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COUNTERCURRENT MUTUAL SEPARATION OF Y^{91} AND Pm^{147} BY SOLVENT EXTRACTION USING PULSE COLUMN

Feb. 1962

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ABSTRACT

The use of continuous solvent extraction has been proved quite satisfactory in separating Y^{91} from Pm^{147} , using synthetic mixtures of the nuclide pair on a tracer level. The separation was conducted in a compound perforated plate pulse column using a solvent containing 10% HDEHP, di(2 ethyl hexyl) phosphoric acid, in toluene. Flow rates to the extraction column were maintained around 1 ml/min of feed, 3 ml/min scrub and 12 ml/min solvent giving one volume change per hour. Pulse frequency was maintained at 50 cycles/min and pulse amplitude at 1 cm.

Varying Pm:Y ratio (by activity) in the feed from 0.08 to 135, and using the above conditions, Pm in excess of 99% and Y in excess of 97% purity were obtained. Steady state was reached in 12 hours, or 12 volume changes. The approach to steady state was found to be the same for any one component in all runs conducted. However, this approach was much slower than that predicted by calculations reported in the literature for discrete stages.

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INTRODUCTION

The mutual separation of fission product lanthanides and yttrium has, in general, been accomplished by ion exchange techniques in which advantage is taken of the ease with which the equivalent of multi-stage operation may be accomplished. However, in those instances in which the separation factor for a given pair of elements is sufficiently large and the respective extraction distribution ratios of suitable magnitude, it may be argued that a liquid-liquid extraction technique is preferable. Suggested advantages of the liquid-liquid extraction technique are: the rapid attainment of true equilibrium within a given stage, the ease of operating in truly countercurrent fashion, the assurance that a stage will operate chemically in a reproducible manner due to the homogeneity of the extractant, and the theoretically limitless decontamination factors to be realized.

Since a liquid-liquid extraction system involving di(2-ethyl hexyl) phosphoric acid as extractant in toluene as carrier diluent as the solvent phase and an aqueous mineral acid solution as the opposing phase has been shown to preferentially partition Pm^{147} and Y^{91} (5), a study of the application of a pulsed column technique to the mutual separation of Pm^{147} and Y^{91} was undertaken.

In the system chosen, a toluene solution of di(2-ethyl hexyl) phosphoric acid vs. an aqueous nitric acid solution, Y^{91} is extracted preferentially with respect to Pm^{147} into the solvent phase. The distribution ratio, K , for each of these tracer-level solutes is directly third-power dependent upon the concentration of

extractant in the solvent phase and inversely third-power dependent upon the concentration of hydrogen ion in the aqueous phase.⁽⁵⁾

Consequently, the respective K values may be varied over a wide range. (It should be noted that the separation factor, β , defined as $K_Y: K_{Pm}$, remains unchanged throughout this range of variation.)

Since the solutes, Y^{91} and Pm^{147} , were present in trace concentrations never exceeding 1×10^{-8} M, the K values were constant throughout a given column. This follows from the fact that the solvent and aqueous phases were in mutual equilibrium before contacting within the column, and the transfer of Y^{91} and/or Pm^{147} from one phase to another produced no composition changes large enough to affect macro chemical behavior. The interpretation of data is greatly simplified because of this constancy of K values, especially in the approach to steady state region.

EXTRACTANT

The extractant used in the present work is di(2-ethyl hexyl) phosphoric acid (HDEHP). Previous work by Peppard et al.⁽⁵⁾, indicated that conditions are quite favorable for separating Y^{91} from Pm^{147} when these nuclides are distributed preferentially between 0.2M nitric acid and a 10% solution of HDEHP in toluene. Since one of the aims of this investigation, was the demonstration of the feasibility of the Y^{91} - Pm^{147} separation on a commercial scale, examination of the suitability of the technical grade extractant was undertaken.

The principal contaminant in technical grade HDEHP is the mono(2-ethyl hexyl) phosphoric acid, H_2MEHP . The effect of this contaminant in decreasing the effectiveness of the mutual separation

was studied by determining distribution ratios of Y^{91} and Pm^{147} between 0.2M nitric acid and synthetic mixtures of HDEHP and H_2MEHP dissolved in toluene. The concentration of HDEHP was kept the same in all the experiments (10%, v/v).

It is clear from Table 1 and Figure 1, that the K values of both Y^{91} and Pm^{147} increase with the increase of the amount of added H_2MEHP . However the slope of the Pm^{147} line in Figure 1 is higher than that for Y, indicating that this increase in K values is accompanied by a definite decrease in β values.

Pre-equilibration of 0.2M nitric acid with the organic phase was found to increase both distribution ratios. However, the increase is more pronounced with Pm^{147} with a net decrease in the β values. It is also clear from Table 2, that technical grade HDEHP gives K values quite high compared to the pure extractant, with a β value half as large.

To investigate whether it would be possible to by-pass a purification process for the technical grade extractant, an attempt was made to try to nullify the effect of H_2MEHP by adding increasing amounts of tri(2-ethyl hexyl) phosphate, (TEHP)⁽⁴⁾. It was found however, as seen from Table 2, that up to about 10% TEHP was needed in order to obtain distribution ratios and a separation factor comparable to those obtained using pure HDEHP.

Resorting to a purification procedure, the H_2MEHP was removed by scrubbing a 20% solution of technical grade extractant in toluene with ethylene glycol⁽⁷⁾ at a volume ratio of 3:1. This was repeated, until after the third scrub quality of the extractant was almost the same as that of the pure material. Furthermore, difficulties en-

TABLE 1
EFFECT OF ADDITION OF H_2MEHP TO $HDEHP^*$

% $HDEHP^{**}$	% H_2MEHP^{**}	K_Y	K_{P_m}	β
10	0	21.5	0.0258	832
10	1	23.9	0.039	613
10	2.38	28	0.0645	434
10	4.87	36.6	0.132	277

*Toluene diluent, Aqueous phase: $0.2M$ HNO_3 . In the procedure of determination of distribution ratios, neither of the phases was pre-equilibrated with the other.

**Percent by volume in toluene solution.

countered later in running the pulse columns using such solvent necessitated that the three ethylene glycol scrubs be followed by three hydrochloric acid scrubs and three nitric acid scrubs to remove traces of iron and silica. The procedure adopted and followed throughout the work, then, was as follows:

(1) Contacting a 20% solution of technical grade HDEHP in toluene for 30 minutes with one-third of its volume of ethylene glycol. This was followed by a settling period of 15-20 minutes and the cycle repeated to a total of three scrubs.

(2) The scrubbed solvent was contacted with 5M HCl at a ratio of 1 HCl: 10 solvent, for 30 minutes followed by a 15-20 minute settling period. This was done three times.

(3) At the same ratio of 1:10 the solvent was given 3 contacts with nitric acid (1M) for 15 minutes. After each contact, the acid was diluted to 0.2M and stirring continued for another 15 minutes. The phases were then allowed to separate for 15 minutes. After the last scrub, the settling period was extended to several hours to ensure complete phase separation.

(4) The concentration of HDEHP was then determined by titration with a 0.1M NaOH. The solvent was diluted to 10% HDEHP immediately before use in the extraction.

ANALYTICAL PROCEDURES

1. Individual nuclides

For the determination of distribution ratios for each nuclide, the radiometric method reported by Peppard et al.⁽⁷⁾ was followed.

TABLE 2
EFFECT OF ADDITION OF TRI(2-ETHYL HEXYL) PHOSPHATE, TEHP,
AND OF ETHYLENE GLYCOL SCRUBBING*

Percentage (v/v) of extractant and additives in diluent (technical toluene)	K_Y	K_{P_m}	β
Pure HDEHP (10%)	26.9	0.044	613
Technical HDEHP (10%)	444	1.36	326
Pure HDEHP (10%) + H ₂ MEHP (5%)	132	1.27	104
Pure HDEHP (10%) + H ₂ MEHP (5%) + TEHP (5%)	33.7	0.113	298
Technical HDEHP (10%) + TEHP (5%)	19.7	0.06	328
Technical HDEHP (10%) + TEHP (10%)	17.4	0.04	435
Technical HDEHP purified by scrubbing 3 times with Ethylene Glycol (10%)	31.2	0.0412	757

*All distribution shown in this table were determined using a 0.2M nitric phase previously pre-equilibrated with an equal volume of the organic phase.

2. Yttrium⁹¹ in Promethium¹⁴⁷

Absorption data determined for both elements at various levels of activity are shown in Table 3. From these data it was concluded that counting beta activity through a 96.43 mg./cm² aluminum absorber will cut down all counts due to Pm¹⁴⁷ by a factor of approximately 10⁴ relative the counting rate without absorber. Through the same absorber the counting rate of Y⁹¹ is 47% of that in the absence of the absorber. This method has proved satisfactory for the determination of Y⁹¹ in Pm¹⁴⁷, Table 4.

For cases in which Y⁹¹ activity in AP₁ was quite low, large aliquots, up to 2 ml, were evaporated using platinum dishes 4 cm in diameter and 4 mm deep. This method was used only for Y⁹¹ determination. The fraction of Y⁹¹ activity passing through 96.43 mg./cm² absorber under these geometric conditions was determined, using several known levels of Y⁹¹ activity and found to be 0.41.

3. Promethium¹⁴⁷ in Yttrium⁹¹

(A) The previous method is not sufficiently accurate for use in this case. Further successive extractions of Y⁹¹ from the mixture are necessary before final determination can be made. To accomplish this, three successive contacts with a 10% solution of HDEHP in toluene was found appropriate in case where the Y-Pm mixture is present in 0.2M nitric acid. The aqueous layer after the third contact will contain a ratio of Pm¹⁴⁷ to Y⁹¹ high enough to enable the use of the method outlined in Section 2 of Analytical Procedures. However, in cases where the residual activity is too low for direct counting, both components may be

TABLE 3
RELATIVE EFFICIENCY OF COUNTING RATES OF SPECIFIC Y^{91} AND Pm^{147}
SAMPLES THROUGH ALUMINUM ABSORBERS

Absorber mg/cm ²	c/m with absorber: c/m without absorber for specific counting rates without absorber					
	Y^{91}			Pm^{147}		
	5×10^2	1×10^4	8×10^5	2.5×10^3	3×10^4	1.5×10^5
19.1	0.883	0.88	----	3.5×10^{-2}	3.5×10^{-2}	----
40.0	0.757	0.764	----	----	8×10^{-4}	----
60.6	0.64	0.623	----	----	----	----
96.4	0.465	0.465	0.47	----	----	1.4×10^{-4}

TABLE 4
ANALYSIS OF SYNTHETIC MIXTURES OF $Y^{91}-Pm^{147}$
BY ABSORPTION TECHNIQUE

(Y/Pm) ratio, by activity		% error
Actual	Determined	
9.99×10^{-4}	9.95×10^{-4}	0.4
4×10^{-3}	3.98×10^{-3}	0.5
2×10^{-2}	1.95×10^{-2}	2.5
10.5	9.88	6.2
94.3	86.6	7.7

TABLE 5
CONCENTRATION OF ACTIVITY USING PYROPHOSPHATE

Aqueous activity level 150 cpm/ml

% "pyrophosphate" in toluene	Org./Aq. ratio	Activity level of org. cpm/50λ	Percent Recovery
0.1	1:10	615	82
0.1	1:5	363	97
0.5	1:5	374	~ 100

extracted quantitatively by contacting the aqueous mixture with a 0.5% octyl pyrophosphate solution in toluene at a ratio of 1:5 by volume, thus concentrating the activity by a factor of 5. The optimum ratio and concentration of pyrophosphate were arrived at by varying both in a series of experiments for extracting synthetic mixtures of known activities, and as seen from Table 4 the chosen conditions give the best combination of activity level and recovery.

(B) As mentioned later, the stripping solution used to recover Y^{91} product from the solvent following the separation, is 2M nitric acid. The method outlined in (A), therefore, could not be used to analyze for Pm^{147} in Y^{91} at this acid concentration. It was concluded from the work by Peppard et al.^(1,6) that a 0.15F solution of octyl-H-phenyl phosphonate HEH[ϕ P] in cyclohexane would preferentially extract Y^{91} out of a mixture of Y^{91} and Pm^{147} in 2M nitric acid. Under these conditions, distribution ratio for Y^{91} and Pm^{147} were found to be 17.2 and 0.0102, respectively, insuring reasonably good mutual separation.

The procedure followed here, was to contact 30 ml. of the 2M nitric acid solution with 10 ml. of 0.15F HEH[ϕ P] solution in cyclohexane in a 60 ml. separatory funnel for 3 minutes followed by a 15-minute settling period. The organic layer was then discarded and the aqueous layer contacted with a fresh 10 ml. portion of the HEH[ϕ P] solution. The procedure was repeated to give a total of 5 extractions. The resulting aqueous layer now contains greater than 96% of the Pm^{147} and less than 0.01% of the Y^{91} .

In addition to removal of Y^{91} , since the Y^{91} product in the continuous separation usually contains only very small amounts of Pm^{147} , some method of concentration of Pm^{147} by a factor of at least 10 was needed. As mentioned, earlier, the pyrophosphate concentration method gave best results at a ratio of 1:5. Hence, it was felt that concentration by evaporation would be more adequate. Following the Y-removal procedure, accurately measured volume of the aqueous phase were slowly evaporated to dryness, 5 ml. at a time in a 10 ml. beaker under a heat lamp. This was continued until 25 ml. had been evaporated. The sides of the beaker were then carefully rinsed with 2 ml. of 6M HCl and the sample evaporated to dryness again. The dry material was dissolved in 2 ml. of 6M HCl. This solution in which the original activity has been concentrated by a factor of 12.5, was then assayed for its Pm^{147} and Y^{91} content by plating and counting with and without 96.43 mg/cm² aluminum absorber.

EQUIPMENT

The laboratory scale unit used in this investigation consists of a perforated plate column, a packed column, and accompanying pumps, tanks, samplers, controllers and stainless steel piping and tubing. The columns, along with remote pumping heads, samplers and all piping, tubing and tanks which in the process of running the unit contain radioactive material, are housed in a properly shielded and ventilated hood. The other tanks, pumps, and timers are located in the open at the side of the hood. A flow diagram of the unit is shown in Figure 2.

Columns:

Column-1 is composed of a four-foot extraction section and a two-foot scrub section. These sections are Pyrex glass pipes $3/4$ inch in diameter, connected by a Pyrex glass tee which serves as the feed entry point. The assembly contain 48 perforated plates, each one mounted rigidly on a common $1/8$ inch diameter support rod at $1\ 1/2$ -inch intervals by means of $1/4$ -inch diameter tubular spacers. The plate is 0.005 inch thick, $3/4$ inch in diameter and contains seventy two 0.032 inch diameter circular perforations the total free space is 13% of the total plate area. At the top and the bottom, the column is fitted with two disengaging sections. Each section consists of a section of Pyrex glass pipe and tee, with a 1-inch diameter and an overall length of 18 inches. The column holds 960 ml. total volume of liquid.

Column-2 is a packed column consisting of a 6 foot section of $3/4$ inch inside diameter Pyrex pipe packed with glass Raschig rings 0.24 inch x 0.24 inch. Attached to the top and bottom of this packed section are the glass pipe disengaging sections, which are identical with those of Column-1. The column holds 925 ml of liquid.

Flow of liquids:

(a) Aqueous feed to Column-1, (AF_1)

The feed to Column-1 being a radio-active solution is pumped in a shielded and ventilated hood by means of a Lapp pulsafeeder pump with remote head. With this arrangement, the pulsafeeder power unit can work in an open area. The remote head, kept inside the hood housing the columns, is connected to the pump by means of a

hydraulic line. The pulsafeeder pump employed is a combination piston-diaphragm pump employing positive displacement. The pump is supplied with a device for the adjustment of the stroke length. The remote head and check valve assembly are constructed of 18-8 stainless steel with a diaphragm made of Kel-F (trifluorochloroethylene polymer). The overall diameter of the remote head is 6 inches.

(b) Organic extractant to Column-1, OX_1

Aqueous scrub to Column-1, AS_1

Aqueous extractant to Column-2, AX_2

Pumping and metering of those streams is effected by the use of Milton Roy reciprocating, positive displacement pumps.

A step-valve arrangement with double ball checks is installed at the head of the pump. All pump parts that come into contact with the liquid being pumped are made of 18-8 stainless steel. Teflon (tetrafluoroethylene polymer) packing is used.

(c) Aqueous product from Column-1, AP_1

Organic product from Column-1, OP_1

These streams, being active, metered and pumped remotely with arrangements similar to that used for AF_1 . The OP_1 phase flows by gravity from the top of Column-1 through an intermediate reservoir, the level in the reservoir being controlled by the respective rates at which OP_1 is withdrawn from Column-1 and introduced as feed, OF_2 , to Column-2. The volume of the liquid held in the reservoir and the lines between the two columns is about 125 ml. under average conditions.

(d) Aqueous product from Column-2, AP_2 .

The flow of this stream is controlled by the use of the jackleg described below.

(e) Organic waste from Column-2, OW_2

The stream flows by gravity from the top of the column. Its rate of discharge is controlled by the level of the interface and the rate of flow of OF_2 .

Pulsing mechanisms:

The pulsing of the liquid in both columns is done by means of Lapp Pulsafeeder pumping units employing reagent heads remotely located from the power units and connected to the columns by hydraulic lines. The pumps are the same as that used for AF_1 , except that the remote head does not utilize a check valve assembly.

In Column-1, the action of the pulsing unit causes the countercurrently flowing streams to reciprocate through the plates' perforations, thereby effecting the break-up of the dispersed phase into minute droplets. In Column-2, the liquid is pulsed similarly through the packed section.

The pulsing units are fitted with an adjustment for varying the stroke (or pulse) amplitude and with Graham Variable Speed Power Transmission Units as variable pulse frequency controls. Both adjustments are performed manually.

The pulse amplitude for each column is measured by means of a centimeter scale affixed to a sight glass. The frequency is determined by means of a timer based on the revolutions of a shaft connected to the Graham Variable Speed Power Transmission Unit.

Interface control:

The interface control in Column-1 is accomplished by fixing the rate at which the heavy effluent stream, AP_1 , is metered by a Lapp Pulsafeeder pump.

In Column-2, the interface is controlled by the use of a jackleg which can be raised and lowered manually. The jackleg is constructed partly of 3/8 inch stainless steel tubing and partly of 3/8 inch coiled polyethylene tubing. The jackleg is comprised of the heavy phase effluent line connected to the bottom of and rising parallel to the column and a pipe tee (vent and spillover point) at the top. In operation, the coiled polyethylene tubing provides the flexibility for raising and lowering the spillover point. The principle of operation involves the balancing of the total column head against the total head of the jackleg. In this manner the interface can be controlled at will.

Flow rate measurement:

Measurement of flow rates is determined as an average rate rather than an instantaneous rate. Two methods are employed:

(1) Burette and stop watch, for AX_1 , AS_1 and AX_2 . This method involves filling a burette to the desired volume, momentarily turning the tank valve off, and feeding from the burette while timing the measured volume pumped. The burette is located on the suction side of the pump.

(2) Calibrated bulb and timer arrangement for AF_1 , AP_1 and AP_2 . In this method the operation is completely automatic except for the initial starting of the flow measurement by means of a contact switch. The instrument consists of three parts:

- a) A bulb of calibrated volume with an electrode at each end.
- b) An electric circuit actuated through the two electrodes and consisting of relays, pilot lights, a switch and a reset type timing device.

c) A solenoid valve.

The flow rate is determined by the time taken for the liquid level to pass from one electrode to the other in the calibrated bulb. A circuit diagram for the feed flow rate arrangement is shown in Figure 3.

Sampling:

The sampling assembly consist of an enclosure (a steel box) containing hypodermic needles, glass vials each fitted with Buna-N-type rubber diaphragm top, externally mounted syringes, valves, a vent and liquid carrying lines. This arrangement is used for sampling AP_1 and AP_2 by applying suction through the syringe. For OP_1 , the syringe is not needed, since the liquid flows by gravity from the high level intermediate reservoir. Sampling of OW_2 is done through a drainage valve fitted on the downstream line.

PRELIMINARY RUNS

Two series of experiments were conducted using the pulse columns in order to determine the effect of operating conditions on the stability and efficiency of the extraction and stripping operations. Flow rates of feed, extractant, scrub and stripping solution as well as pulse frequency and amplitude were varied. In these experiments Y^{91} tracer alone was used. The results showed that the purpose of this study using the available columns would be best served by operating in the emulsion region, using the following conditions:

Aqueous as continuous phase.

1 ml/min feed (tracers in 0.2M nitric acid)

12 ml/min extractant (10% HDEHP in toluene).

3 ml/min scrub (0.2M nitric acid).

12 ml/min stripping solution (2M nitric acid).

50 cpm frequency and 1 cm amplitude for extraction column.

70 cpm frequency and 1.3 cm amplitude for stripping column.

Procedure for a typical run:

The columns are filled with the aqueous phase while the column is pulsing. The pulse amplitude and frequency are adjusted to the required values. The organic extractant OX_1 , scrub AS_1 and stripping solution AX_2 , are then introduced, their flow rates being measured and adjusted. Meantime, the pump drawing the aqueous product AP_1 from column-2 is adjusted so that the flow rate of AP_1 approximates as closely as possible, the combined flow rates of AF_1 and AS_1 . The flow of feed is then begun and the time of initial introduction recorded as the beginning of the run. The interface at the top disengaging section of column-1 is adjusted to a given level. As soon as the organic product, OP_1 , starts flowing in the reservoir between the two columns, the liquid in this reservoir is maintained at a fixed level which is controlled by adjusting the pump drawing OP_1 from Column-1 and feeding it as OF_2 to Column-2. In turn, the interface at the top of the Column-2 is adjusted manually by lowering or raising the jackleg.

At equal intervals the flow rates of all streams are measured. Samples are drawn from all effluent streams at preset intervals.

At the end of the run all incoming streams are shut off, the columns emptied, and Column-1 and Column-2 filled with AS_1 and AX_2

respectively. To prepare the columns for a subsequent run, they are emptied and then refilled and left to pulse for approximately an hour. The procedure is repeated until samples drawn show that the residual activity is well within the tolerable limit. This limit depends upon the level of activity to be encountered in the subsequent run. Usually, the total residual activity in the product streams is brought down to about 10-50 cpm/ml by following this procedure.

RESULTS AND DISCUSSION

Operating Conditions:

Four series of runs with a total number of 9 experiments were conducted varying only the Pm/Y ratio in the feed from 0.08 up to 135, keeping the rate of flow of influent streams rates as close as possible to those outlined on page 17. Total activity in the feed was always kept at 10^5 - 10^6 cpm/ml. The average flow rates of influent streams are given in Table 5, along with percentage variation of the rate of flow of each stream from the reported average. The table shows that the highest percentage variation for AF_1 , AS_1 and AX_2 in any of the runs is $\pm 4.1\%$ and that for OX_1 is $\pm 7.2\%$. The organic to aqueous ratios in the scrub and extraction sections of Column-1 are given in Table 6. The ratio in the scrub section $(O/A)_m$ is given by:

$$(O/A)_m = \frac{AS_1}{AX_1} \text{ and the ratio in the extraction section by:}$$

$$(O/A)_n = \frac{AS_1 + AF_1}{AX_1} .$$

Average flow rates over the whole duration of each run were used in the calculation of pertinent ratios.

TABLE 5
FLOW RATES OF INFLUENT STREAMS

Run	(Pm/Y) feed	AF ₁		AS ₁		OX ₁		AX ₂	
		ml/min*	Percent** variation	ml/min	Percent variation	ml/min	Percent variation	ml/min	Percent variation
31	0.0825	1.077	0.4	2.87	1.0	11.6	7.2	24.4	2.1
32	0.0837	1.075	0.5	2.95	2.7	11.1	3.2	24.6	2.1
33	0.093	1.093	0.1	3.05	3.1	12.5	4.8	26.0	4.1
41	0.96	1.031	0.4	2.90	1.7	12.3	1.5	13	3.1
42	1.18	1.079	0.2	3.05	3.0	12.1	3.2	12.8	1.7
51	112	1.108	4	2.90	4.1	11.7	4.7	11.9	2.3
52	135	1.108	3.2	3.02	3.0	12.2	6.9	12.7	1.5
61	0.167	1.131	3.2	3.03	4.0	12.1	3.5	12.0	1.5
62	0.171	1.072	3.2	2.78	4.8	11.9	6.7	11.7	3.1

*Average of all rate measurements made during the run.

**Highest absolute percentage variation from the average flow.

TABLE 6
FLOW RATIOS IN COLUMN-1

Run	(O/A) _m	(O/A) _n
31	2.94	4.04
32	2.75	3.78
33	3.03	4.11
41	3.23	4.25
42	2.92	3.96
51	2.92	4.04
52	2.95	4.04
61	2.90	3.98
62	3.10	4.30

Material Balance:

The last set of flow rate readings was taken as the basis for an overall material balance around the whole unit. In the material balance input items are $AF_1 + AX_1 + AS_1 + AX_2$, while output items are $AP_1 + AP_2 + OW_2$. Table 7 indicates that except for run-31 material balance checks fall within $\pm 2\%$ range.

Purity of Products:

Steady state values of Pm^{147} and Y^{91} activities in effluent streams, AP_1 and AP_2 , were taken to represent composition of end products. It is worth mentioning that in all the runs the activity in the organic waste from Column-2, OW_2 , was negligible, even after reducing the aqueous to organic flow ratio in this column from about 2 in series 3 to about 1 in the rest of the series. Hence, it is reasonable to assume that the composition of AP_2 also represents the composition of organic product from Column-1 OP_1 with a delay corresponding to the residence time in Column-2.

It is clear from data presented in Table 8 that the solvent extraction system was able to produce very high purity of both Pm^{147} and Y^{91} products from a wide variety of feed compositions. Compositions in this table are calculated on solvent free basis. From these results, it is concluded that Pm^{147} product with a purity $> 99.9\%$ could be obtained from a feed mixture containing 20% Pm^{147} or higher. With feed mixtures of lower Pm^{147} content, the product would be 99-99.8% pure. As for the Y^{91} product, it is seen that purities $> 99\%$ are to be expected when the Y^{91} represents 50% or higher of the activity in the feed mixtures. Even when the feed mixture contains $< 50\%$, the Y^{91} product would still be of a

TABLE 7
OVERALL MATERIAL BALANCE

Run	Input ml/min	Output ml/min	Absolute Percent* difference
31	39.9	41.1	3
32	39.8	39.8	0
33	42.7	42.3	0.9
41	29.9	29.4	1.6
42	29.4	29.2	0.7
51	27.4	27.3	0.4
52	29.0	28.4	1.7
61	28.0	28.1	0.4
62	28.2	28.1	0.4

$$* \text{Absolute Percent difference} = \left| \frac{\text{Input} - \text{Output}}{\text{Input}} \right| 100$$

purity ranging between 97-99%. Recovery of each component in its product was always better than 99%.

(Y/Pm) ratio:

As shown in Figure 4, the (Y/Pm) ratio in the Pm^{147} product AP_1 , at steady state varies linearly with a slope of 1 with (Pm/Y) ratio in the feed. The deviation observed in the points corresponding to experiments in series-5 is evidently due to the difficulty in analyzing for Y^{91} in this region. Such a region requires analyzing for a few parts of Y^{91} per million parts of Pm^{147} which is well beyond the limits of the method used in this investigation. However, Table 9 and Figure 4 demonstrate satisfactorily that in most of the runs, Column-1 gave a decontamination factor of approximately 2000.

Number of Stages and HETS in Column-1:

The method used in determining the number of theoretical stages in the column was based on the work presented by Klinkenberg^(2,3). In this method the rejection ratios R_1 and R_2 for Pm^{147} and Y^{91} respectively are as follows:

$$R_1 = \frac{(\chi_A - \chi_F)\chi_0}{(\chi_F - \chi_0)\chi_A}$$

$$R_2 = \frac{(\chi_A - \chi_F)(1 - \chi_0)}{(\chi_F - \chi_0)(1 - \chi_A)}$$

χ being the fraction of the component remaining in the aqueous phase (in the present case Pm^{147}) on solvent free basis. It is immaterial whether the fractions are expressed in weight, mol, volume fractions. For the purpose of this work the fraction of

TABLE 8
PRODUCTS COMPOSITIONS

Run	Feed (x_1)*	Promethium product (x_1)	(x_2)**	Yttrium product (x_2)	(x_1)
31	0.076	0.9 ₂ 25	7.5×10^{-3}	0.9 ₃ 5	5×10^{-4}
32	0.077	0.9 ₂ 35	6.5×10^{-3}	--	--
33	0.085	0.9 ₂ 36	6.4×10^{-3}	0.9 ₃ 3	7×10^{-4}
61	0.144	0.9 ₂ 71	2.9×10^{-3}	0.9 ₂ 9	1×10^{-3}
62	0.146	0.9 ₂ 7	3×10^{-3}	0.9 ₂ 87	1.3×10^{-3}
41	0.488	0.9 ₃ 63	3.7×10^{-4}	--	--
42	0.547	0.9 ₃ 75	2.5×10^{-4}	0.998	1.2×10^{-3}
51	0.991	0.9 ₄ 4	6×10^{-5}	0.978	0.022
52	0.993	0.9 ₄ 65	3.5×10^{-5}	0.975	0.025

* (x_1) Fraction of Pm^{147} , solvent free basis

** (x_2) Fraction of Y^{91} , solvent free basis

TABLE 9

VARIATION (Y/Pm) RATIO IN Pm PRODUCT WITH FEED COMPOSITION

Run	$(\text{Pm}/\text{Y})_F$	$(\text{Pm}/\text{Y})_P$	D.F.
31	0.083	133	1600
32	0.084	154	1840
33	0.093	156	1680
61	0.167	345	1950
62	0.171	334	2060
41	0.96	2700	2820
42	1.18	4030	3420
51	112	1.64×10^4	147
52	135	2.86×10^4	212

each component was based upon its beta activity. So that if a mixture of Pm^{147} and Y^{91} gives 70 cpm due to Pm and 30 cpm due to Y, χ would be 0.70.

A reduced extraction factor χ introduced by Klinkenberg was defined as:

$$\chi = \frac{\log E_2}{\log \beta} \quad \text{so that } 0 < \chi < 1.$$

Values of R_1 , R_2 and χ corresponding to the experiments conducted in this investigation are given in Table 10.

Klinkenberg's method calls for the calculation of n , the number of stages in the extraction section including feed stage and m stages in the scrub section including feed stage, for six cases.

Case 1:

When $\chi = 0$ and $\frac{n}{m} = R_2$

m is obtained by trial and error in the following equation:

$$R_1 = \frac{\beta^{-m}(\beta^{-R_2 m} - 1)}{\beta^{-m} - 1}$$

in our particular case $R_2 \gg 1$, hence $\beta^{-R_2 m} \ll 1$ which simplifies the equation to

$$R_1 = \frac{1}{\beta^m - 1}$$

Case 2:

When $2m = n$

$$\beta^m = \frac{-0.5 + \sqrt{0.25 + R_2}}{-0.5 + \sqrt{0.25 + R_1}} \quad \text{which again could be}$$

simplified to

$$m = \log \frac{\sqrt{R_2 - 0.5}}{\sqrt{R_1 + 0.25 - 0.5}} \bigg/ \log \beta$$

TABLE 10
REJECTION RATIOS AND REDUCED EXTRACTION FACTORS

Run	R_1	R_2	X
31	6.1×10^{-3}	1610	0.709
33	7.6×10^{-3}	1685	0.715
41	9.3×10^{-4}	2760	0.708
51	2.03×10^{-4}	164	0.711
52	1.87×10^{-4}	235	0.712
61	6.6×10^{-3}	2000	0.708
62	8.23×10^{-3}	1840	0.72

For this case

$$X = \frac{\log \sqrt{R_2 - 0.5}}{m \log \beta}$$

Case 3:

When $X = 0.5$

$$m = \frac{2 \log (R_2) (1 + R_1)}{(\frac{R_2}{R_1}) (1 + R_2) \log \beta}$$

$$\text{and } n = \frac{2 \log \frac{1 + R_2}{1 + R_1}}{\log \beta}$$

Those equations could be rewritten for

$R_1 \ll 1$ and $R_2 \gg 1$, as

$$m = \frac{2 \log (1/R_1)}{\log \beta} \quad \text{and } n = \frac{2 \log R_2}{\log \beta}$$

Case 4:

Here $n = m$, thus $n = \frac{\log (R_2/R_1)}{\log \beta}$

in which case

$$X = (\log R_2)/(\log R_2 - \log R_1)$$

Case 5:

If $m = 2n$, β^n and X are given by:

$$\beta^n = \frac{0.5 R_2 + \sqrt{0.25 R_2^2 + R_2}}{0.5 R_1 + \sqrt{0.25 R_1^2 + R_1}}$$

and,

$$X = \frac{\log (0.5 R_2 + \sqrt{0.25 R_2^2 + R_2})}{n \log \beta}$$

Case 6:

When X becomes equal to 1, then $n = mR_1$

and $R_2 = \frac{\beta^m (\beta^{R_1 m} - 1)}{\beta^m - 1}$, which for $\beta \gg 1$ becomes

$$R_2 = \beta^{R_1 m} \quad \text{or} \quad m = \frac{\log R_2}{R_1 \log \beta}$$

From the data presented in Table 8, n , m , $n + m - 1$ and X values were calculated for the above six cases as shown in Tables 11 and 12. The reduced extraction factors obtained in each experiment and shown also in Table 10 were used to determine the number of theoretical stages from plots such as that shown in Fig. 5. The number of stages and the average height equivalent to a theoretical (HETS) are given in Table 13. The HETS were based on the total height of the plate sections in the column and the total number of plates obtained as described above.

Optimum Conditions

Optimum feed location: Minimum number of stages and optimum feed location as determined from plots as in Figure 5, are shown in Table 14, along with the optimum value for (m/n) as calculated from Scheibel's equation (8):

$$\frac{m}{n} = \sqrt{\frac{\log (1/R_1)}{\log R_2}} \left(1 + \frac{\log R_2 R_1}{(1/R_1) \log R_2} \right)$$

As shown in Table 15, the optimum feed location obtained using the results of the experiments showed that the calculated feed stage location (the feed point taken at the center of the

TABLE 11
NUMBER OF STAGES FOR KLINKENBERG'S CASES

Case		1	2	3	4	5	6
Item	Run						
n	31	1300	2.78	2.34	1.98	1.57	1.17
	33	1310	2.68	2.36	1.95	1.59	1.18
	42	3040	3.6	2.5	2.36	1.80	1.25
	51	220	3.5	1.62	2.16	1.49	0.8
	52	320	3.62	1.74	2.23	1.55	0.87
	61	1600	2.8	2.41	2.00	1.60	1.2
	62	1420	2.72	2.39	1.96	1.57	1.19
m	31	0.81	1.39	1.62	1.98	3.14	192
	33	0.78	1.34	1.55	1.95	3.18	155
	42	1.10	1.8	2.21	2.36	3.60	1350
	51	1.35	1.75	2.70	2.16	2.98	4000
	52	1.36	1.81	2.73	2.23	3.10	4630
	61	0.8	1.4	1.59	2.00	3.20	182
	62	0.77	1.36	1.52	1.96	3.04	145

TABLE 12
REDUCED EXTRACTION FACTORS FOR KLINKENBERG'S CASES

Run	Reduced Extraction Factors		
	Case 2	Case 4	Case 5
31	0.422	0.593	0.746
33	0.437	0.602	0.740
42	0.348	0.532	0.697
51	0.228	0.374	0.533
52	0.239	0.388	0.560
61	0.430	0.602	0.753
62	0.440	0.610	0.785

TABLE 13
NUMBER OF THEORETICAL STAGES AND HETS

Run	n	m	n+m-1	HETS*(ft)
31	1.56	2.80	3.36	1.78
33	1.64	2.87	3.51	1.71
42	1.78	3.7	4.48	1.34
51	1.12	4.58	4.70	1.28
52	1.20	4.50	4.70	1.28
61	1.70	2.74	3.44	1.74
62	1.70	2.59	3.29	1.82

*HETS is the height equivalent to a theoretical stage = $\left(\frac{H}{n+m-1}\right)$

H is the height of the plate section of the column, 6 ft.

feed stage) agrees with the actual feed point in the column to within 20%.

Optimum flow ratios: Using reduced extraction factors read off plots as in Figure 5 corresponding to the minimum number of total stages, an average optimum flow ratio was calculated from equation

$$X = \frac{\log E_2}{\log \beta} \quad \text{which, when using average flow ratio}$$

$(O/A)_{av.}$ gives

$$\log (O/A)_{av.} = X \log (K_2/K_1) - \log K_2.$$

Table 16 shows that on the average the ratio used was approximately 3 times the optimum.

Steady State:

Steady state characterized by constant composition of products, has been realized in 12 hours for Pm in AP_1 and 11.8 hours for Y in AP_1 , corresponding to 12 volume changes of Column-1. A delay of about 1.5 hours for Y in AP_2 has been noticed. This delay is primarily due to the fact that AP_2 is drawn off Column-2, and it corresponds to the residence time in a volume equivalent to the volume in Column-2 and the reservoir and line in between the two columns.

Approach to Steady State:

In all the runs conducted, the pulse frequency and amplitude were kept constant. The flow ratios were also kept constant to within $\pm 10\%$. The only variable involved was the (Pm/Y) ratio in the feed. Hence, it is expected that either component would follow the same approach to steady state regardless of the (Pm/Y) ratio in the feed.

TABLE 14
COMPARISON BETWEEN FEED LOCATION IN EXPERIMENTAL
AND OPTIMUM CONDITIONS

Run	(m/n) Exptal.	(m/n) opt.		Ratio of (m/n) Exptal. : (m/n) opt.
		From Curves*	Calculated**	
31	1.8	0.819	0.818	2.19
33	1.82	0.803	0.810	2.26
42	2.08	0.936	0.938	2.22
61	1.61	0.806	0.814	1.99
62	1.53	0.822	0.800	1.88

*Taken from curves such as those shown in Fig. 6, m and n are taken as those corresponding to minimum $m+n-1$.

**Calculated using Sheibel's equation. (8)

TABLE 15
DEVIATION OF CALCULATED FEED POINT LOCATION
FROM ACTUAL LOCATION

Run	Experimental Conditions			Optimum Conditions		
	(HETS) ^a	(H _m) ^b	Percent ^c deviation	(HETS)	(H _m)	Percent deviation
31	1.78	4.09	35	2.06	2.59	10
33	1.71	4.39	40	2.09	2.66	11
42	1.34	4.29	38	1.62	2.89	15
51	1.28	5.22	54	1.88	3.51	25
52	1.28	5.12	52	1.78	3.46	24
61	1.74	3.9	32	2.03	2.53	9
62	1.82	3.8	30	2.10	2.60	10

a Average height equivalent to a theoretical stage = $\frac{6}{m+n-1}$.

b Feed point location, H_m, distance from the top in feet,
feed point taken midway in the feed stage.

c Percent deviation of the determined feed point from the actual
point in the column used, based on the total height of plate
section; $(\frac{(H_s) - 2}{6}) 100$.

TABLE 16
COMPARISON BETWEEN OPTIMUM FLOW RATIOS
AND ACTUAL FLOW RATIOS

	X opt. (a)	$[(O/A)_{av}]_{opt.} (b)$	$[(O/A)_{av}]_{actual}$	$\frac{actual}{optimum}$
31	0.535	1.15	3.49	3.04
33	0.56	1.36	3.57	2.63
42	0.515	1.01	3.44	3.41
51	0.43	0.73	3.48	4.8
52	0.435	0.59	3.50	5.9
61	0.55	1.27	3.44	2.71
62	0.56	1.36	3.70	2.72

(a) Reduced extraction factor as determined from plots as in Figure 5 corresponding to minimum number of total stages.

(b) Average flow rate ratio of organic/aqueous, calculated from X opt.

The percentage of the steady state value for each component in the end products is given in Table 17 as a function of time. Figures (6-8) show that within the experimental errors involved all runs regardless of the $(Y/Pm)_F$, follow a single curve for each component. It is also clear from the plots that Y in the Y product AP_2 , arrives at the true steady state value approximately 1.5 hour later than Y in AP_1 , the Pm product. Taking this delay into consideration, points were read off Figure 8 and following subtraction of one hour from the value of the abscissa, they were replotted on the curve representing the approach to steady state of Y in Pm product redrawn from Figure 7. Only the part beyond 5 hours running time was considered. As seen from Figure 9, with this adjustment of the abscissa the two curves could be superimposed. This shows that Y in either of the two products follows the same approach to steady state.

Again, from Figure 15, it is seen that the Y curve shows less departure from steady state δ_t , than the Pm curve. Consider this departure as given by⁽⁹⁾:

$$\delta_t = (1 - \frac{P_t}{P_s}) , \quad \text{where}$$

$$P_s = \frac{E^m + n - E^n}{E^m + n - 1} , \quad \text{and}$$

$$(P_t)_Y = f(p^n, n, t)$$

$$(P_t)_{Pm} = f(q^m, m, t)$$

In the last equations $p = \frac{E}{E+1}$, $q = \frac{1}{E+1}$, (n) number of stages in extraction section including the feed in case of Y and

TABLE 17
APPROACH TO STEADY STATE

Pm in Pm product (AP_1)			Y in Pm product (AP_1)			Y in Y product (AP_2)		
Run	time, hrs.	%	Run	time, hrs.	%	Run	time, hrs.	%
62	3.0	33	52	3.0	60.0	51	1.0	24.0
52	3.25	40.5	52	4.0	72.6	41	2.7	57.8
33	3.5	68.3	33	4.5	84.7			
42	4.0	72.8	42		81.0	42	3.8	73.5
51		72.3	33	5.2	90.6	41	4.0	74.3
33	4.25	80.7	42		88			
42	5.0	87.3	31	6.0	92.6	42	5.0	84.2
33	5.2	86.5	42		91.7	41	5.4	87.0
42	6.0	92.5	52		94.0	42	6.0	87.0
31	6.25	92.8	33	6.2	92.9	41	6.1	88.3
41	7.0	93.4	31	7.0	96.0	33	6.2	88.2
33	7.5	96.4	32		96.2	51	6.3	91.7
41	8.0	96.1	42	7.1	95.5	42	7.0	94.1
42		96.1	31	8.0	98.2	62		92.8
51		97.8	52		98.1	41	7.2	92.3
52		95.9	62	9.0	96.8	33	7.25	93.0
42	8.7	96.9	62	10.0	99	42	8.0	95.0
41	9.0	96.2	62	11.0	97.8	41	8.1	96.8
41	10.0	98.1	62	11.8	100	52	8.2	96.5
52		98.3	61	12.0	100	41	9.2	98.0
61	11	98.3				41	9.9	97.0
51	11.5	99.6				51	10.0	98.7
61	12	100				62	11.2	98
62		100				62	11.8	98.5

(m) number of stages in the scrub section including the feed case of Pm, and t number of cycles which for the purpose of this comparison will be the same for both components. From Table 13, an average value of n is seen to be approximately 1.7 and $m \approx 2.75$. The extraction factor, E, will be taken as 90 for Y and 0.16 for Pm.

It is then clear that $(P_s)_Y > (P_s)_{Pm}$ and $p^n > q^m$. Therefore from the nature of the functions above⁽⁹⁾, it follows that $(P_t)_Y > (P_t)_{Pm}$ and $(\delta_t)_Y < (\delta_t)_{Pm}$ which explains why at a certain time Y is closer to steady state than Pm.

Conclusions:

1. Pm^{147} was successfully separated from Y^{91} by liquid-liquid extraction, using the system 10% HDEHP in toluene versus 0.2M nitric acid. Such a method gave product purities better than 99% for Pm and 97% for Y with recoveries greater than 99%.

2. An average decontamination factor of 2000 for Pm from Y was obtained in most of the runs conducted. It was lower than 2000 only in cases where the Y in the Pm product was too low (in the order of 5 p.p.m.) to be accurately determined by the analytical procedure used.

3. Total number of theoretical stages in the extraction column were determined by Klinkenberg's method⁽³⁾ to have an average value of 3.5 corresponding to a HETS of 1.71 ft.

4. The organic aqueous flow ratio used was found to be 3-4 times higher than the optimum ratio that corresponds to minimum number of stages required for the separations accomplished.

5. The calculated feed points corresponding to optimum conditions agreed with the actual feed point in the column to within

10-15% of the total column height. However, the feed points corresponding to the actual operating conditions show that the feed moves down in the extraction section of the column, thus decreasing the effective height of this section by up to 40% of the total column height.

6. Steady state was attained in 12 hours, from the time the feed was introduced in the column, corresponding to 12 column throughputs under average operating conditions.

7. As expected, the approach to steady state proceeded at a much slower rate than predicted for a system of discrete stages operated under the same conditions.

8. The success with which Pm-Y were separated would encourage trying similar systems for separating nuclide pairs of lower β values such as Pm-Eu.⁽⁷⁾

9. This study proved tracer-level work to be an appropriate method for investigating pulse column design variables and operating characteristics.

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SYMBOLS AND NOMENCLATURE

HDEHP	Dl(2-ethyl hexyl) phosphoric acid.
H ₂ MEHP	Mono(2-ethyl hexyl) phosphoric acid.
TEHP	Tri(2-ethyl hexyl) phosphate..
HEH(ϕ P)	Octyl-H-phenyl phosphonate.
AF	aqueous feed, Pm ¹⁴⁷ and Y ⁹¹ in 0.2M nitric acid.
AS	aqueous scrub, 0.2M nitric acid.
AX	aqueous extractant, 2M nitric acid.
OX	organic extractant, 10% HDEHP in toluene.
AP ₁	aqueous Pm product.
AP ₂	aqueous Y product.
OP	organic Y product.
OW	organic waste.
E	extraction factor (KO/A).
HETS	height equivalent to a theoretical stage.
K	distribution ratio, concentration of nuclide in the organic phase/its concentration in the aqueous phase.
n	number of theoretical stages in the extraction section including the feed stage.
m	number of theoretical stages in the scrub section including the feed stage.
m+n-1	total number of stages
O/A	organic: aqueous throughput ratio
χ	fraction of the component by activity, on solvent free basis.

X reduced extraction factor = $\log E_2 / \log \beta$

β ratio of distribution ratios K_2/K_1 .

χ_F, χ_A, χ_O fraction of Pm by activity in feed, Pm product and Y product respectively, on solvent free basis.

subscripts 1 and 2 for influent and effluent streams refer to Column-1 and Column-2 respectively for E, K and R refer to Pm and Y respectively.

Figure 1. EFFECT OF ADDITION OF H₂MEHP TO HDEHP ON DISTRIBUTION RATIOS OF Y AND Pm.

Figure 2. SOLVENT EXTRACTION UNIT.

RH Remote Pumping Head
R Rate Bulb

RPH Remote Pulsing Head
S Sampler

FIGURE 2

1. One inch pyrex glass Tee, disengaging section.
2. 3/4 inch to 1 inch pyrex disengaging section.
3. 3/4 inch by 48 inch pyrex section.
4. 3/4 inch pyrex Tee.
5. 3/4 inch by 24 inch pyrex section.
6. 3/4 inch by 72 inch pyrex section.
7. Sample boxes for AP_1 , OP_1 , AP_2 .
8. Rate bulbs for automatic rate timing for AF_1 , AP_1 , AP_2 .
9. Rate bulb timers and controllers, placed outside enclosure.
10. Remote pulsing heads.
11. Remote pumping heads.
12. Graham Transmissions coupled with Lapp Pulsafeeders.
13. Lapp Pulsafeeder pumps.
14. Milton Roy pumps.
15. Sight glass, pulse amplitude measurement.
16. Jack Leg for interface control, Column-2.
17. Spring check valves, stainless steel.
18. Fifteen gallon stainless steel drum.
- 18-A Fifteen gallon S. S. drums inside S. S. secondary and shielded with 3/4 inch lead.
19. Solution feed tanks.
20. Burettes for manual rate timing.
21. Intermediate reservoir for OP_1 .

Figure 3. AQUEOUS FEED BULB CIRCUIT DIAGRAM.

- A Manual switch, when closed supplies power to circuit which closes relays R_2 and R_3 .
- S Contact switch, when pushed closes relay R_1 .
- V Solenoid bulb, normally open, closes only when R_1 is closed.
- B Rate bulb, normally full, starts emptying when V is closed.
- e_3 Electrode attached to R_3 when liquid level drops below it, the circuit is broken causing the relay to open and start timer.
- e_2 Electrode attached to R_2 , when liquid finally drops below this electrode R_2 opens and the timer stops, at the same time R_1 and the solenoid valve open causing rate bulb to refill by gravity from the feed tank.

Figure 4. RELATIONSHIP BETWEEN (Pm/Y) RATIO IN FEED (AF_1) AND (Y/Pm) RATIO IN Pm PRODUCT (AP_1).

Figure 5. DETERMINATION OF NUMBER OF THEORETICAL STAGES USING KLINKENBERG'S METHOD FOR RUN (61).

Figure 6. APPROACH TO STEADY STATE FOR Pm IN Pm PRODUCT (AP_1).

Figure 7. APPROACH TO STEADY STATE FOR Y IN Pm PRODUCT (AP_1).

Figure 8. APPROACH TO STEADY STATE FOR Y IN Y PRODUCT (AP_2).

Figure 9. COMPARISON BETWEEN THE APPROACH TO STEADY STATE OF Y AND Pm.

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