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PURIFICATION OF DEGRADED TRIBUTYL
PHOSPHATE-HYDROCARBON DILUENT
SOLUTIONS BY DISTILLATION:
STATUS SUMMARY

W. Davis, Jr.

OAK RIDGE NATIONAL LABORATORY

operated by

UNION CARBIDE CORPORATION

for the

U. S. ATOMIC ENERGY COMMISSION

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CHEMICAL TECHNOLOGY DIVISION

Chemical Development Section B

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DILUENT SOLUTIONS BY DISTILLATION: STATUS SUMMARY

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ABSTRACT

Molecular distillation and rapid, nonrefluxing volatilization at ~0.5 mm Hg pressure successfully purified TBP—Amsco 125-82 solutions that had been extensively degraded by irradiation in a Co⁶⁰ gamma source or by use in fuel processing in the ORNL Pilot Plant. Distillation at ~50 mm Hg with reflux produced large amounts of tar in the still pot. Infrared analyses of the products of both molecular and rapid distillation showed that the degradation products which are important in subsequent performance of recovered solvent-diluent solutions have higher boiling points than TBP and are primarily nitro compounds.

A continuous flash distillation unit was designed to purify degraded TBP—Amsco 125-82 solutions at a rate of 150 liters/hr with a TBP loss ≤1% of that charged. Preliminary estimates indicate a capital cost of \$202,400 for such a distillation unit erected in an existing building. This cost includes a moderate amount of shielding for the parts handling the bottoms product. The throughput of the column was set at 200 liters/hr to ensure that an average of 150 liters of solvent per hour can be handled over an extended time. The estimated operating cost is \$0.02 per liter (\$0.078 per gallon), exclusive of taxes, insurance, amortization, and general overhead, and \$0.03-0.04 per liter (\$0.11-0.15 per gallon) including these fixed costs.

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1.0 INTRODUCTION

Experimental and design studies to evaluate the feasibility of purifying degraded tributyl phosphate—hydrocarbon diluent solutions by distillation are summarized. Such purification is necessary because both radiation (1) and nitric acid (2) decompose TBP to products, including dibutyl and monobutyl phosphates, that have detrimental effects on TBP solvent extraction processes for recovering uranium, thorium, and plutonium from nuclear fuels (3,4) or from ores (5,6).

Degraded TBP-hydrocarbon diluent solutions are now purified by chemical processes based primarily on washing with sodium carbonate or sodium hydroxide solutions which may or may not contain potassium permanganate, and on washing with nitric acid solutions (7,8,9). In some cases filters are also used. However, it is uncertain whether such chemical purification of the organic extractant solution will be adequate in the processing of very-high-burnup fuels. Even if chemical purification is adequate, it is highly probable that solvents from a first and second cycle of solvent extraction of highly irradiated short-decayed nuclear fuels would require separate purification systems, as shown from an analysis of Thorex runs on short-decayed fuel (7).

The phases of the study of distillational purification summarized here are: (a) a brief study of molecular distillation of irradiated TBP—Amsco 125-82 at ORNL; (b) a detailed study of low-pressure distillational purification and subsequent extraction-decontamination testing of irradiated TBP—Amsco 125-82 at Georgia Institute of Technology (10); and (c) a design study of a flash distillation plant for purifying degraded TBP—hydrocarbon diluent at a rate of 150 liters/hr, made by Weinrich and Associates (11).

2.0 EXPERIMENTAL STUDIES

2.1 Molecular Distillation of TBP—Amsco 125-82

In each of two experiments, Amsco 125-82 and TBP were nearly quantitatively recovered from partially decomposed solutions by molecular distillation. All colored decomposition products and all products that increase extraction of zirconium or retention of zirconium in the $\text{Zr}^{95}\text{-Nb}^{95}$ extraction-scrubbing test remained in the less volatile, but uncharred, pot residue.

The first test was made with 50 ml of TBP—Amsco 125-82 solution irradiated to 122 whr/liter in a nominal 10,000-curie Co^{60} source under conditions* close to those described by Irish and Reas (12) for the Purex process. This was contacted with several equal volumes of 2.5% Na_2CO_3 solution to remove uranium, nitric acid, and acidic phosphate decomposition products. Molecular distillation, in a falling-film still, yielded colorless fractions of Amsco at $\sim 35^\circ\text{C}$ and of TBP at $\leq 70^\circ\text{C}$. The pot residue, 0.5 ml ($\sim 1\%$ of the charge), was orange-red, and infrared analyses showed large quantities of nitro compounds present.

In the second test the still charge was TBP—Amsco 125-82 solution that had been removed** from the ORNL Pilot Plant at a time when the amount of radioactivity in the solvent was unusually high. It had decayed about 8 months and had been centrifuged to remove solids. Four distillate fractions were taken (Table 1): the diluent at $\sim 35^\circ\text{C}$ and three TBP fractions at $\sim 70^\circ\text{C}$. Each was used to prepare 1 M solutions of TBP in Amsco 125-82 by adding either new carbonate-washed TBP to the distilled Amsco or new Amsco to the distilled TBP. These solutions were then (a) contacted with equal volumes of 2 M HNO_3 containing $\text{Zr}^{95}\text{-Nb}^{95}$ tracer, counted, scrubbed three times with equal volumes of 3 M HNO_3 , and then recounted or (b) contacted with solid calcium hydroxide, then with the $\text{Zr}^{95}\text{-Nb}^{95}$ tracer solution, and finally counted. The first TBP fraction, in fresh Amsco, was superior to the control TBP-Amsco solution, while the other two TBP fractions and the Amsco fraction were very similar to the control with respect to extraction and scrubbing of zirconium.

*

These conditions consisted in irradiating, while agitating, a mixture of 270 ml of 30 vol % TBP in Amsco 125-82 with 100 ml of 1.84 M HNO_3 containing 310 g of uranium per liter. Radiation power densities were 5.1 watts per liter of composite aqueous-organic solution. After irradiation, the organic phase was separated from the aqueous and scrubbed eight times with 0.01 M HNO_3 . The first four scrubs were made with an aqueous/organic volume ratio of 0.32, the last four with a ratio of 0.80. The resulting organic solution had an average uranium concentration of 3.29 g/liter, acidity < 0.01 M, 32.1 vol % TBP, 2.10 g of H_2O per liter, and a density at 25°C of 0.8227 g/ml. Approximately 10 liters of this solution was used in the laboratory distillation studies at ORNL and at Georgia Institute of Technology.

**

Drained on April 13, 1959, from a line between tank T-4 and pump T-4-P, i.e., before the solvent entered the solvent recovery equipment.

Table 1. Zirconium Extraction-Scrubbing Tests with Molecular Still Fractions of Degraded Pilot Plant Solvent

Fraction	% of Diluent or TBP Represented by the Fraction	Zr ⁹⁵ -Nb ⁹⁵ Activity ^a in 1 M TBP—Amsco Solution, c ml ⁻¹ sec ⁻¹		
		After		After Ca(OH) ₂ Treatment and Extraction
		Extraction	Scrub	
Diluent	100	232	13	200
TBP-1	40	104	5	91
TBP-2	31	234	8	207
TBP-3	15	224	6	192
Control (fresh TBP— fresh Amsco)	-	165	13	208

^a Contacted with 2 M HNO₃ solution containing tracer Zr⁹⁵-Nb⁹⁵, 2 x 10⁴
c ml⁻¹ sec⁻¹.

2.2 Low-pressure Distillation of Irradiated TBP—Amsco 125-82 Solutions

Studies of low-pressure distillation of TBP—Amsco 125-82 solutions made at Georgia Institute of Technology have been reported by Sicilio, Goodgame, and Wilkins (10). Their results with 122-whr/liter irradiated material (see footnote on preceding page) showed almost quantitative recovery of Amsco 125-82 and TBP in a rapid volatilization at ~0.5 mm Hg pressure with no reflux. The TBP and Amsco so recovered showed extraction, scrubbing, and stripping properties similar to those of similarly distilled as-received materials with respect to ruthenium and uranium tracers. Recovery was almost quantitative, not only from sodium carbonate-treated solutions but also from the untreated, acidic, organic solution. These evaporations involved removal of Amsco 125-82 in the temperature range 35 to 110°C and of TBP in the temperature range 110 to 120°C. The pot residues, corresponding to a few per cent of the still charges, had an apparent boiling point of ~170°C at ~0.5 mm pressure. This indication that the decomposition products have lower volatilities than TBP is consistent with results obtained in molecular distillations (Sect. 2.1).

Distillation with reflux would be a very dubious method for purifying degraded TBP-hydrocarbon diluent solutions even after only small amounts of decomposition by radiation or thermal reactions with nitric acid. In a refluxing still at 50 mm Hg pressure, distillation of irradiated TBP—Amsco 125-82 containing only a few grams of uranyl nitrate per liter and $< 0.01 \text{ M HNO}_3$ produced extensive decomposition (tarring) in the pot. Even after the TBP-Amsco solutions had been washed with aqueous sodium carbonate solution to remove uranium and acid, precipitates and decomposition products were formed in the still pot.

Tests. The solutions were tested as follows: After the flash, or rapid volatilization, separation of Amsco and TBP from decomposition products, each component was separately distilled in an ~40 plate refluxing still at 20 to 50 mm Hg pressure. From the Amsco distillations, 21 to 24 separate fractions were taken and used as a diluent for "as received" TBP; from the TBP distillations 7 to 10 fractions were taken and then diluted with "as received" Amsco 125-82. Each of these was then used in either extraction-scrub or extraction-strip studies to determine how effectively extracted ruthenium or uranium could be removed by scrubbing with 4 M HNO_3 or by stripping with 0.01 M HNO_3 . Aqueous solutions used in the extraction step contained tracer quantities of Ru^{106} - Rh^{106} and U^{233} . The aqueous solutions used in extraction-scrub studies also contained 4.86 M HNO_3 and those used in extraction-strip studies contained 4.47 M HNO_3 .

3.0 DESIGN AND OPERATION OF CONTINUOUS VACUUM STILL FOR SOLVENT PURIFICATION

(Prepared by Weinrich and Associates)

Batch distillation of TBP-naphtha solvent mixtures involves a number of problems. Probably the most important is the tendency of any ester, such as TBP, to decompose on long heating. In addition, the wide boiling point difference between the TBP and the naphtha presents certain problems in control of the distillation. A relatively simple continuous distillation unit was therefore designed to eliminate these problems with special attention given to simplifying the control problem, particularly at startup and shutdown. An advantage of continuous distillation is that the naphtha helps to vaporize the TBP. Because of the wide boiling point difference, the naphtha is a superheated vapor at the

TBP boiling point and therefore acts like any inert gas, such as steam, in a distillation tower. The salient design features for an operation with a net throughput of ~150 liters/hr are described here along with operating philosophy. Because no continuous operating information is available, considerable flexibility was incorporated in the design.

The basic problem in this design is that probably < 1% of the TBP charge has been converted to degradation products. This 1% includes any high-boiling products resulting from degradation of the naphtha. With a 30% TBP solution, the bottoms product ultimately desired is less than about 0.3% of the feed. To achieve this goal it is nearly mandatory to operate the still with bottoms recycle.

3.1 Design

The flowsheet is shown in Fig. 1, and the equipment is listed in Table 2. Solvent mixture from a storage tank is pumped through a continuous coil of pipe, which is heated with steam under controlled pressure. Sufficient surface is provided to vaporize TBP substantially completely in the coil, thereby minimizing residence time. From the coil the TBP-naphtha vapors flash into the vacuum still. Above the feed point is a short de-entraining section, and above this a trap tray removes the TBP containing some naphtha. The amount of naphtha in this stream will depend on temperature, but probably should not be more than 10%. After de-entrainment, the vapors pass up through the chimney of the trap tray. The upper section of the column acts as a rectifier to separate liquid TBP, containing a small amount of naphtha, and naphtha vapors which pass overhead to a total condenser and an accumulator. The partial condenser condenses the TBP, which is withdrawn from the trap tray.

If the column is operated at 50 mm Hg absolute pressure at the top, the trap tray and feed point pressures might be 55 mm and 60 mm, respectively. With the flow rates selected, the TBP partial pressure at the feed point is only about 0.2 of the total pressure, or 11 mm at the trap tray. Temperature at the trap tray should be about 150°C but may be slightly lower due to dissolved naphtha. At the top of the column only naphtha vapor will prevail, and at 50 mm its condensing temperature is only ~90°C. Pure naphtha vapors condense and pass to the overhead accumulator.

Fig. 1. Flash distillation system for TBP-Amsco solvent (prepared for ORNL by Weinrich and Associates, Dec. 15, 1960).

The partial condenser at the top of the column produces the reflux necessary to fractionate naphtha from TBP and to condense the latter, which is removed from the trap tray. It will be necessary to control the heat removal at this point since, if too much heat is removed, the trap tray temperature will drop below the normal condensing temperature of TBP and cause excessive reflux below the trap tray. However, some reflux must be present to prevent the de-entraining section from becoming dry, and the desired gradient from feed point to trap tray can be easily determined by operation.

Table 2. Equipment List for a 150-liter/hr Flash Distillation Unit

Distillation column	18 in. i.d. x 25 ft		
Tanks and vessels	<u>gal</u>		
Charge tank	1,050		
Overhead TBP surge tank	50		
Overhead Amsco surge tank	100		
TBP storage	300		
Amsco storage	600		
Bottoms storage	50		
Pumps	<u>gpm</u>	<u>gph</u>	<u>Type</u>
Feed		52.8	Positive displacement
Bottoms recirculation		5	Positive displacement
TBP to storage	5		Centrifugal
Naphtha to storage	5		Centrifugal
Naphtha recycle		5	Positive displacement
Exchangers	<u>sq ft</u>		
Feed heater	20		
Recycle vaporizer	2		
Condenser	20		

Designing the column as above described provides recycled pure naphtha as the stripping vapor for the bottom and makes easier the control of a column with overhead withdrawal of components boiling as far apart as TBP and naphtha. Taking the heavy component, TBP, as a sidestream just above the feed tray gives an overhead temperature which is that of the saturated vapor of the lower boiling constituent, so that the column is stable and is not sensitive to small changes in the feed rate and the reflux ratio.

Undoubtedly a number of other types of de-entrainers would work satisfactorily, and when a unit of this type is finally designed, alternative methods should be analyzed in detail. Packed towers are used in this type of service, and the design presented here is certainly a simple, straightforward solution to the problem.

At the feed point, the mole fraction of TBP in the vapor is still 0.2, or ~12 mm pressure, which corresponds to a condensing temperature of about 154°C. Effluent from the coil would be set about 10°C higher in order to vaporize the maximum TBP from nonvolatile residue, i.e., in the range 160-165°C. Such temperatures can easily be obtained with steam pressure of 125 psig.

As pointed out above, handling of the bottoms stream requires flexibility in the design, as there is no way to predict the best way to handle this stream without some operating data. In order to prevent the bottom of the column from going dry and forming solids which would eventually plug it, the flowsheet shows the bottoms stream being continuously recycled to the feed heater. At the start of the run the feed temperature can be low; but as the content of degradation products builds up, this temperature can be raised to the design point.

It is not known how far the bottoms can be stripped before handling difficulties are encountered. It would be desirable to reduce the loss of TBP to the point where the bottoms can be discarded without further consideration. However, if this is not possible, the bottoms can be stored for any given period and then be rerun in naphtha solution. For example, a 50% solution of bottoms could be made in naphtha and rerun with naphtha stripping, which should produce a final bottoms product containing only a few percent of TBP and naphtha. During this operation no fresh TBP-naphtha would be charged.

The flowsheet shows a condition where the bottoms withdrawal rate is 3 liters/hr, which corresponds to 5% of the TBP feed rate. Under these conditions, it would certainly be necessary to store the initial bottoms for rerunning to a final bottoms. By concentrating the degradation products, it should be possible to reduce the TBP losses to a small fraction of 1%.

3.2 Capital and Operating Costs

Based on the design described above and on the list of equipment included in the plant, the estimated capital cost of the 150 liter/hr unit is \$202,400 (Table 3), assuming that the distillation equipment is erected in an existing building.

Table 3. Estimate of Equipment Costs

Basis: installation of process equipment in an existing building; supporting frames and minor concrete pads to rest on building floor; all necessary services and utilities now existing at convenient locations for connections to same; all equipment type 316 stainless steel

1. Column and tanks (commercial quality)	\$ 27,000
2. Pumps, exchangers, and instruments (commercial quality)	19,500
3. Supports, piping, insulation, wiring, and misc.	16,000
4. Minor shielding on feed system, plus lead bricks around bottoms storage and lower section of column	10,000
5. For nuclear quality fabrication, special tests, inspections, etc., 100% x item 1 plus 50% x item 2	36,750
6. Installation labor at 25% x items 1,2,3,4 = 25% x \$72,500	18,125
7. Subtotal	\$ 127,375
8. Construction overhead, profit and field expense at 20% x items 1, 2,3,4, plus 100% x item 6	32,625
9. Subtotal	\$ 160,000
10. Engineering at 15% x item 9	24,000
11. Contingency at 10% x items 9 and 10	18,400
	<hr/>
	\$ 202,400

The two major operating costs are operating labor and maintenance, the former of which is estimated at half the time of one operator. At \$2.50 per hour, the salaries for one operator per shift would be \$60.00. With overhead, this would come to approximately \$100.00 per day. Based on operation for 5 days per week for 50 weeks per year and an assumed maintenance of 6% per year of the capital investment, the daily maintenance charge would be $202,400 \times 0.06/250 = \48.60 . Adding to this the cost of half an operator per shift brings the total to \$98.60. Utility costs (Table 4) are insignificant and can be neglected. Thus, the total cost is $98.60/200 \times 24 = \$0.0205$ per liter of solvent handled or 7.8¢ per gallon. Amortization, taxes, and general overhead would bring the total to nearly 15¢ per gallon. Costs of this magnitude are not uncommon in the handling of fine chemicals.

Table 4. Utilities

Steam (200 psig)	
For process heating	120 lb/hr
For vacuum jet	100
Total	<hr/> 220 lb/hr
Cooling water	25 gpm
Electricity	3 kw

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