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CHEMISTRY - SEPARATION
PROCESSES FOR PLUTONIUM
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THE VALVE-ACTUATED PULSE COLUMN DESIGN AND OPERATION

AEC RESEARCH AND DEVELOPMENT REPORT

BY

L. L. BURGER AND L. H. CLARK

TECHNICAL SECTION
ENGINEERING DEPARTMENT

DECEMBER 3, 1951

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Chemistry-Separation
Processes For Plutonium
And Uranium

AEC RESEARCH AND DEVELOPMENT REPORT

THE VALVE-ACTUATED PULSE COLUMN DESIGN AND OPERATION

By

L. L. Burger and L. H. Clark
Chemical Research
Separations Technology Unit

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-4-

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THE VALVE-ACTUATED PULSE COLUMN
DESIGN AND OPERATION

INTRODUCTION

The pulse column, originally described by Van Dijck⁽¹⁾ for liquid-liquid extraction combines many advantageous features that are present in batch contactors and in packed columns. Its virtue of having mechanical energy imparted to the extraction process in a countercurrent column was recognized by C. Groot who introduced the pulse column to the extraction problems of the atomic energy program⁽²⁾ and demonstrated the greater efficiency of the pulse column over the conventional packed column. Application of the column to the Redox process was described by Burns, Groot and Slansky⁽³⁾ and subsequently the unit came into widespread use at AEC installations.

The many design and operational variables such as plate spacing, size and number of holes per plate, pulse size and frequency are largely interdependent, and when one adds to these the variations in the physical properties of the systems, he is confronted with design and operational problems that are largely subject to solution only trial and error.

Some effort has been expended in an attempt to obtain theoretical relations between some of these variables and various aspects of pulse column behavior such as drop size, rate of movement of drops, throughput and extraction efficiency during a given part of the cycle.⁽⁴⁾ These considerations were based on an idealized pulse column cycle consisting of four stages: (1) dispersion of phase I into phase II, (2) settling or disengaging of phase I, (3) dispersion of phase II into phase I, and (4) settling of phase II. Unfortunately, in ordinary pulse column operation these stages are not clearly defined due largely to the fact that only one phase is pulsed and to a certain amount of "back-up" in the connecting lines. 902

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Further, the sine-wave nature of the conventional pulse cannot lead to a sharp separation of the above four stages.

A study of the fundamental extraction process was already underway at Hanford Works, and it was apparent that the idealized four stage cycle could be a convenient tool for further extraction studies particularly if the four stages could be made independent. At the same time such a study should provide information for more efficient design and operation of pulse columns.

A convenient method of meeting the above requirements is the control of each stream with solenoid valves. The flow or pulse size of a particular phase then becomes proportional to the product of the pressure ahead of the valve and the time interval that the valve is open. The second phase remains stationary during this interval. After a suitable independently timed delay, the second phase is then pulsed in the same manner.

The advantages of this scheme are a much greater degree of separation of the operating variables and the wide range of operating conditions. The purpose of this report is to present details of construction and operating data for a small research-type valve-actuated pulse column.

SUMMARY

A countercurrent extraction column is described which employs timed solenoid valves and pressurized feeds to provide a pulsing action to disperse the phases. The steps in the pulsing cycle are independent and thus provide greater separation of the operating variables than is possible with conventional pulse columns. The column described is particularly useful as a research tool for the study of extraction mechanism although a larger installation operating on the same principles should be quite workable. The 4-stage-cycle or mixer-settler type column operation has an

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inherent advantage in that the rapid coalescence and redispersion of phases are believed to lead to greater extraction than simple movement of a dispersed phase through a continuous medium. In addition, the sharp pulse provided should produce greater turbulence than the more conventional pulsing arrangement. Some results of operation on the extraction and stripping of uranyl nitrate using tributyl phosphate as a solvent are included. These data correspond to Metal Recovery and Purex type systems.

DESIGN AND CONSTRUCTION

A complete schematic diagram is given in Figure 1 and details of the column construction are shown in Figure 2. Aside from the column itself, the essential components are the pumps which provide the feeds at the pressure tanks at the desired pressure, the valves which regulate the pulsed flow into and out of the column and the timer which controls independently the time of flow of each feed and the delay interval between the "feed pulses". The pumps used are positive displacement gear type having a displacement of 1.75 cc per revolution (Zenith Products Company, size # 3). They are driven by 1/15 Hp constant-speed motors at an output shaft speed of about 150 r.p.m. The pressure tank is actually an air ballast of about 1 liter capacity and serves to provide a steady pressure, ahead of the pulse valves. The actual pressure is controlled by the bypass valve.

Solenoid valves (Skinner Electric 115 V, 60 cycles) synchronized in pairs, control the flow or pulse into and out of the column. Available in several types, the ones employed here have a 3/32 inch orifice and are capable of operation up to 75 psig. The valves, as well as pumps and connecting lines, are stainless steel and gasketed where necessary with fluorothene.

The electrical circuit is shown by the dashed lines in Figure 1.

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The heart of this system is the electronic timer which provides four timed circuits individually controlled by four dials on the instrument. Circuit 1 controls the on-time for valves 1 and 3 or the organic inlet and outlet valves, respectively. Circuit 2 provides a delay or dead time following the organic pulse during which there is no flow of liquid in the column. Circuit 3 governs valves 2 and 4 of the aqueous stream and circuit 4 controls the delay period following the aqueous pulse. Once the start switch is closed the cycle is repetitive, each circuit activating the following one. The time intervals of each of the four steps can be varied between 0.1 and 1.2 second. The timer calibrations are shown in Figure 3 and were obtained by using the 60 cycle supply voltage as a time standard. One lead from each timed circuit was attached through a 1000 ohm resistance to a wire spring contact resting on a metal plate. The other lead from each circuit was connected to the plate. A strip of paper about 24 inches long was then painted with a solution of potassium iodide containing a little glycerine and placed between the contacts and the plate. With the timer dials set at selected values the current was turned on while the paper was pulled at a constant rate under the contacts. A series of sharp brown dashes $1/120$ second in length appeared on the paper where free iodine was liberated and the number of dashes gave the time interval directly. These records remained legible for a period of several weeks. The complete schematic of the timer is given in Figure 4. One section of each 6SN7 contains a variable "R-C" circuit. This timed circuit triggers the second section, dropping its plate current to zero which sends a positive pulse to the second 6SN7. Relays in the plate circuits of tubes 1 and 3 provide power to the solenoid valves.

In addition to the timer, external on-off and delay switches provide independent control over the outlet valves of each stream. The control switch indicated in Figure 1 permits the outlet valve to be continuously closed, continuously open, or connected directly to the timer. The delay switch is an auxiliary in the outlet valve circuit which permits the outlet

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valve to open a short time after the inlet valve opens. This control aids in the interface control in the column. Figure 5 shows the diagram of the delay switch. With the constants shown the variable resistance R provides a delay up to about 75 milliseconds.

The column itself is constructed of one inch diameter "True-Bore" tubing which is flared at each end to a two inch diameter. The end sections are shown in details in Figure 2. For the "four-stage" operating principle described above, double-faced or compound⁽⁵⁾ plates are very desirable, the organic-wetted side, fluorothene, being on the bottom for use with a solvent which is lighter than the aqueous phase. At the present time the column has 17 compound plates containing 0.026 inch diameter holes with 15% open area and separated by one inch spacers.

Provisions were made for operating this system at elevated temperatures by the installation of nichrome wire heaters on each inlet tube and a plexi-glass water jacket surrounding the column. The water jacket was constructed in a square design to permit motion pictures of the column in operation with minimum optical distortion. Thermocouples were placed at strategic points as indicated in Figure 1.

OPERATION

The flow rate of the feed or extractant into the column is dependent upon several factors. For a given size solenoid valve, the flow of each phase is proportional to the pressure in the ballast tank and the length of time the valve is open. By regulation of delay times, i.e., the time no liquid is flowing into the column, total flows may be increased or decreased without affecting the flow ratio. The frequency, or complete cycles per minute, is then determined by the sum of the durations of the four stages of the cycle and may be measured with a stop-watch or computed from the valve-open and delay times.

With this column neither phase can be called continuous under the usual terminology, and successful operation of the column is dependent upon the maintenance of steady interfaces at both the top and bottom of the column. A shift of the interface position may be the result of (1) a volume change due to extraction, (2) entrainment of the dispersed phase, (3) air bubbles in the column, or (4) leakage through the closed valves or different operating characteristics of the valves. Factor (1) is inevitable and must be corrected for. Factor (2) generally occurs although the amount may be small. There is no direct evidence of factors (3) or (4) with the present equipment. Control of the interface is the most difficult operating problem and can be achieved to some extent by trial and error manipulation of on-off times and operating pressures. The use of the delay circuits in the outlet valve aids in building up the volume of that phase. A more direct control is provided by the bleeder valves at either end of the column. These are solenoid operated and when required are synchronized to open during the pulse from the opposite end of the column. Thus, while the organic phase is being removed at the top of the column, a small portion of the essentially unchanged aqueous feed can be bled out, through the combination needle valve-solenoid valve to lower the top interface. This feed, a very small fraction of the total, is recycled to the aqueous feed line.

The column has been tested on Metal Recovery and Purex systems involving 15% and 30% tributyl phosphate in a hydrocarbon type diluent.

Typical operation for two different plate geometries are represented by the data in Tables I and II. These plates were double-faced (fluorothene-stainless steel) and were drilled with either .039 inch or .026 inch diameter holes having an open area of 9% and 15.5%, respectively. These two sets of plates are referred to as plate design A and plate design B, respectively. With these plates separated at one inch or two inch intervals, successful operation has been achieved over the following range of

TABLE I
TYPICAL OPERATING CONDITIONS OF THE VALVE-ACTUATED PULSE COLUMN

Run No.	ORGANIC PULSE						AQUEOUS PULSE					
	Operating Pressure (psig)	Pulse Time (sec)	Pulse Amplitude (cm)	Jet Velocity (cm/sec)	Delay Time (sec)	Flow (cc/min)	Operating Pressure (psig)	Pulse Time (sec)	Pulse Amplitude (cm)	Jet Velocity (cm/sec)	Delay Time (sec)	Flow (cc/min)
<u>Plate Design B *</u>												
V-PC-1g(IA)	21	0.10	0.82	53	1.05	115	20	0.20	0.46	15	0.82	65
V-PC-1p(IA)	16	0.12	0.83	44	0.55	156	8	0.23	0.35	10	0.71	66
VI-PC-1g(IC)	50	0.13	0.58	28	0.71	100	52	0.12	0.68	37	0.79	118
IX-PC-1h(IC)**	25	0.10	0.44	29	0.83	90	20	0.12	0.68	37	0.45	138
<u>Plate Design A</u>												
II-PC-1L(IC)	22	0.10	0.37	24	1.05	53	12	0.18	0.54	19	0.82	76
I-PC-3a(RC)	35	0.10	0.48	31	1.05	69	15	0.14	0.43	20	0.82	62

*See text, p. 9.

** 2 inch plate spacing employed for this run. All others were 1 inch.

TABLE II
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)
				Organic	Aqueous	Org-Aq	Aq-Org	Org	Aq	UNH	HNO ₃		
Metal Recovery RC Data (Plate Design-A)*													
I-PC-2a	0.78	410	28	0.10	0.15	1.05	0.82	28	15	107	110	20.7	32.1
I-PC-2b	0.82	408	28	0.10	0.15	1.05	0.82	28	15	92	104	21.3	32.7
I-PC-3a	0.89	384	28	0.10	0.14	1.05	0.82	35	15	105	102	19.4	36.2
I-PC-3h	0.92	610	28	0.15	0.16	1.05	0.80	33	23	98	84	19.2	39.5
I-PC-3f	1.00	677	28	0.15	0.16	1.05	0.80	33	22	103	99	19.4	40.5
Purex IC Data (Plate Design-A)													
II-PC-1L	1.45	380	28	0.10	0.18	1.05	0.82	22	12	97	102	63.6	14.9
II-PC-1j	1.48	310	28	0.10	0.18	1.05	0.82	22	12	95	102	67.3	16.2
II-PC-1k	1.48	320	28	0.10	0.18	1.05	0.82	22	12	105	100	73.8	15.7
II-PC-1m	1.53	380	27	0.10	0.21	1.05	0.82	22	12	103	107	66.3	14.9
II-PC-1n	1.62	384	27	0.10	0.21	1.05	0.82	23	12	96	95	61.5	16.0
Purex IC Data (Plate Design-B)													
VI-PC-1g	1.18	640	34	0.13	0.12	0.71	0.79	50	52	101	110	59.5	8.5
VI-PC-1e	1.47	457	28	0.10	0.12	1.06	0.82	43	45	102	104	42.2	8.5
VI-PC-1d	1.49	468	30	0.10	0.12	1.06	0.71	42	45	103	116	39.0	8.3
VI-PC-1c	1.58	528	39	0.10	0.12	0.62	0.71	40	42	99	109	29.0	8.1
VI-PC-1a	1.66	415	30	0.10	0.12	0.92	0.82	40	40	107	120	39.4	8.5
Purex IA Data (Plate Design-B)													
V-PC-1g	1.77	530	28	0.10	0.20	1.05	0.82	21	20	91	97	2.6	8.0
V-PC-1o	1.95	530	28	0.11	0.25	0.98	0.75	18	10	101	98	0.88	7.0
V-PC-4b	2.03	560	43	0.13	0.14	0.48	0.65	15	13	102	100	0.87	7.5
V-PC-1b	2.14	618	29	0.15	0.19	1.05	0.65	27	22	104	97	1.04	7.8
V-PC-1p	2.37	654	37	0.11	0.23	0.55	0.71	16	8	97	102	0.66	8.0

* See text, p. 9.

operating conditions:

Feed pressures	4-50 psig
Frequency	20-100 cycles per minute
Pulse amplitude	1/8 - 1 inch
Feed pulse time	0.07-0.3 sec
Jet velocity	10-50 cm sec ⁻¹
Delay intervals	0.2-1.5 sec
Flow rate	400-1100 gal ft ⁻² hr ⁻¹
Temperature	25°-70°C.

In Table I actual operating conditions of typical experiments are shown. Table II lists actual operating data for typical room temperature runs. Metal Recovery runs were based on Flowsheet HW# 4⁽⁶⁾ and Purex runs were based on Flowsheet ORNL # 2.⁽⁷⁾ No runs were made on the Metal Recovery "RA" system.

Shell "Spray Base" was used as the diluent giving the organic phase a viscosity of about 18 millipoises and a density of 0.81 gms/cc at 25°C. Although these data represent typical runs on the present equipment, they do not necessarily represent optimum extraction conditions. It is encouraging to note, however, that column efficiencies were in general quite good and for the Purex system HETS values of 7-8 inches were obtained for both extraction and stripping. Detailed consideration of changes in operating variables such as temperature, pulse size, pressure and flow rates, as well as design variables such as hole size and plate separation will be given in another report.

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FIGURE 1
SCHEMATIC DIAGRAM OF VALVE-ACTUATED PULSE COLUMN

FIGURE 2
END SECTION OF PULSE COLUMN

FIGURE 3
CALIBRATION CHARTS OF ELECTRONIC TIMER

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-18-

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FIGURE 5
WIRING DIAGRAM OF DELAY SWITCH

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