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THE VALVE ACTUATED PULSE COLUMN-II A STUDY OF THE TEMPERATURE EFFECT AND DESIGN VARIABLES

BY

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TECHNICAL SECTION
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FEBRUARY 16, 1953

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February 16, 1953

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A STUDY OF THE TEMPERATURE EFFECT AND DESIGN VARIABLES

I. INTRODUCTION

The "idealized" pulse column operation consists of a four-step flow pattern which can be described by: (1) dispersion of the extractant, (2) settling or coalescence of the extractant, (3) dispersion of the feed and (4) settling of the feed. In a previous report, ⁽¹⁾ a "valve-actuated" pulse column was described with which it was possible to control each of the above four steps independently. In the more conventional pulse column operation there is considerable departure from this sequence in that one phase usually remains continuous. Further, there is no sharp boundary between the dispersing and coalescing steps. However, there are many factors in common for both the former type of operation, "mixer-settler", and the latter which has been designated "emulsion" operation.

There are theoretical reasons and in some cases experimental data as a guide for suggesting many changes in design and operational variables. Since maximum extraction occurs on interface formation, for example, it would seem that continual formation and re-formation of small drops is desired, provided that a reasonable flow rate is maintained. Since viscosity is decreased and diffusion increased with rising temperature, it is to be expected that more efficient extraction would occur at higher temperatures. In the latter case the shifted equilibrium distribution is a further factor.

This report lists some results of testing a number of pertinent variables such as plate design and spacing, pulse frequency and amplitude, and temperature.

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(1) By increasing the operating temperature, a greater extraction efficiency is achieved. This is true even when accompanied by an adverse equilibrium shift.

(3) A decrease in plate hole size increases efficiency by producing a better dispersion of smaller drops. High flow rates can be maintained even with 0.020 inch diameter holes.

(5) Increased temperature facilitates operational control of the column and at the same time permits operation at higher frequencies and flow rates due to more rapid dispersion and coalescence.

The column and associated equipment are the same as described in the first report. ⁽¹⁾ The plate section used was only 17 inches in length and one inch in diameter. Four types of plates were used in this study and are described in Table I.

<u>Designation</u>	<u>Hole Diam. In.</u>	<u>% Open Area</u>	<u>Facing Type</u>
A	0.039	9	Double, fluorothene- stainless steel
B	0.026	15.5	
C	0.020	23	Stainless steel, Kel-F coated on one side
D	slots 3/16 x 0.014	9	

The plates were oriented in the column so that the rising organic phase strikes the organic wetted fluorothene and the falling aqueous phase strikes the water-wetted stainless steel surface. Plate spacings used in these studies were 1/2 inch, 1 inch and 2 inches.

The frequency, or number of pulse cycles per minute is the reciprocal of the combined time intervals for the pulse "on times" and the delay "off times."

Extraction and stripping operations for both metal recovery systems using 12 1/2 % TBP⁽²⁾ and Purex systems using 30% TBP have been carried out with the column. The Purex IC stripping system was chosen for a more complete study. The organic phase consisted of 30% TBP in Amsco 125-90W diluent and contained 200 g/l uranyl nitrate and 6-8 g/l nitric acid. The present Purex flowsheet⁽³⁾ shows a concentration of 155 g/l $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and about 1 g/l HNO_3 in the IC feed, but this change would not materially affect the operation. Distilled water was used for the stripping phase.

Temperatures of the incoming streams were controlled by nichrome heating elements wound on the inlet tubes. The column temperature was further controlled with the aid of a transparent jacket through which thermostated water was circulated. Temperatures at strategic points were measured with thermocouples.

Since data for these extraction systems at varying temperatures were not available or at best incomplete, the calculations for the runs were originally based on the 25 C equilibrium curves. Upon the completion of some very efficient Purex IC experiments, it became apparent that the accepted 25 C Purex IC equilibrium curve (EP-28) did not accurately represent the system being investigated. The product (ICU) was falling on a point below or on the EP-28 equilibrium line giving an indeterminable number of stages. In order to correctly evaluate the Purex IC experiments, equilibrium data for this system at 0, 25, 40 and

60 C have been experimentally determined.⁽⁴⁾ These data are given in Table II and plotted in Figure 1 for the countercurrent extraction of 200 g/l UNH, 6 g/l HNO₃ with distilled water. With the aid of these data, HTU's can be computed at the actual temperature of the experiment.

TABLE II
EQUILIBRIUM DATA FOR PUREX IC AT VARIOUS TEMPERATURES

Aqueous Phase Composition g/l UNH	Organic Phase Composition, g/l UNH			
	0 C	25 C	40 C	60 C
400	240	231	228	221
200	208	186	164	143
100	143	110	82	58
60	79	49	32.5	21
20	8.0	3.4	2.1	1.22
10	1.40	0.52	0.33	0.20
5	0.23	0.086	0.054	0.032
2	0.035	0.021	0.015	0.008
1	0.02	0.013	0.008	0.005

IV. RESULTS AND DISCUSSION

A. EFFECT OF TEMPERATURE

The data given in Table III are representative runs for a total of 225 experiments. It is seen from this table that the waste loss has been reduced for all the systems by elevating the temperature.

For the Purex IC system using the .020 inch diameter holes, the uranium concentration of the waste streams are in the region where accurate analysis is difficult and a comparison of individual runs is not very meaningful. The increased extraction at the higher temperature can still be seen although for these runs the differences are not as great as for the others. It is worth recalling that as the extraction apparatus becomes more efficient, a point will be reached where the only benefit from increased temperature operation of the IC column will be the shifted equilibrium line.

TABLE III

OPERATING DATA FOR PULSE COLUMN

Run No	Flow Ratio Aq/Org	Flow (Total) gal/ft ² /hr	Cycles per Min	Valve "On" Time (sec)		Valve "Off" Time (sec)		Operating Pressure (psig)		Mat. Bal. (%)		Waste Conc. UNH(g/l)	HETS (in)	HTU (in)	Temp °C
				Organic	Aqueous	Org-Aq	Aq-Org	Org	Aq	UNH	HNO ₃				
Purex IC Data (Plate Design A-1) 14 experiments															
II-PC-1m	1.53	380	27	0.10	0.21	1.05	0.82	22	12	103	107	66.3	15.4	12.3	25
II-PC-1a	1.55	605	35	0.10	0.21	1.05	0.35	25	15	99	102	19.0	10.7	6.29	60
II-PC-1e	1.47	530	35	0.10	0.21	1.05	0.35	25	14	109	109	10.9	8.5	5.06	75
Purex IC Data (Plate Design B-1) 21 experiments															
VI-PC-1d	1.49	468	30	0.10	0.12	1.06	0.71	42	45	103	116	39.0	9.45	7.46	26
VII-PC-2b	1.47	437	35	0.10	0.21	0.74	0.66	19.5	35	101	96	1.0	5.67	2.84	52
Purex IC Data (Plate Design B-2) 17 experiments															
IX-PC-1b	1.53	505	28	0.10	0.23	1.05	0.79	35	22	93.5	87	52.4	13.3	10.20	26
IX-PC-1h	1.53	670	40	0.10	0.12	0.83	0.45	25	20	99	96	41.8	10.0	8.14	26
X-PC-1b	1.44	640	27	0.10	0.25	1.05	0.82	14	13	104	98	23.0	9.6	6.47	59
X-PC-1f	1.48	625	48	0.10	0.13	0.74	0.29	16	7	101	111	2.75	6.48	3.41	59
Purex IC Data (Plate Design C-1/2) 14 experiments															
XII-PC-1f	1.40	175	33	0.15	0.24	0.86	0.50	14.5	18	99	110	0.14	2.63	1.61	25
XII-PC-1e	1.55	185	33	0.15	0.24	0.86	0.50	14.5	17.5	105	118	0.10	3.22	1.68	25
XIII-PC-1a	1.40	395	33	0.15	0.24	0.86	0.50	14	19	99	110	0.042	5.15	1.83	63
XIII-PC-1f	1.54	403	38	0.15	0.40	0.66	0.37	27	15	103	130	0.026	4.90	1.77	63
Purex IC Data (Plate Design C-1) 38 experiments															
XVI-PC-1c	1.47	311	32	0.15	0.74	0.57	0.42	32	8.5	100	105	9.5	4.42	3.48	25
XIV-PC-1g	1.56	327	41	0.15	0.31	0.69	0.35	16.5	17.5	103	124	3.5	4.54	3.13	25
XIV-PC-1h	1.50	349	59	0.15	0.28	0.56	0.07	13.5	23	102	132	1.74	3.81	2.48	25
XVI-PC-2d	1.50	710	33	0.15	0.98	0.34	0.42	34	8	99	108	0.13	5.15	2.08	62
XV-PC-1d	1.58	627	41	0.15	0.30	0.56	0.47	22	17	108	95	0.053	5.08	1.92	62
XV-PC-1g	1.49	510	62	0.15	0.22	0.20	0.40	22.5	28	100	114	0.024	4.64	1.73	62

TABLE III (contd)

OPERATING DATA FOR PULSE COLUMN

Run No	Flow Ratio Aq/Org	Flow (Total) gal/ft ² /hr	Cycles per Min	Valve "On" Time (sec)		Valve "Off" Time (sec)		Operating Pressure (psig)		Mat Bal (%)		Waste Conc UNH(g/l)	HETS (in)	HTU (in)	Temp °C
				Organic	Aqueous	Org-Aq	Aq-Org	Org	Aq	UNH	HNO ₃				
Purex IC Data (Plate Design C-2) 21 experiments															
XVII-PC-1g	1 50	434	31	0 15	0 45	0 81	0 49	35	12	103	100	45 9	10 6	8 54	25
XVII-PC-1k	1 51	425	55	0 15	0 56	0 45	0 01	37	9	98 6	87	14 1	5 56	4 27	25
XVIII-PC-1l	1 45	700	48	0 15	0 82	0 29	0 01	37	9	95	98	0 64	5 54	2 65	60
Purex IC Data (Plate Design D-1) 19 experiments															
XX-PC-1f	1 46	495	50	0 15	0 16	0 63	0 34	10	43	102	100	61 3	14 7	11 5	24
XX-PC-2c	1 48	1070	59	0 15	0 58	0 31	0 01	14	14	101	89	12 1	5 0	3 84	60
Purex IC Data (Plate Design D-2) 12 experiments															
XIX-PC-1j	1 49	450	27	0 15	0 61	0 83	0 50	41	31	101	99	55 1	13 6	10 4	25
Metal Recovery RA Data (Plate Design A-1) 18 experiments															
IV-PC-2a	0 615	928	86	(Conventional Type Operation)						107	96	15 8	15 5	---	23
IV-PC-2r	0 60	920	180	(Conventional Type Operation)						99	105	8 6	12 0	---	65
Metal Recovery RC Data (Plate Design A-1) 31 experiments															
I-PC-2a	0 78	410	28	0 10	0 15	1 05	0 82	28	15	107	110	20 7	32 1	---	25
I-PC-4d	0 80	490	43	0 10	0 13	0 57	0 59	24	12	103	98	12 5	21 0	---	35
I-PC-5l	0 78	400	28	0 15	0 15	1 15	0 82	21	12	107	94	2 9	12 8	---	63
Purex IA Data (Plate Design B-1) 21 experiments															
V-PC-4b	2 03	560	43	0 13	0 14	0 48	0 65	15	13	102	100	0 87	7 5	---	28
V-PC-4i	2 03	400	50	0 10	0 12	0 52	0 45	9	5	99	100	0 058	5 7	---	57

The IC equilibrium curves at the higher temperatures fall below that of the 25 C curve. Thus, in terms of transfer stages, the beneficial effects of operating a stripping column at the higher temperatures are lower stage heights as well as a fewer number of stages for a given waste loss. For example, in the RC column with a flow ratio (aq/org) of 0.785 and a feed composition of 41.7 g/l UNH, 6.3 g/l HNO₃, in 12.5 volume per cent TBP in shell Spray Base, the number of theoretical stages to reduce the RCW to 0.1 g/l UNH would be 3.0 and 2.3 stages at 25 and 40 C, respectively. For the Purex IC system with a flow rate (aq/org) of 1.50 and a feed composition of 200 g/l UNH, 6 g/l HNO₃, 30 volume per cent TBP in Amsco-125, the number of theoretical stages to reduce the ICW to 0.02 g/l UNH would be 5.9 and 3.7 at 25 and 60 C, respectively.

Typical results are compared in Table IV on a HETS basis for the Purex IC System using a 17 inch extraction section:

TABLE IV

HETS VALUE CALCULATED FOR DIFFERENT EQUILIBRIUM CURVES

<u>Run No.</u>	<u>C Temp.</u>	<u>EP-28 (25 C)</u>	<u>Our 25 C Curve</u>	<u>Our 60 C Curve</u>	<u>ICW (g/l UNH)</u>
lg	62	1.84	2.88	4.64	0.024
li	60	2.28	3.19	5.54	0.64
la	63	1.62	2.81	5.15	0.04

The effect of temperature on the number of transfer units is graphically represented in Figure 2. Each point represents the average of three to six runs each. If the assumption is made that the number of transfer units is a measure of the transfer rate, then an overall "activation energy", ΔE^* , can be calculated for the transfer of uranyl nitrate from the organic to the aqueous phase.

$$\frac{d \ln N_t}{d 1/T} = - \frac{\Delta E^*}{R}$$

For the Purex IC data, the calculated "activation energy" is found to fall in the range of -1.97×10^3 to -3.78×10^3 cal/mol. The significance of such a calculation is rather nebulous when one considers the basis for the HTU concept.

In the case of the RA and IA extraction systems the shift of the equilibrium line will hinder extraction of uranium by the organic phase. It will be noted from Table III, however, that even for these systems a rise in temperature increased the efficiency of extraction. This is a result of the fact that an extraction column is a non-equilibrium system and the rate constants of the forward reaction to a first approximation determine the operating efficiency.

In addition to the more favorable extraction efficiency of the column at elevated temperature, it was usually observed that coalescence of drops was improved greatly at higher temperatures which resulted in decreased emulsification and increased column throughput. This is probably largely a viscosity effect.⁽⁵⁾ It will be seen from Table V that a change in temperature from 25 C to 60 C reduces the viscosity of both the organic phase and the aqueous phase by about 45-50%.

B. EFFECT OF HOLE SIZE

The efficiency of the column increases with decrease in plate hole diameter. Figure 3 gives this relationship for the Purex IC system. This increased efficiency is believed largely due to the nature of the dispersion. The very small droplets developed by the 0.020 inch diameter holes present an extremely large area for surface contact and hence, improved extraction.

The flow capacity of the column decreases somewhat with the smaller plate hole diameter, as may be noted from Table III.

An attempt was made to determine the effect of hole shape. From the experiments using the slotted holes it is evident that the slotted plates

TABLE V
VISCOSITIES OF METAL RECOVERY
AND PUREX STREAMS AT VARIOUS TEMPERATURES

<u>Solution</u>	<u>Millipoises</u>				
	<u>0 C</u>	<u>25 C</u>	<u>40 C</u>	<u>60 C</u>	<u>75 C</u>
H ₂ O	17.9	8.9	6.56	4.69	3.80
RCU, HW # 4	19.1	9.5	7.1	5.0	4.1
RAFS, HW # 3	36	18.2	13.5	9.6	7.8
RAX, 12.5% TBP in Amscol 125-90W	28	15.3	11.7	8.4	7
RAX, 12.5% TBP in Spray Base	34	18.4	13.8	10.1	8.4
ICW, 30% TBP in Spray Base ORNL # 2	38	20.5	15.1	11	9.3
ICF, 30% TBP in Spray Base ORNL # 2	66	33.3	23.7	17	14
RAU, 12.5% TBP in CCl ₄	21	14.1	11.2	8.5	7

did not give as efficient extraction as the .020 inch diameter hole plates. Ease of operation was very good and high flow rates could be obtained, an interesting observation when considered in conjunction with the small (9%) open area of these plates. However, it was noted that the slotted plates did not give a fine dispersion but instead produced large drops. Although this condition may be conducive to high throughput, the low contact area does not favor efficient extraction. It must be admitted that the effect of varying hole shape has not been adequately treated since irregular shaped holes have a marked effect on the liquid jet formed. Slotted plates have not been tested on "emulsion type" operation. When it is more difficult to form one type of dispersion, e. g., organic in aqueous

than the opposite type, aqueous in organic, then an assymetric hole is a possible aid. The double-faced plate used in these studies provides in a sense an assymetric hole because of its wetting characteristics.

C. PLATE SPACING

The increase in efficiency of the pulse column as the plate spacing is decreased is shown for the Purex IC system by Figure 4. The flow capacity of the column goes up with increase in the plate spacing. By doubling the plate spacing; i. e., from 1 inch to 2 inches, the maximum throughput is raised about 40 to 70 per cent. Low viscosities become very important with plate design involving small holes and close spacings.

At the 1/2 inch spacing it was difficult to obtain complete coalescence between plates during the "off" time between pulses. This resulted in an emulsion type operation which helped lower the capacity of the column.

D. FREQUENCY

The extraction efficiency of the column varies directly with the frequency as is shown in Figure 5. With this type of column operation it became increasingly difficult to maintain the interface as the frequency was increased above 50. The capacity of the column increased directly as the frequency until an emulsion was reached. At this point, the interfaces could no longer be maintained. Successful column operation was achieved at frequencies ranging from 25 to 100 cycles per minute. The true limiting frequency for this type of operation was not actually determined since the optimum combination of on-times and off-times for minimum workable cycle time appeared to be outside the limit of the timing mechanism employed.

E. PULSE SHAPE

If the principle is accepted that efficient transfer between phases necessitates the formation and re-formation of as large an interfacial

area as is consistent with subsequent phase separation, then it follows that a pulse having a sharp wave front, e. g., a square wave or saw-tooth wave as opposed to a sine wave, is desirable. This has been the author's premise from the start. It may be noted that the efficiency of this column in terms of either HTU's or waste loss for the systems studied is quite high even with the more unfavorable combinations of design variables. It is believed that the pulse shape which approaches the square wave, contributes greatly to this efficiency. Aside from changing the pressure of the feed streams and the duration of the pulse, no attempt was made to control the pulse form primarily because no method was at hand for actually measuring the wave form. Increasing the pressure appears to increase the efficiency although the significance of this is clouded by the fact that the feed pressure cannot be changed independently of other variables.

F. LIMITING EFFICIENCY OF THE COLUMN

The limiting efficiency of any apparatus will be approached asymptotically. It is not surprising that as a number of beneficial factors are combined the degree of improvement with each succeeding factor becomes less. This is illustrated by the effect of temperature on HTU values. For more efficient plate designs, e. g., small holes, the drop in HTU as the temperature rises becomes less. In making a comparison of HTU's or waste losses however, other factors must be kept in mind. The most important one is the shift of the equilibrium curve with changing temperature. As was discussed earlier, for the "C" column at a higher temperature a fewer number of transfer units will be needed to produce a given waste loss.

The different transfer rates of uranyl nitrate and nitric acid enters the picture in a complicated manner since each has a salting effect on the other. No attempt has been made to evaluate this problem although it is probably more important in the Purex "IA" extraction column, for example, than in the "IC" column studied here. Finally it should be mentioned

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that analytical errors are larger in the low concentration region tending to give more scatter to highly efficient runs.

V. CONCLUSION

These studies have utilized the mixer-settler principle of pulse column operation with each step of the four stage cycle separated. It seems clear that with mixer-settler operation the use of small holes in the plates - thus producing small drops is desirable. Forming the drops more often by closer plate spacing and higher frequency is also advantageous and the nature of the pulse should be such that the velocity of liquid through the holes is high enough to produce dispersion over a large portion of the pulse time. The use of double-faced plates which promote more rapid coalescence permits one to extend the range of the above variables and realize to a fuller degree the benefits of mixer-settler operation.

In addition to the increased extraction efficiency achieved by elevated temperature operation, there are definite operational advantages. The first noticeable change is the improvement in coalescence. The better phase separation at elevated temperatures permits operation at higher frequencies which permits increased throughput as well as easier interface control. When there were definite tendencies for emulsion formation, raising the temperature of both aqueous and organic streams reduced the trend.

A practical limit on elevated temperature operation is imposed by the flash point of the diluent. For Amsco 125-90W it is 133 F and for "Spray Base" 150 F. ⁽⁶⁾ A second consideration is chemical stability. TBP hydrolysis although slow is accelerated as the temperature is raised and could lead to high plutonium losses or poor decontamination through complex formation. R. L. Moore ⁽⁷⁾ using batch contacting with TBP at higher temperatures obtained results which indicated that this might be a problem.

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There still is reason to believe that the reactions are slow enough to permit the more rapid column operations at 50-60 C, at least in cases when high fission products decontamination is not a problem.

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ABSTRACT

The valve-actuated pulse column is employed to give phase separation on each half cycle in the countercurrent flow cycle and provides independent control over each step in the cycle. The dependence on temperature of operation, on pulse frequency and amplitude, and on plate design and spacing has been studied for extraction and stripping of uranium with tributyl phosphate in a hydrocarbon diluent.

It is found that the efficiency increases quite significantly as the temperature is raised regardless of the direction of transfer and this increased efficiency is accompanied by greater operational freedom. A decrease in hole size, decrease in plate separation and increase in frequency all lead to higher efficiency in agreement with the concept that small drops continually formed coalesced and reformed, lead to maximum interphase transfer. The high extraction efficiency of the column is probably aided further by the relatively sharp pulse produced.

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FIGURE 1

FIGURE 2

DEPENDENCE OF EXTRACTION EFFICIENCY ON OPERATING TEMPERATURE

FIGURE 3

DEPENDENCE OF EXTRACTION EFFICIENCY ON HOLE DIAMETER

FIGURE 4

DEPENDENCE OF EXTRACTION EFFICIENCY ON PLATE SPACING

FIGURE 5

DEPENDENCE OF EXTRACTION EFFICIENCY ON FREQUENCY

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