LITERATURE SEARCH
TBP-KEROSENE SOLVENT DEGRADATION

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February 11, 1958

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NATIONAL LEAD COMPANY OF OHIO
Cincinnati, Ohio
Contract No. AT(30-1)-1156
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ABSTRACT

A search of the classified and unclassified literature concerning the degradation of a tributyl phosphate (TBP)-kerosene solvent has been conducted. Chemical Abstracts, Nuclear Science Abstracts, Abstracts of Classified Reports, and the card file of the National Lead Company of Ohio's Library were included in the search. Pertinent information is presented, in abstract form, as it applies to: (1) Extractant (TBP) Degradation; (2) Diluent (Kerosene) Degradation; and (3) Solvent Clean-up Techniques.
The problem of tributyl phosphate (TBP)-kerosene solvent degradation is of major significance in the efficient recovery of uranium by liquid-liquid extraction. Initial investigation of the problem quickly reveals its extremely complex nature.

As an aid to the investigation and solution of the solvent degradation problem in the National Lead Company of Ohio Refinery, a review of the classified and unclassified literature was undertaken. Chemical Abstracts, Nuclear Science Abstracts, Abstracts of Classified Reports (each from its inception to December, 1957), and the National Lead Company of Ohio's Library card file were included in this search. The subject headings included are listed in the Appendix.

This search revealed a large but repetitive volume of literature concerning solvent degradation. The abstracted material presented is a representative, but by no means complete, summary of the pertinent information concerning: (1) Extractant (TBP) Degradation; (2) Diluent (Kerosene) Degradation; and (3) Solvent Clean-up Techniques.
EXTRACTANT (TBP) DEGRADATION

AERE-CE/R-968 (UNCLASSIFIED)


The separation of monobutyl phosphate (MBP) from dibutyl phosphate (DBP) is accomplished by a carbon tetrachloride extraction procedure. A slight alkaline hydrolysis (0.5%) of tributyl phosphate (TBP) takes place at 100°C, but does not go beyond the DBP stage. When treated with 5N HNO₃ and H₂SO₄, the TBP is stable but a non-acidic phosphorus-containing impurity breaks down by hydrolysis. At 100°C, complete hydrolysis of the TBP is brought about by treatment with acids. Under these conditions the hydrolysis rate is of the order of HCl>H₂SO₄>HNO₃. The non-acidic impurity in TBP is probably tetrabutyl pyrophosphate. This material is removed by steam stripping of the TBP.

AERE-CE/M-19 (CONFIDENTIAL)


To purify commercial TBP treat with K₂MnO₄ and 2 per cent caustic soda until no further decoloration of permanganate occurs. Wash with H₂O, and heat at 50 mm until most of the water evaporates. Distill at 140°C under 3 mm.

Physical Properties

Density: 25°/4° 0.973; 20°/20° 0.973 to 0.983.

Refractive Index (25°): 1.424.

B.P.: 289°C at 760 mm/d
177°C to 178°C at 27 mm
160°C to 162°C at 15 mm

Flashpoint: 295°F (146°C).

Viscosity: 3.885 centipoises at 20°C
3.41 centipoises at 25°C

Stable to HNO₃ (at least up to 8M).
Lane, E. S., Tributyl Phosphate Purification by Distillation Over Alkalies, February, 1952.

Commercial TBP instantly reduces 0.1N neutral KMnO₄. Purification was carried out by washing the acidic impurities out with 2 equivolume washes of sat. NaHCO₃, followed by a water wash and distillation at 10 to 15 mm Hg pressure. It was then fractionated until a product of constant composition was obtained. Distillation was also carried out over solid anhydrous Na₂CO₃. At 140°C, the distillation was stopped and 11 per cent low-boiling impurities were recovered mostly as n-butyl alcohol. Oxidative washings were also done with little success.

Single passage down a 12-inch by ½-inch column of Ca(OH)_2 reduced the acid value of the commercial TBP to zero.

HW-17542 (SECRET)


Monobutyl phosphate (MBP) and dibutyl phosphate (DBP) are separated by an ether extraction. MBP is titrated as a dibasic acid with pK values of 1.9 and 6.6, and reacts mole per mole with neutral uranyl nitrate hexahydrate (UNH) solutions to give UF₂(C₄H₉)PO₄.

DBP is a monobasic acid with a pK of 1.9. It complexes uranium (VI) strongly in the organic phase in the TBP process.

HW-19959 (CONFIDENTIAL)


The hydrolysis of TBP proceeds through TBP → DBP → MBP → H₃PO₄, with TBP → DBP the rate-determining step. Hydrolysis products in the TBP decrease the efficiency of the re-extraction of the uranium from the solvent, and also increase the distribution coefficients of cations which have extraction mechanisms similar to that of uranium.

Pretreatment of TBP with 1M sodium hydroxide, followed by a water wash, removes the hydrolysis products effectively. In the presence of uranium and nitric acid, the rate of hydrolysis of TBP is very slow at room temperature.

Treatment of 15 per cent TBP-85 per cent Amsco-125-90W with 3M nitric acid at 105°C for six days resulted in a high percentage of nitrogen in the solvent. Treatment of the nitrated samples with a NaOH-Na₂CO₃ solution gave a colorless product but did not reduce the uranium distribution coefficient (E₉₀) of a uranyl nitrate hexahydrate (UNH) solution to the value obtained using fresh solvent.

TBP was mixed with nitric acid at various concentrations at room temperature. There was no evident effect. When the mixture was heated to boiling, there was evidence of a reaction.
DILUENT DEGRADATION

ORNL-530 (UNCLASSIFIED)


The presence of mono- and di-butyl phosphate in TBP caused a marked increase in the plutonium and fission product distribution coefficient.

Some component of the diluent has been observed to react with nitric acid, even at room temperatures. Once this component is removed by reaction with strong acid, the remaining components are very stable.

The used mixed solvent is recovered by washing with one-tenth volume of 3M HNO₃, one-tenth volume of 1.5M NaOH, one-tenth volume of 1.0M NaOH, and finally, one-tenth volume of water.

ORNL-1116 (UNCLASSIFIED)


Treatment of kerosene with 6.1M HNO₃ above 100°C gave a yellow color. On cooling, the aqueous phase deposited a white powder which was a mixture of carboxylic acids that had resulted from oxidation of the methyl groups on aromatic rings. Solubility of TBP in water decreases with increasing nitric acid and uranium concentration. TBP is very stable to HNO₃.

ORNL-1367 (SECRET)


Organic addition compounds and decomposition products of the aliphatic-aromatic diluent may be formed in the Thorex process by oxidation, nitration, and/or nitrite addition in the presence of nitric and nitrous acids.

Nitro compounds were found to contribute to the extraction and retention of activities, as well as to crud formation. Preconditioning the diluent with silica gel adsorbs the olefins and aromatics present and is effective in removing the deterioration products resulting from the nitric acid attack. Addition of a small quantity of silica gel to 1.0M NaOH in later solvent treatment appears to form a more stable extractant.
The Amsco diluent must be pretreated with oleum to prevent crud build-up at the interface. This also prevents discoloration of the diluent. Pretreatment forms sulfo-compounds which can be washed from the diluent with water or alkaline solutions.

The TBP should be pretreated with calcium hydroxide to remove any acidic phosphates.

For solvent recycling, recommended clean-up is 0.2M Na₂CO₃ (A/O = 3/4) used twice, followed by deionized water (A/O = 1/4).

Aromatic and olefin content, boiling range, flash point, density, index of refraction, and viscosity measurements for 13 organic diluents are presented. Each property is separately discussed, and equations relating the properties of the pure diluents to mixtures with TBP are presented.

Studies on the stability of TBP-diluent mixtures to nitrous-nitric acid mixtures have shown the TBP to be inert, but a diluent with appreciable aromatic and olefinic content will nitrate rapidly, with evidence of a red color. The uranium extraction behavior of the extractant was only slightly affected by the nitration products. The red impurity was removed readily by treatment with 10 per cent NaOH. These nitration products will not form rapidly under plant conditions.

A flow sheet for the chromatographic separation of substances thought responsible for poor solvent quality is reported.

Evidence for the photochemical hydrolysis of TBP is presented.

The decomposition of solvent at 71°C was reported. The solvents tested were stable to nitric acid, but the addition of nitrous acid caused extreme discoloration, increased the uranium distribution coefficients, increased coalescence times, lowered uranium transfer rate, lowered dispersion time, and increased foaming in uranyl nitrate hexahydrate (UNH) calcination. Use of nitrite inhibitors, such as urea or sulfamic acid in the aqueous feed solution, reduced the detrimental effect of nitrous acid on the solvent.


Chromatographic separation of used plant solvent and infrared analysis gave identification of aliphatic nitro compounds, carboxylic acids, and nitroso compounds. Indications of aromatic nitro and nitroso compounds and ketones and aldehydes were found. Several impurities of TBP are also present. Use of Na₂CO₃ does not efficiently remove the majority of these products.


The effect of nitrite ion, acid, nitrate, uranium concentrations, and temperature on the rate of solvent deterioration is measured, and a rate law is presented. Radiation damage to diluents is also discussed.


Degradation of 30 per cent TBP-Shell Spray Base with 6M HNO₃ -0.1M nitrite is responsible for greatly increased $E_A^O$ values of neptunium. The increased extraction of neptunium is due to degradation of the solvent to form new compounds which preferentially extract neptunium.

The presence of unsaturated hydrocarbons in amounts as low as tenths volume per cent is deleterious to solvent extraction, since these products are not removed by normal carbonate treatment as are the products of oxidation of aromatic hydrocarbons. Samples with low absorption in ultraviolet are more stable.
SOLVENT CLEAN-UP PROCEDURES

AERE-C/M-105 (OFFICIAL USE ONLY)


The effect of solvent pretreatment by a 5 per cent NaOH wash on 20 per cent TBP in kerosene was investigated. It was found that with unwashed solvent, it was difficult to remove the uranium by water-washing. A 5 per cent NaOH wash, followed by a water wash, gave a solvent that did not retain uranium.

AERE-C/M-118 (OFFICIAL USE ONLY)


Pretreatment of solvent with either 5 per cent NaOH or 1M Na₂CO₃ results in identical extraction of plutonium and uranium. It may also be advantageous to follow the alkali and water washes with a 5N HNO₃ wash and a water wash.

CF-53-6-110 (UNCLASSIFIED)


A vertical solid-liquid mixer-settler was used to contact the solvent with a Ca(OH)₂ slurry (330 to 400 g/l). This treatment effected the removal of a major portion of the crud-forming impurities in the solvent.

DP-174 (CONFIDENTIAL)


Continued recycling of the TBP-Ultrasene solvent used in the Purex process alters the extraction properties of the solvent, as is evidenced by increasing zirconium and niobium retention after the re-extraction of uranium. Caustic washing, although removing zirconium, does not remove the affinity for zirconium. The abnormal complexing of zirconium is the earliest manifestation of solvent degradation in the Purex system. Plant and laboratory samples gave evidence of the presence of ketones and esters. Laboratory samples also showed the presence of organic nitrogen compounds, but these did not cause firm complexing of zirconium. Zirconium-complexing agents were formed from olefinic compounds present in the Ultrasene diluent. Treatment of
Ultrasene, prior to use, with chromic acid and washing with hot phosphoric acid stabilized the diluent. Removal of degradation products by silica gel, charcoal, chemical washes, and steam or vacuum distillation was investigated.

Vacuum distillation was the most effective means of removing degradation products, but decomposition of the TBP occurred at the temperatures (180°C) corresponding to higher pressures.

ISC-234 (UNCLASSIFIED)


Zirconium is separated from hafnium by solvent extraction with tributyl phosphate-dibutyl ether solvent. The zirconium is recovered by water stripping. Degradation products of the solvent were removed by washing four times with 1.5M H₂SO₄ at an A/O of 3/1. Sulfuric acid was removed by three water washes at an A/O of 2/1.

KAPL-820 (SECRET)


A solvent recovery procedure has been developed to remove fission-product activity, uranium, plutonium, and solvent degradation products from the Purex solvent. This procedure includes two water washes (1/10 vol), one 0.1M NaOH wash (1/5 vol), one water wash (1/10 vol), and one 0.05M HNO₃ wash (1/10 vol).

KAPL-914 (Del) (SECRET)

Report of the Chemistry and Chemical Engineering Section for February, March, and April, 1953, p. 27, 1953.

A recommended solvent wash procedure for the Purex ICW stream is as follows:

<table>
<thead>
<tr>
<th>Wash</th>
<th>Volume</th>
<th>Concentration</th>
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<tbody>
<tr>
<td>First</td>
<td>1/10 Vol</td>
<td>0.5M Na₂CO₃</td>
</tr>
<tr>
<td>Second</td>
<td>1/10 Vol</td>
<td>0.02M HNO₃</td>
</tr>
<tr>
<td>Third</td>
<td>1/10 Vol</td>
<td>0.1M NaOH</td>
</tr>
<tr>
<td>Fourth</td>
<td>1/10 Vol</td>
<td>0.02M HNO₃</td>
</tr>
</tbody>
</table>

KAPL-959 (Del) (SECRET)

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First 1/10 Vol of 0.5M Na₂CO₃
Second 1/10 Vol of 0.02M HNO₃
Third 1/10 Vol of 0.1M NaOH
Fourth 1/10 Vol of 0.02M HNO₃

KAPL-959 (Del) (SECRET)

Residual uranium was removed from organic solvents by a 1/10 vol water wash, 1/10 vol 0.1M NaOH wash, 1/10 vol water wash, 1/10 vol 0.05M HNO₃ wash. Sodium carbonate has advantages only where DBP is to be removed.

Washing the solvent with sulfuric acid or bromine water removed tracer I¹³¹, which had been held up by an impurity.

HW-18352 (UNCLASSIFIED)


A variety of chemicals were tested on poor solvent in an effort to restore the original quality of the solvent. The ultimate improvement was found to be more a function of the number of washes than of the actual composition of the wash. Na₂SO₄ or H₂SO₄ (5 to 10 per cent) gave the best results.

HW-27298 (SECRET)


Solvent (30 per cent TBP in Shell Deodorized Spray Base) was treated with 3 per cent Na₂CO₃ (O/A = 2/1), followed by 0.1M HNO₃ (O/A = 2/1), and centrifuged. The time required for the removal of the entrained aqueous phase from the solvent phase and the disengagement time were measured.

Three washings of solvent by 5 per cent NaOH precipitated the uranium as the diuranate and reduced the uranium content of the solvent to 8.7 × 10⁻⁵ lb/gal.

A stable aqueous-in-organic emulsion was formed in the RC column as a result of suspended solids. Two per cent HNO₃ was contacted with the solvent to coalesce the dispersion and to settle the solids to the interface, from which they could be removed by decantation. This was followed by a 5 per cent Na₂CO₃ wash to remove the HNO₃ and DBP.

HW-29228 (Excerpt, Del) (SECRET)

Chemical Development Unit Progress Report for August 1953, p. 11, August, 1953.

The rate of uranium transfer is dependent upon solvent history. Reductions of 30 to 35 per cent in the rate of transfer have been noted for Plant ROC as compared with a solvent prepared from fresh chemicals. "Darco scavenging" or an alkaline wash of the solvent essentially restores the original characteristics of the solvent.
Data currently available indicate tentatively that neither the solvent nor the solvent-washing techniques are responsible for the abnormal amounts of sodium in the recent TBP plant-recovered uranium.

HW-31962 (SECRET)


HW-47782 (SECRET)


A sodium carbonate-KMnO₄ wash solution is more effective than a sodium carbonate solution in removing impurities from crude TBP. In a test using Purex solvent, the Na₂CO₃-KMnO₄ solution was 20-fold more effective in removing zirconium contamination, but about equal with sodium carbonate solution in removing ruthenium and iodine contamination.

HW-50584J (SECRET)


About 85 per cent of the solvent inventory was washed with 0.01M KMnO₄-5 per cent Na₂CO₃ solution, followed by a 2 per cent nitric acid wash. The gamma activity of the solvent was reduced from 10,000 to 3600 μC/gal.

ORNL-557 (CONFIDENTIAL)


The presence of butyl alcohol and mono- and di-butyl phosphates had no significant effect on the extraction of uranium, but the back extraction from the TBP was not easily accomplished. Washing the solvent with a 1.0M nitric acid wash, followed by two 1.0M NaOH washes and two H₂O washes, was sufficient to restore the solvent to its initial quality.

ORNL-708 (UNCLASSIFIED)

Chemical Technology Division Progress Report for Month of March 1950, p. 11, June, 1950.
Washing TBP solvent with 0.1M sodium hydroxide decreased the beta distribution coefficient by a factor of 4 to 10 by removing butyl acid phosphates. The chemical stability of the Varsol was markedly increased by sulfonation.

ORNL-1045 (SECRET)


Used solvent is recovered in a batch process. Washing is done with a 0.2 volume wash with 0.1M Na₂CO₃, followed by two 0.1 volume demineralized-water washes. Contact time for the carbonate wash is one hour, and a final settling time of 20 minutes is allowed.

ORNL-1141 (SECRET)

Chemical Technology Division Quarterly Progress Report for Period Ending August 20, 1951, p. 21, March, 1952.

Dibutyl phosphate and monobutyl phosphate (the hydrolysis products of tributyl phosphate) are 99 per cent removed from the solvent by a single one-minute, one per cent Na₂CO₃ wash; 99.8 per cent is removed by a 10-minute wash.

ORNL-1224 (UNCLASSIFIED)


The development of a continuous solvent recovery process is reported. The investigation was carried out in a ½-inch-diameter pulse column having a 9-foot carbonate-contacting section (0.1M Na₂CO₃ being the contacting agent), and a 4-foot water wash section (to remove the entrained sodium carbonate solution).

ORNL-1519 (Del) (SECRET)


Recovery of used solvent is done by means of a pulse column and a centrifuge. The columns are "organic continuous". The aqueous phase is 0.1M Na₂CO₃ flowing at an O/A ratio of 5/1. The washed solvent is passed through a centrifuge for clarification and removal of solids.

Calcium hydroxide slurries of about 360 g/l effectively removed impurities from Thorex-process solvent. Contact between solvent and slurry was made in mixer-settlers. An equipment diagram and a flowsheet are given.
APPENDIX

The following is a list of the subject headings included in this literature search:

ABSTRACTS OF CLASSIFIED REPORTS; NUCLEAR SCIENCE ABSTRACTS

Butex Process

Solvents

Butyl Phosphates

Chemical reactions of, with HNO₃ and Al(NO₃)₃
Diluents for Hydrolysis, rate of
Hydrolysis and radiation stability of Hydrolysis of, in contact with HNO₃ and UO₂(NO₃)₃ Purification, storage, deterioration of Solvent properties and decontamination of used

Butyl Phosphate Complexes

Butyl Phosphate - Hexane Systems

Butyl Phosphate - Kerosene - Nitric Acid Systems

Butyl Phosphate - Kerosene Systems

Butyl Phosphate - Nitric Acid Systems

Butyl Phosphate - Nitric Acid - Thorium Nitrate Systems

Butyl Phosphate - Nitric Acid - Uranyl Nitrate Systems

Hydrocarbons

Chromatographic analysis Diluents Radiation effects

Kerosene

Decontamination of, for Purex Process by Centrifugation
Nitric Acid
Chemical reactions of, with TBP
Corrosive effects
Organic compound systems
Solvent extraction

Nitrous Acid

Organic Compounds
Butyl phosphate
Effects of radiation on

Phosphates
Solvent extraction
Solvent properties
Uranium recovery

Purex Process
Decontamination
Nitration products
Solvent extraction
Solvent processing
Solvent recovery
Solvents
Waste Disposal

Redox Process
Process studies and process chemistry
Solvent treatment

Separation Processes
Solvent partition
Waste disposal
Slurrex Process
Solvent extraction
Solvent processing

Solvent Extraction Processes

Solvents
Recovery

TBP Process
Solvent extraction
Solvent processing
Solvents

Tributyl Phosphate Process (Canadian)

Uranium
Extrusion
Recovery
Solvent extraction
Solvent partition

Uranium Compounds
Uranyl nitrate systems
Uranyl nitrates

Uranyl Nitrates
Solvent extraction
Solvent partition

CHEMICAL ABSTRACTS

Butyl Phosphate

n Butyl Phosphate
Degradation
of kerosene
prevention
products
removal of

Dibutyl Phosphate

Diluent
Composition of
Degradation of
Stability of

Extraction
Solvent

Kerosene
Composition of
Degradation of
Oxidation of
Stability of
Treatment of

Liquid-Liquid Extraction

Monobutyl Phosphate

Nitric Acid
Action on kerosene
Action on tributyl phosphate

Nitrous Acid
Action on kerosene
**Phosphates**
- Butyl
- n-Butyl
- Dibutyl
- Monobutyl
- Tributyl

**Purex Process**

**Slurrex Process**

**Solvent**
- Clean-up
- Rehabilitation
- Stability
- Treatment

**Solvent Extraction**
- Tributyl phosphate
  - Of uranium

**Tributyl Phosphate**
- Degradation of
- Extraction with
- Purification of
- Stability of
  - In uranium extraction

**Uranium**
- Extraction of
- Manufacture of
- Recovery of

**Uranyl Nitrate**
NATIONAL LEAD COMPANY OF OHIO LIBRARY CARD FILE

Amex Process

Butyl Phosphate - Carbon Tetrachloride Systems

Butyl Phosphate Complexes

Butyl Phosphate - Ethyl Ether Systems

Butyl Phosphate - Hexane Systems

Butyl Phosphate - Hexane - Uranyl Nitrate - Water Systems

Butyl Phosphate - Kerosene - Nitric Acid Systems

Butyl Phosphate - Kerosene Systems

Butyl Phosphate - Kerosene - Thorium Nitrate - Water Systems

Butyl Phosphate - Nitric Acid Systems

Butyl Phosphate - Nitric Acid - Thorium Nitrate Systems

Butyl Phosphate - Nitric Acid - Uranyl Nitrate Systems

Butyl Phosphate Systems

Butyl Phosphate - Thorium Nitrate Systems

Butyl Phosphate - Thorium Nitrate - Water Systems

Butyl Phosphate - Water Systems

Butyl Phosphates

Diluents

Kerosene

Purex Process

TBP Process

Tributyl Phosphate Process (Canadian)