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A REVIEW OF THE RELATIVE MERITS OF PACKED COLUMNS, PULSE COLUMNS,
MISER-SETTLER MECHANISMS, AND CENTRIFUGAL CONTACTORS

CHAP. TITLE

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Introduction

Solvent extraction is, at the present time, the optimum method for the separation of uranium, plutonium, and fission products from irradiated reactor fuel. The selection of the optimum contactor for continuous countercurrent solvent extraction is, therefore, a prime factor to be considered in planning and designing chemical recovery facilities for processing irradiated reactor fuel. Three types of contactors, namely, packed columns, pulse columns, and mixer-settler mechanisms, have received considerable attention at several atomic energy project sites in recent years. A review of the relative merits of these contactors concurrently with the pilot planting of the Purex Process in all three types of contactors is considered in order, and is presented herewith. Consideration of some of the other contactors which have found satisfactory application in industry is also worthwhile at this time. Hence, the centrifugal contactor is included in this review, because of certain apparent advantages which suggest themselves for project application. This review is not considered definitive because studies now in progress will permit a more complete analysis and recommendation at a later date.

As a basis for analysis, the tabular comparison of packed columns and mixer-settlers presented in HW-11416, Recommended Choice of Contactors for Redox Plants (October, 1948), was reviewed. In this report, Hanford indicated its selection of packed columns over mixer-settlers for the Redox Plant. The new tabulation was prepared on the basis of experience since 1948, and includes the pulse column and the centrifugal contactor.

Packed column contactors have been used extensively at all levels of activity at Oak Ridge National Laboratory, and at natural uranium activity levels ("cold") at the Hanford Works. Large size scale-up factors have been tested in the "cold" development work at the Hanford Works. Laboratory scale packed columns have been used by other investigators throughout the AEC.

Pulse columns in various sizes (1" to 16") have been tested with natural uranium at Hanford. Laboratory scale pulse columns have been employed at several AEC sites. A pilot plant pulse column system employing two and four inch columns is nearly ready for operation at Oak Ridge National Laboratory. This new system parallels an equivalent packed column installation and will be used at all levels of activity.

A complete mixer-settler pilot plant is in operation at Knolls Atomic Power Laboratory at ORNL activity levels. Considerable mixer-settler experience on various types of units including the "box-type" mixer-settler has been gained in industry, particularly by Standard Oil Development Company who originally developed the box-type units for project application. This work was the result of an AEC sponsored survey of contactors by the Standard Oil Development Company.

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In addition, investigations using mixer-settlers as a tool for laboratory scale equilibrium studies at very low activity levels have been made at Argonne National Laboratory. As yet no extended operational and mechanical performance data have been obtained for mixer-settlers operating at production activity levels.

Characteristics of the optimum liquid-liquid extractor for project application include the following:

- (1) long operating life with low maintenance requirements,
- (2) small size permitting a minimum bulk of shielding,
- (3) simplicity of operation and control,
- (4) adaptation to easy replacement,
- (5) flexibility permitting easy conversion to new process conditions.

Summary

Extensive experimental, pilot plant, and size scale-up studies on packed columns have established the reliability of this type contactor for project applications. Pulse columns and mixer-settlers are presently undergoing intensive project development, because they appear to present certain advantages over the packed column contactor. A clear-cut choice of type of contactor best suited to particular project applications is not presently possible. However, the comparisons offered in the attached tabulation with respect to the Purex Process may serve a useful purpose (1) for guidance of further contactor studies or (2) for an immediate or preliminary choice of contactor in the event that this is necessary. Currently planned contactor development will permit a closer analysis and, hence, a sound selection for specific process application at a later date.

TABULAR COMPARISON OF PACKED COLUMN, PULSE COLUMNS, MIXER-SETTLERS,
AND CENTRIFUGAL CONTACTORS

Basis: 1.5 tons U/day; Purex type process; 1A contactor used for
specific comparison

I OPERATION	PACKED COLUMNS	PULSE COLUMNS	MIXER-SETTLERS	CENTRIFUGAL CONTACTORS*
1.0 Start-up and shut down				
1.1 Number of volume changes required to reach equilibrium (99%) in 1A contactor, starting with contactor filled with non-U solutions	Approximately 4 volume changes (4 hours)	(2 hours)	May be less than 4 volume changes (4 hours or less)	Estimate approximately 4 volume changes (~5 minutes)

REMARKS: Pulse column slightly favored over packed columns and mixer-settlers by this comparison.

1.2 Mechanical (including instrumental) adjustments required	One interface controller	One interface controller	Multiple interface controllers	Rotor speed and "back" pressure controller
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REMARKS: Feed adjustments common to all contactors. Column contactors favored in this comparison.

1.3 Relative difficulty of start-up after emergency shut-down of short duration	Established procedures are adequate and incur no increase in waste losses. Equilibrium easily recovered after shut-down of short duration.			Rapid start-up; no waste losses.
1.4 Effect of extended shut-down (particularly radiation damage to solvent)	Relatively small interface exists between phases, thereby minimizing radiation damage	Relatively large interface exists between phases: hence large vol. of solvent exposed to radioactivity. Radiation damage at large interface may be appreciable; hence might not permit prolonged shut down.		Relatively small interface exists between phases, thereby minimizing radiation damage.

REMARKS: Columns favored by this comparison.

* Centrifugal contactors have so far not been tried on AEC processes.
All information tabulated in this report concerning centrifugal contactors is taken from industrial sources.

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TABULAR COMPARISON OF PACKED COLUMN, PULSE COLUMNS, MIXER-SETTLERS,
AND CENTRIFUGAL CONTACTORS

Basis: 1.5 tons U/day; Purex type process; 1A contactor used for specific comparison

II MAINTENANCE	PACKED COLUMNS	PULSE COLUMNS	MIXER-SETTLERS	CENTRIFUGAL CONTACTORS
1.1 Plugging	No tendency toward plugging in more than 3 years operation of ORNL columns on variety of processes. HW level silicas handled o.k.	Probably can handle solids; long term test necessary. (Short test at ORNL indicated no plugging)	Probably can handle solids; test necessary.	Can handle solids; may require intermittent flushing (presumably at very long intervals)

REMARKS: This comparison is presently incomplete, thus does not permit a clear choice based on fact.
(Centrifugal contactor should provide clean raffinate simplifying evaporator operation).

1.2 Moving Parts	None	Pulse generator which can be placed in cold area for direct maint. Piston seal (not necessarily on hot stream).	Multiple agitation; failure may cause emergency conditions, necessitating removal of either individual agitator or eventually an entire bank. Agitator seals	Seals, rotor, bearings
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REMARKS: Packed columns favored over other contactors.
Pulse column favored over M-S in this comparison.

1.3 Electrical Services	None	Simple circuit for pulse generator motor in cold area.	Multiple circuit for agitation mechanisms. Motors in cold area.	Simple circuit for prime mover.
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REMARKS: Columns slightly favored by this comparison, with packed columns in first place.

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TABULAR COMPARISON OF PACKED COLUMN, PULSE COLUMNS, MIXER-SETTLERS,
AND CENTRIFUGAL CONTACTORS

Basis: 1.5 tons U/day; Purex type process; 1A contactor used for specific comparison.

<u>MAINTENANCE</u>	<u>PACKED COLUMNS</u>	<u>PULSE COLUMNS</u>	<u>MIXER-SETTLERS</u>	<u>CENTRIFUGAL CONTACTORS</u>
1.4 Total Replacement	Probable maximum height of 50 ft. makes replacement possible by existing techniques and design.	Probable maximum height of 27 ft. permits easy removal.	Bulk and number of contactors may determine ease of removal. Multiple electrical and instrumental connections.	Replacement of complete unit simple and easy.

REMARKS: Mixer-settler favored by this comparison, primarily on basis of lower vertical structure requirements.

1.5 Ease of Decontamination	Packed column expendable; packing hard to decontaminate.	Column expendable. Perforated plates relatively easy to decontaminate compared with ring packing.	Flushing techniques may not be satisfactory because of (1) tortuous horizontal circuit and (2) agitator seals preventing use of pressure techniques. Non-expendable unit.	Low volume flushing techniques should be adequate. Mechanical agitation under pressure should be feasible. Unit inexpensive.
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REMARKS: Relative frequency of required decontamination may be in the following descending order:

1. Centrifugal Contactor - most frequent
2. Mixer-Settler -
3. Pulse Column
4. Packed Column - least frequent

On the basis of this comparison, contactor preference would be in the following descending order: pulse column, packed column, and mixer-settler.

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 TABULAR COMPARISON OF PACKED COLUMN, PULSE COLUMNS, MIXER-SETTLERS,
 AND CENTRIFUGAL CONTACTORS

Basis: 1.5 tons U/day; Purex type process; 1A contactor used for specific comparison.

III <u>DESIGN CONSIDERATIONS</u>	<u>PACKED COLUMNS</u>	<u>PULSE COLUMNS</u>	<u>MIXER-SETTLERS</u>	<u>CENTRIFUGAL CONTACTORS</u>
1. Lower limit of density difference accommodated	Lower limit is about 0.1 in almost all types of contactors except centrifugal contactor.			Less than 0.05 has been effectively used in industrial applications. This is a decided advantage since volume through-put can probably be reduced.
REMARKS: In the Purex Process, the process chemical conditions for the optimum U throughput involve a density difference of about 0.05 or less.				
2. Size and geometry	16 in. diam. x 50 ft. high column	16 in. diam. x 27 ft. high column	10 in. deep 4 ft. wide 16.5 ft. long (20 stages)	Approximately 6 ft. x 2 ft. x 2 ft. overall dimensions; 2 units in series, one for scrub- bing.
REMARKS: (a) Mixer-settlers (and centrifugal contactors) do not require wells or tall structures, hence are favored by this comparison. (b) Criticality: This tabulation is concerned primarily with the 1A contactor, in which criticality is no problem. However, it should be noted that in the plutonium cycles "ever-safe" contactors are required.				
3. Hold-up volume	500 gal.	280 gal.	425 gal.	3 gal. (1.5 gal each unit)
REMARKS: Small hold-up is a big advantage on extended shut-downs; also for change of process conditions. Pulse column slightly favored over packed columns and mixer-settlers by this comparison.				
4. End Section Details	Satisfactory end stream introduction methods demonstrated..	None required		Dripless rotating seals demonstrated on indus- trial installations. Replacement schedule not known.

REMARKS: No preference by this comparison.

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TABULAR COMPARISON OF PACKED COLUMN, PULSE COLUMN, MIXER-SETTLER,
AND CENTRIFUGAL CONTACTORS

DESIGN CONSIDERATIONS

PULSE COLUMNS

CENTRIFUGAL CONTACTORS

0.5 0.2
(Horizontal structure) (Horizontal structure)

Up to 10,000 GPH
multistage industrial
units available and
in operation.

Centrifugal force (several thousand X G) should break up any emulsification resulting from use of very low density difference.

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DESIGN CONSIDERATIONS

PULSE COLUMNS

CENTRIFUGAL CONTACTORS

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TABULAR COMPARISON OF PACKED COLUMN, PULSE COLUMN, MIXER-SETTLER,
AND CENTRIFUGAL CONTACTOR

IV. AEC INSTALLATIONS

PACKED COLUMNS

PULSE COLUMNS

MIXER-SETTLERS

1. Laboratory	Considerable experience at ORNL, HW, ANL, UCRL	Considerable experience at HW, UCRL, ORNL, ANL	Considerable experience at ANL, KAPL, UCRL
2. Pilot Plant	Considerable experience ("hot" and "cold") at ORNL, ANL; "cold" at HW	Considerable "cold" experience at HW. "Hot" installation to go on stream at ORNL 3/51	Considerable ("hot" and "cold") experience at KAPL. Considerable "cold" experience at SOD.
3. Industrial Scale	Considerable experience in scale-up trials at HW. Large installation to go on stream late 1951 or early 1952		Two-stage large scale unit presently under test at KAPL.
4. Scale-up experience	Adequate; HW is proceeding with industrial scale installations.		Project experience limited. Considerable non-project industrial experience available.

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