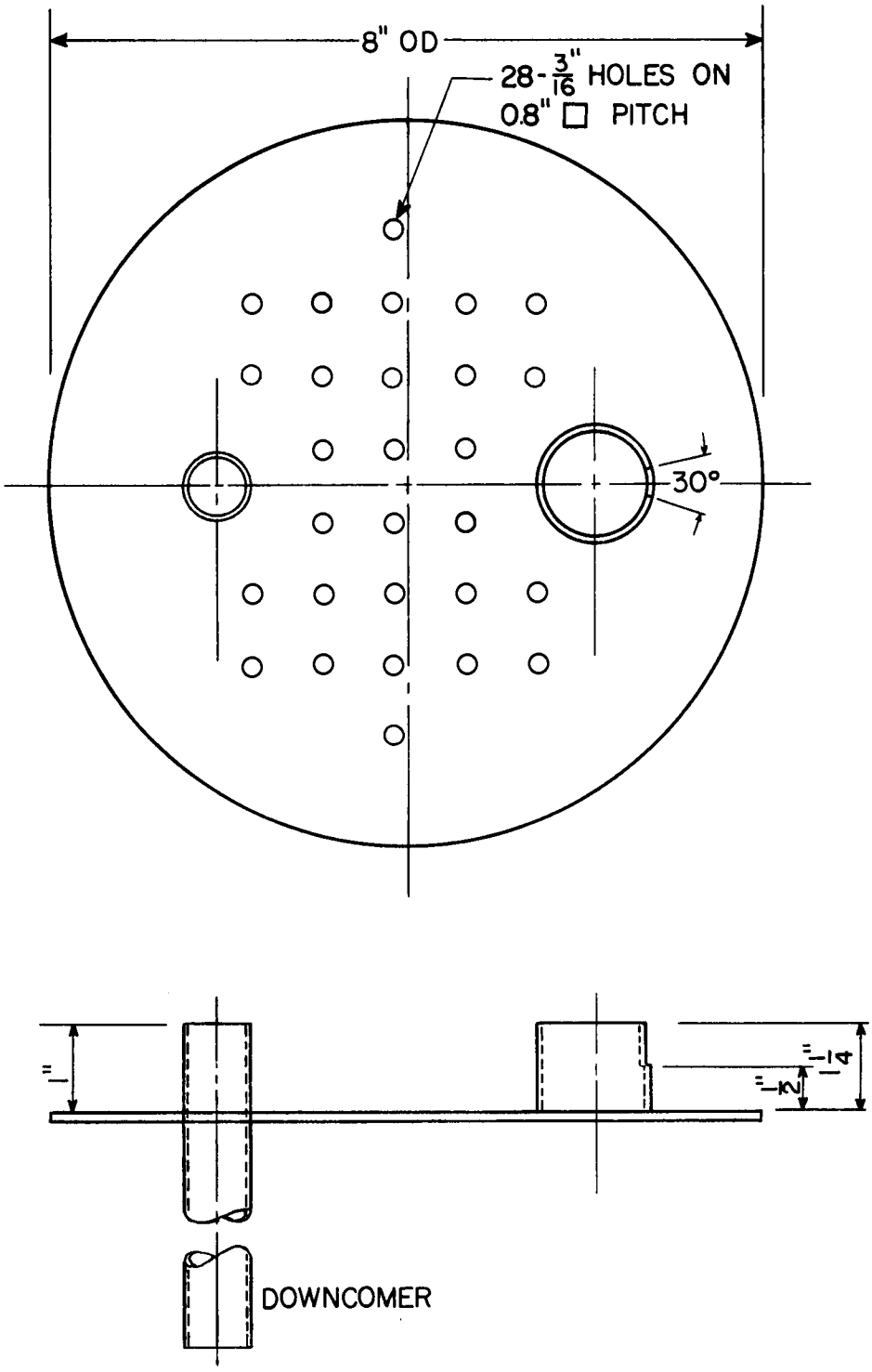

STEAM STRIPPING OF TBP

FIG. 3

SIEVE PLATE

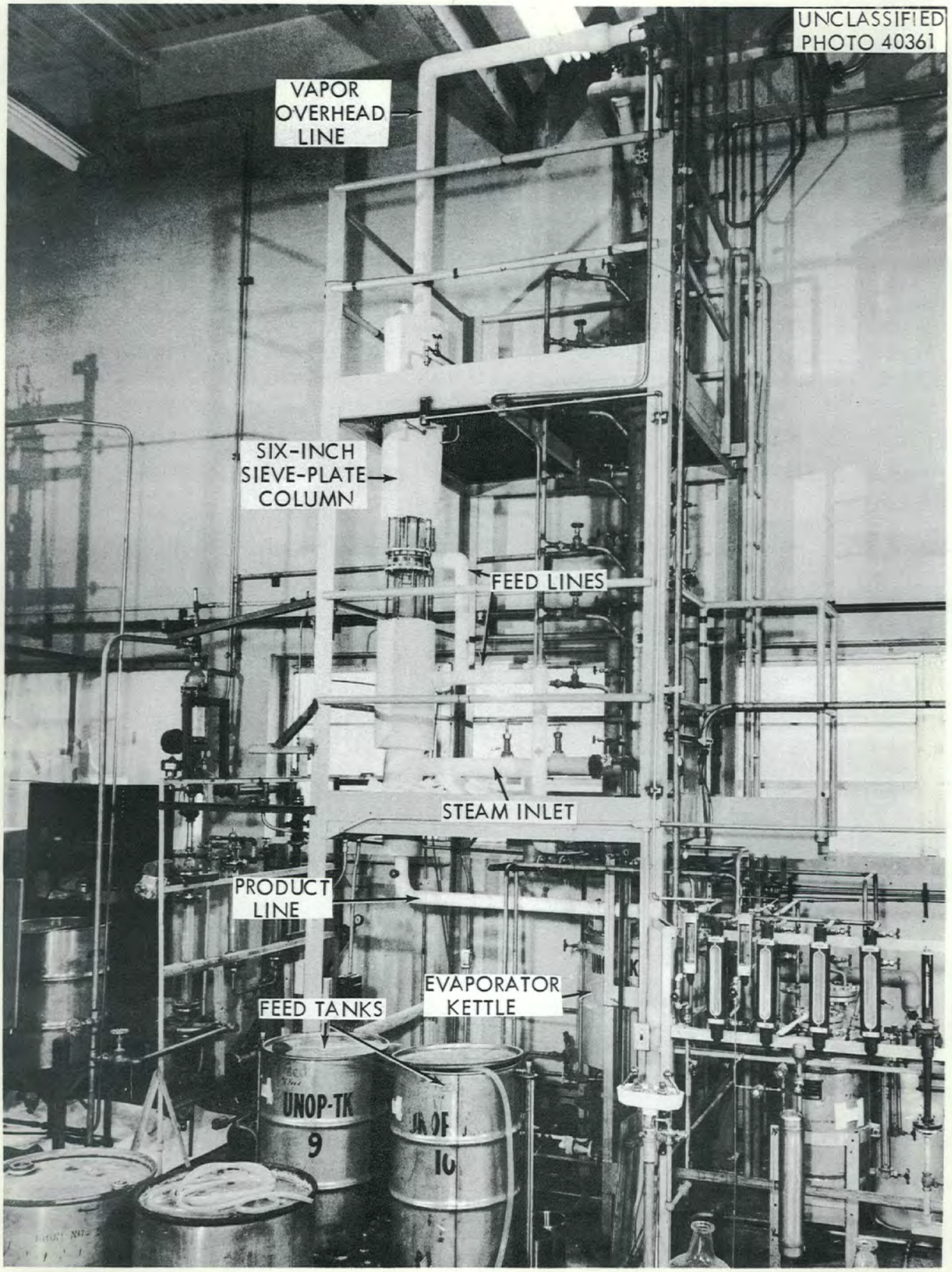

Fig. 4. Over-all View of Equipment for Steam Stripping TBP from Uranyl Nitrate Solutions.

To make a run, steam was admitted to the dry column, and the rate was adjusted so that the measured condensate rate corresponded to the desired f factor. The feed pump was then started and the feed throttled to the desired setting of the precalibrated feed rotameter. If used, steam for preheating the feed, reflux water, and/or entrained organic were started and set at the desired rate at this time.

When the column had run for 30 min, condensate and bottoms rates were measured by bucket and stopwatch, the feed tank level was recorded, and the feed, bottoms, and condensate were sampled. This process was repeated at three additional 30-min intervals, and the run was then terminated.

The samples were analyzed for TBP spectrophotometrically, and for uranium colorimetrically. From the known feed and condensate rates and feed and bottoms compositions, an operating line was established and the number of theoretical plates calculated. Figure 5 is an example of such a calculation on a logarithmic plot. It should be pointed out that fractions of a theoretical plate were calculated from arithmetic, rather than logarithmic, concentration differences.

5.0 RESULTS

5.1 Plate Efficiency

The results of all the runs are given in Table 1. Figure 6 is a plot of average Murphree plate efficiency as a function of the f factor. Most of the data are well represented by a flat curve at about 25% plate efficiency, falling off at higher f factors in the usual way. The five low points in the middle of the curve are attributed to random error. A column of this type operated in the range of f factors from 0.25 to 0.7 may be assumed to have average plate efficiencies of 25% with some confidence.

Data from runs with feed on the second plate were omitted from the plot of efficiency vs f factor since the calculated efficiencies were low and erratic and believed not representative of column performance. It is thought that the introduction of feed on a plate, and vapor condensation in the case of cold feed, reduces plate efficiency. This was suggested by comparison of the results of preheated and cold (ambient) feed on the second plate efficiencies. This conclusion was not borne out by the results of heated and unheated feed on the fourth plate, but the results for heated feed are so scattered that experimental error may cover up the effect.

5.2 Analysis

As mentioned above, the analyses for TBP were done by spectrophotometer.

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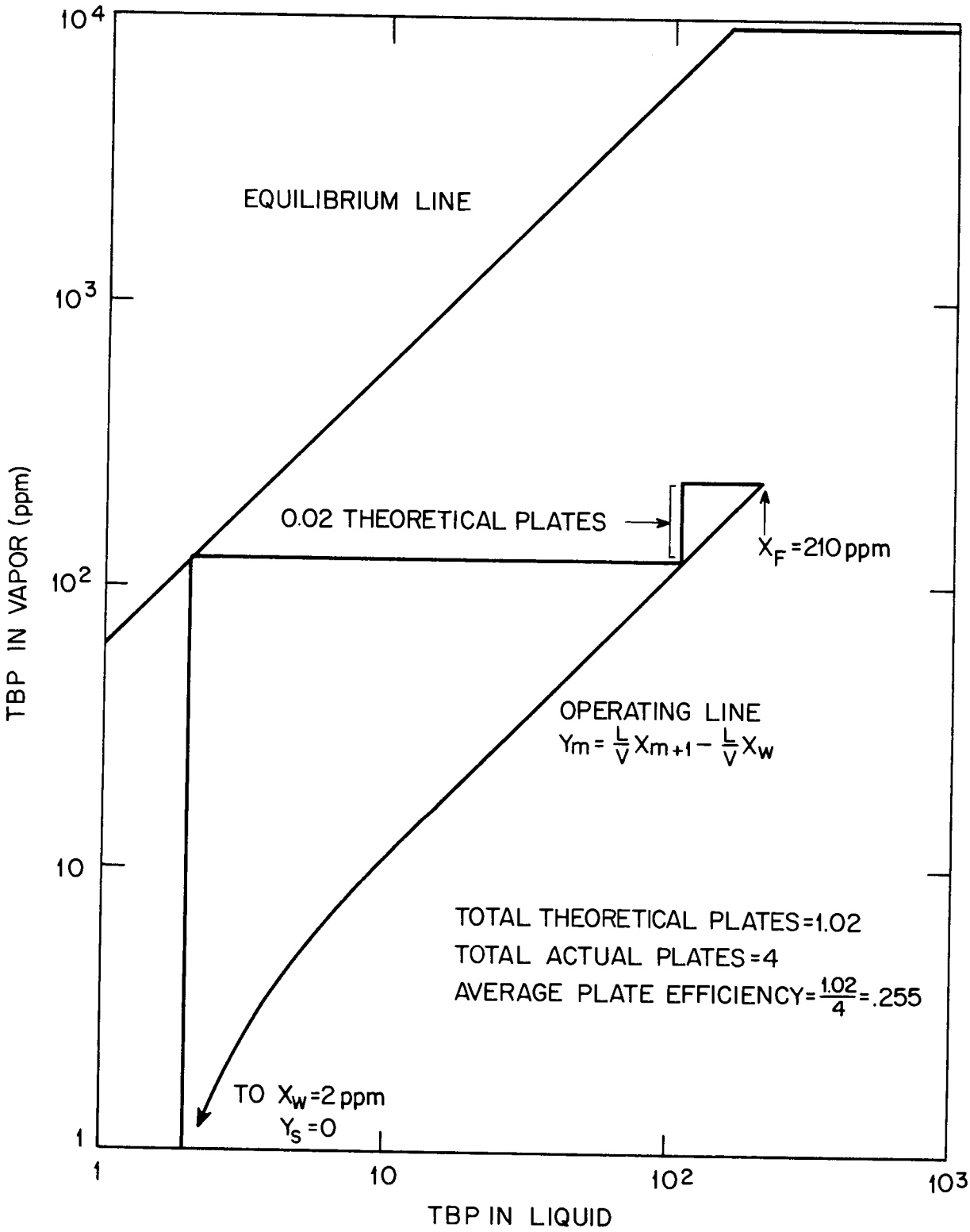

Fig.5. Calculation of Theoretical Plates in Steam Stripping of Tributyl Phosphate from Aqueous Solution.

Table 1. Summary of TBP Steam Stripping Runs

Run No.	Feed Plate	Feed Temp (°C)	Feed			Bottoms			Condensate			TBP Balance (mg/min)				Reflux[a] (ml/min)	Vapor Rate (ml cond/min)	L/V[b]	f[b,c]	Theoretical Plates	Av Plate Efficiency[b] (%)
			Rate (ml/min)	TBP Conc (mg/l)	U Conc (g/l)	Rate (ml/min)	TBP Conc (mg/l)	U Conc (g/l)	Rate (ml/min)	TBP Conc (mg/l)	U Conc (μg/l)	Feed	Bottoms	Condensate	Net Apparent Production						
1	4	20	586	420	~70	661	31	~60	308	430		246	20	132	−94	0	382	1.73	0.36	0.25	06
2a	4	20	546	210	~70	655	6	~60	426	260		117	3.9	111	−2	0	496	1.32	0.47	0.60	15
2b	4	20	542	220	~70	617	3	~60	426	260		119	1.9	111	−6	0	495	1.25	0.47	1.00	25
2c	4	20	550	215	~70	635	4	~60	424	270		118	2.5	114	−1	0	494	1.29	0.47	1.00	25
3a	4	100	567	260	~70	560	2	~60	454	320		147	1	146	0	0	454	1.23	0.43	1.02	26
3b	4	100	567	260	~70	630	4	~60	460	260		147	2	121	−24	0	454	1.23	0.44	1.00	25
4a	4	20	440[b]	360	52.3	568	2	45.8	445	250	70	158	1	111	−46	0	501	1.14	0.47	1.05	26
4b	4	20	360	290	50.1	565	1	43.5	450	220	90	104	5	122	+23	0	496	1.14	0.47	1.05	26
4c	4	20	443	300	45.8	575	2	32.6	445	280	50	129	1	124	−5	0	501	1.15	0.47	1.04	26
5a	2	20	450[b]	290	50.1	578	16	41.4	450	280	70	131	9	126	+4	0	507	1.14	0.47	0.25	13
5b	2	20	453	260	50.1	540	21	42.5	452	280	60	118	11	131	+24	0	509	1.06	0.48	0.18	09
5c	2	20	485	280	47.9	570	21	41.4	452	310	80	136	12	140	+16	0	513	1.10	0.48	0.20	10
6a	2	100	512[b]	520	50.6	555	20	49.0	500	220	40	266	11	110	−145	0	500	1.11	0.47	0.46	23
6b	2	100	498	640	50.7	558	32	49.0	488	290	50	319	18	141	−60	0	488	1.14	0.46	0.36	18
6c	2	100	550	440	50.6	560	33	49.0	490	340	90	242	19	167	−56	0	490	1.14	0.46	0.23	11
7a	4	100	570[b]	520	50.6	565	4	47.9	492	380	70	296	2	188	−106	0	492	1.15	0.46	1.04	26
7b	4	100	558	300	51.2	560	27	47.9	490	410	70	168	15	200	+47	0	490	1.14	0.46	0.19	05
7c	4	100	584	310	50.1	562	9	47.9	495	360	80	181	5	178	+3	0	475	1.14	0.47	0.64	16
8a	4	20	704[b]	240	54.2	848	4	45.2	647	200	170	169	3	168	+2	0	737	1.15	0.70	0.94	23
8b	4	20	695	250	54.2	853	4	45.2	673	300	200	174	3	202	+31	0	761	1.12	0.72	0.94	23
8c	4	20	695	240	54.5	855	1	45.5	647	330	310	167	1	213	+46	0	735	1.16	0.69	1.09	27
8d	4	20	722	240	53.9	857	2	45.2	653	330	340	173	2	216	+43	0	744	1.15	0.70	1.03	26
9a	4	20	209[b]	360	54.5	284	4	42.2	206	360	80	75	1	74	0	0	244	1.16	0.23	1.00	25
9b	4	20	223	350	54.5	290	5	42.2	201	300	70	78	1	60	−11	0	244	1.18	0.23	1.02	25
9c	4	20	198	280	54.5	294	4	42.2	203	270	80	56	1	55	0	0	244	1.20	0.23	1.01	25
9d	4	20	206	280	54.5	274	3	42.2	204	270	60	57	1	55	−1	0	244	1.12	0.23	1.01	25
10a	4	20	430[b]	370	42.5	1153	1	33.8	440	220	90	159	1	98	−60	56	540	2.13	0.51	1.03	26
10b	4	20	377	370	42.5	620	1	32.6	453	300	40	139	1	136	−2	56	540	1.15	0.51	1.06	27
10c	4	20	472	250	42.5	567	<1	32.5	460	310	60	118	1	143	+15	56	540	1.05	0.51	>1.05	>26
10d	4	20	437	210	43.6	493	14	32.5	460	320	40	135	8	152	+25	56	540	1.10	0.51	0.40	10
11a	4	20	721[b]	320	43.6	913	10	34.9	700	270	60	231	9	189	−33	47	814	1.12	0.77	0.48	12
11b	4	20	695	290	43.6	887	2	34.9	700	400	80	201	2	270	+71	47	814	1.09	0.77	1.03	26
11c	4	20	721	280	43.6	937	7	34.9	700	320	80	202	7	224	+29	47	814	1.15	0.77	0.51	14
11d	4	20	746	290	43.6	940	8	34.2	700	340	80	216	8	238	+30	47	814	1.15	0.77	0.64	16
12[d]	4	20	461	1575	48.6	570	9.5	39.5	426	410	10,000	725	6	174	−545	0	500	1.14	0.47	1.09	27
13[d]	4	20	543	1176	48.5	547	10	39.3	532	1240	360	489	5	659	+175	0	590	0.93	0.56	1.04	26

[a]Reflux is water added to sixth plate of column to de-entrain uranyl nitrate.

[b]Calculated values.

[c]f = (superficial vapor velocity)(vapor density)$^{1/2}$.

[d]Values for runs 12 and 13 are averages of four readings during the run.

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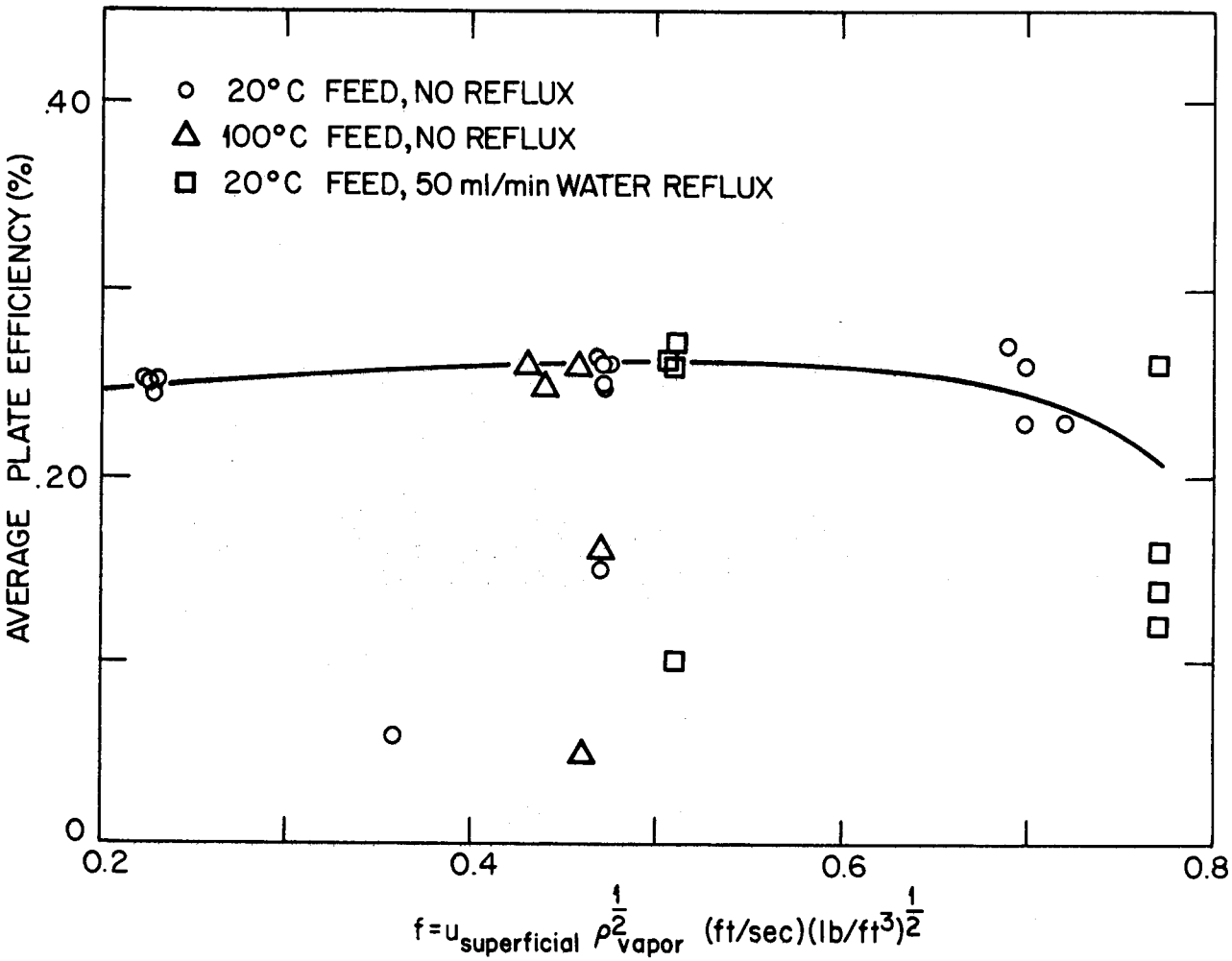

Fig. 6. Average Plate Efficiency vs f Factor in Steam Stripping of Tributyl Phosphate from Aqueous Solution. Conditions: 6-in-dia sieve-plate column, 2.73% free area; L/V =1.15; feed on 4th plate.

The analysis is difficult, involving the extraction of TBP into carbon tetrachloride before measuring the concentration. In the low concentrations, <10 ppm, the results are good only to 1 ppm. Since the number of theoretical plates, and hence plate efficiency, is a very sensitive function of bottoms concentration, the fact that plate efficiencies are as consistent as they are is a tribute to the skill of the group doing the analyses.*

5.3 Hydrolysis

At the temperature in the column TBP hydrolyzes. The reaction is first order with respect to TBP concentration, and is promoted by nitric acid, uranyl nitrate, and light. Extrapolation of the TBP hydrolysis data in HW-40910 gives a hydrolysis rate constant of 10^{-1} per hour at 100°C in 0.25 M $UO_2(NO_3)_2$ and 0.05 M HNO_3, approximately the conditions in the ORNL column. Since the holdup per plate was very close to 1 min at a feed rate of 500 ml/min, the fraction of the TBP lost by hydrolysis on the plate was 1/600, or about 0.2%. In view of other errors in the experiment, this loss is negligible. The TBP balances for the several runs showed no significant loss of TBP in the system, although the data are scattered.

5.4 Entrainment

Figure 7 is a plot of uranium entrainment in the condensate as a function of the f factor. Inspection of the condensate analyses shows that uranium concentrations of 50-100 μg/liter of condensate were carried over in the runs at f factors of 0.23-0.5. At an f factor of 0.7 entrainment rose to about 300 μg/liter. Addition of about 50 ml/min water reflux to the sixth plate (and simultaneous 10% increase of the steam rate) reduced this entrainment to the 50-100 μg/liter level.

The extremely high uranium concentrations in the condensate in the twelfth and thirteenth runs were the result of equipment contamination from another experiment, and were not plotted.

5.5 Operating Range

With L/V = 1.15, the column flooded at an f factor of about 1.0. This flooding rate checks well with a calculated f factor of 1.045 based on pressure drop of the steam through the holes assuming that the familiar orifice equation is applicable. Appreciable weeping occurred at an f factor between 0.1 and 0.2.

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*Done by G. R. Wilson's group, Analytical Chemistry Division, Oak Ridge National Laboratory.

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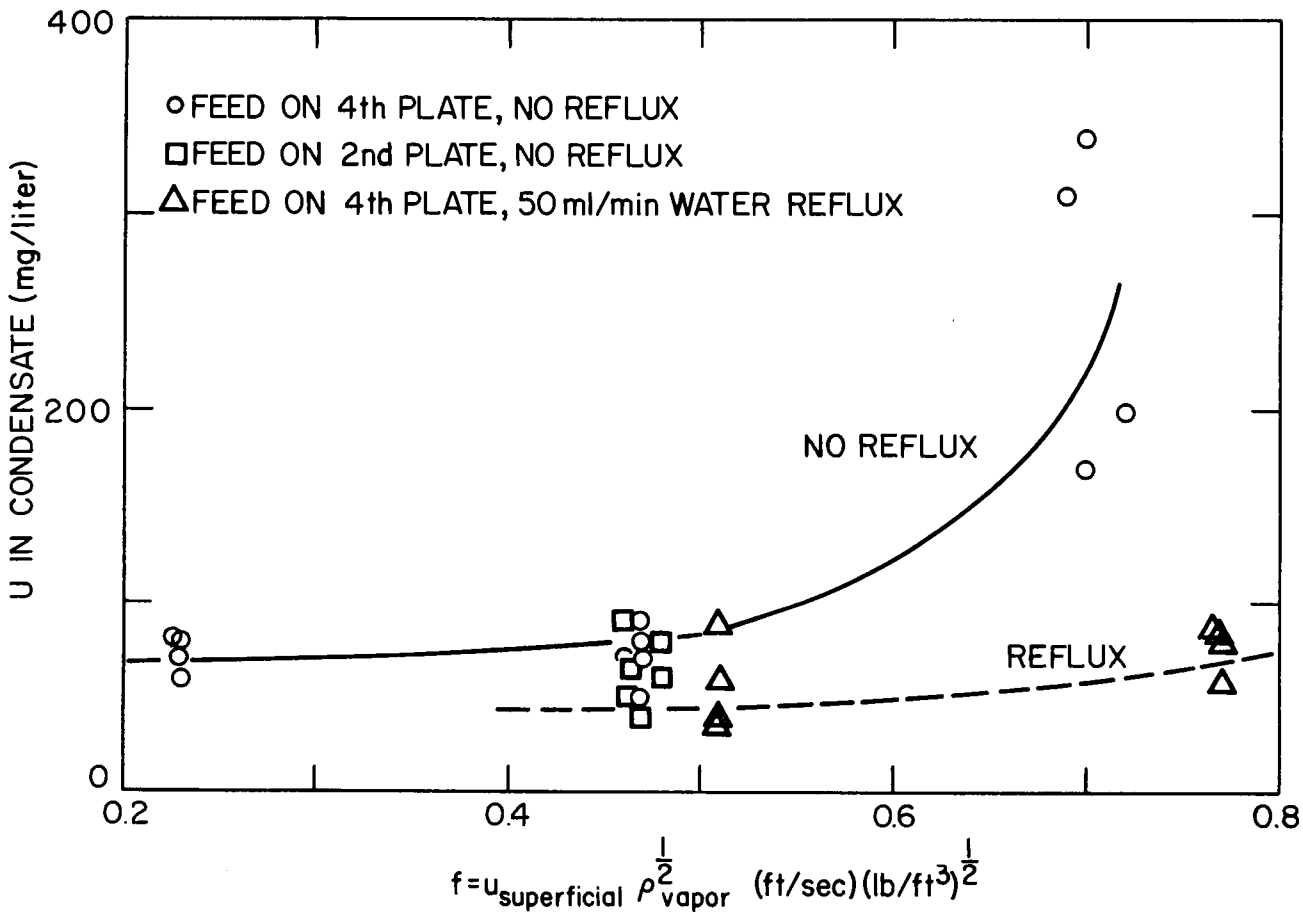

Fig. 7. Uranium Entrainment vs f Factor in Steam Stripping of Tributyl Phosphate from Aqueous Solution. Conditions: 6-in-dia sieve-plate column, 2.73% free area; L/V=1.15; 47g of uranium per liter in feed.