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March 2, 1955

CHEMICAL STABILITY OF PUREX AND URANIUM RECOVERY PROCESS SOLVENT

I. INTRODUCTION

The desirability of operating many of the Purex columns at elevated temperatures, ca. 50-70 C, has made it necessary to obtain data on the stability of the solvent at these higher temperatures. Since the present diluent, Shell Spray Base, will not be entirely safe at the upper temperature limit, a number of diluents with higher flash point have been investigated concurrently with Shell Spray Base.

This work has been restricted to the evaluation of chemical effects only. Radiation effects are currently under investigation by others.

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An attempt has been made in the choice of experimental conditions to evaluate separately the effect of temperature and aqueous phase composition on the diluent and on TBP decomposition, rather than to test only Purex flowsheet operating conditions. This approach is reasonable in that the conditions can be varied to bracket most actual flowsheet chemical compositions.

This report presents data covering the first phases of this investigation, namely, the effect of nitric and nitrous acid on the solvent at 71 C. or higher temperatures.

II. SUMMARY AND CONCLUSIONS

(1) Thirty per cent TBP solutions in Shell Spray Base, Soltrol 170, Bayol-D, and Ultrasene have been found to be satisfactorily stable at 71 C in nitric acid concentrations up to 6.0 M providing nitrous acid is excluded.

(2) In the presence of nitrous acid, all of the above solvents, or the diluents alone, are unstable when in contact with aqueous nitric acid at 71 C and react at rates which increase with increasing nitric acid concentration. Soltrol 170 shows greater resistance to attack than does Shell Spray Base.

(3) The effect of nitrous acid in promoting chemical instability can be eliminated by the addition of nitrite inhibitors to aqueous phases to be contacted with an organic phase. Experiments have shown that 0.2 M urea or 0.2 M sulfamic acid completely eliminates effects due to nitrous acid.

(4) Nitrous acid is not a catalyst but enters directly into the reactions. By analysis, nitrite esters, nitroso compounds, and oxidation products are found among the impurities in solvents exposed to combined nitric acid - nitrous acid attack.

(5) The impurities resulting from the chemical decomposition of Shell Spray Base cause increases in uranium distribution coefficients under dilute "C" column conditions, increases in coalescence times, lowering of the uranium transfer rate, lowering of dispersion time, enhanced fission product retention by the solvent, and enhanced foaming during the course of UNH calcination.

(6) The aromatic content of diluents of the Shell Spray Base type seems to have little effect on the solvent as regards its use in a Purex type process. This seems to be due to the fact that the impurities arising from the aromatic constituents are among those readily removed by aqueous carbonate washing.

III. EXPERIMENTAL

In general, the diluent or solvent (diluent and TBP) was contacted with an aqueous phase under conditions of controlled or known temperature and with stirring, shaking, or mild ebullition. Samples were withdrawn at

appropriate intervals and measured spectrophotometrically for color change. Aliquots were adjusted to 20 per cent TBP for uranium transfer rate measurements and to 30 per cent TBP for uranium distribution measurements* and dispersion and coalescence time measurements. Before these tests, samples were given three successive three per cent aqueous sodium carbonate washes and one water wash and then centrifuged. Chemical exposure of the diluent and solvent samples was continued until the organic had remained stable over a period exceeding its estimated life in the process, or until excessive decomposition had occurred.

Inasmuch as spectrophotometric methods were fast and convenient, changes in color were evaluated in detail. The remaining tests were employed only at the conclusion of the exposure or when color trends appeared to justify further examination.

IV. RESULTS AND DISCUSSION

A. Reflux Conditions

An exposure of Shell Spray Base, Soltrol 170, and methyl cyclohexane was made by permitting reflux of a two-phase system of two volumes of 2.25 M HNO_3 and one of the organic. The results of uranium distribution measurements (E_a^o values) following this treatment are shown in Table I, and a number of miscellaneous observations are tabulated below:

(1) In the absence of TBP, the colored impurities markedly distribute into the aqueous phase, this tendency being greater for methyl cyclohexane which has a lower molecular weight than the average for either Shell Spray Base or for Soltrol 170. With TBP present, this effect is not observed, the decomposition products being organic soluble. This fact may account for the low uranium distribution value obtained for methyl cyclohexane.

(2) A UNH solution (268 g. UNH/l.) was prepared by dissolving UO_3 in an aqueous nitric acid phase which had been refluxed with Shell Spray Base. This solution was found to foam severely upon calcination. Similarly, the impurities in the Shell Spray Base phase, when isolated chromatographically, were found to cause foaming at as low as 40 parts per 10^6 parts uranium[1].

(3) The refluxed Shell Spray Base when adjusted to 20 per cent in TBP gave a value of 24 ppm DBP (when analyzed by the Brite method[2]). This is definite evidence of the interference of solvent impurities with the DBP test, since no DBP could possibly have been present.

(4) Observations reported in (2) and (3) help to explain continued foaming trouble at the UO_3 plant under conditions wherein DBP can reasonably be expected to be absent.

*Distribution test and other tests are described in Appendix I.

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(5) Impurities isolated chromatographically from refluxed Shell Spray Base when added (at the four per cent level) to "as received" Shell Spray Base, 20 per cent in TBP, caused markedly increased fission product retention in the organic phase after one extraction contact followed by three scrub contacts (1).

(6) A partial chromatographic analysis followed by infrared examination shows:

(a) Products isolated from Shell Spray Base are similar to those found in Uranium Recovery Process RCW.

(b) Soltrol 170 yields products similar to those from Shell Spray Base, but higher in nitroso compounds.

(c) Methyl cyclohexane* yields organic acids, nitrite esters, and nitro compounds. At least ten discrete colored bands, each indicative of a single substance, were resolved on the alumina column. Time did not permit their complete isolation and identification, but these observations give some insight into the complexity of the analytical problem of analyzing diluents for impurities.

B. Chemical Stability at 71 C

(1) Effect of Nitrous Acid

Actual boiling of aqueous-organic systems is a condition never anticipated in Purex columns, though had the solvents proved stable, chemical effects at lower temperatures could hardly be of consequence. Close observation of the refluxing systems revealed that the reaction was slow at first but accelerated in later stages with accompanying evolution of nitrogen oxides. This suggested the possibility that nitrous acid was participating.

To check this point under less stringent conditions, samples were placed in flasks equipped with stirrers and condensers and immersed in a bath at 71 C. Two volumes of aqueous to one of organic were used with mild stirring at the interface. Samples were taken at intervals and measured spectrophotometrically. A final sample was taken for the remaining tests. These data are recorded in Table II.

It is evident from these data that as little as 0.01 M NO_2^- has a marked effect in accelerating the decomposition of the solvent. In the absence of nitrite ion, the diluent or solvent appears to be

*The sample employed was chromatographed on silica gel until spectrophotometrically pure.

stable at this temperature in the presence of 2.25 M HNO_3. There are some discrepancies in the data, e.g., in the presence of TBP, 0.01 M NO_2^- does not seem to exert as large an effect as in the absence of TBP. It is not known whether this is due to the tendency of TBP to complex (and thus sequester) nitrous acid or simply to uncontrolled nitrite concentration.

In connection with these data, plant operating experience indicates that uranium distribution ("C" test) values in excess of 0.02 lead to operating difficulties. This conclusion results from correlation of a large amount of plant performance data and uranium distribution data taken routinely. Since uranium transfer rate and dispersion and coalescence time measurements are not made routinely, no such correlation exists for these measurements, and it is not possible to establish a similar "go - no go" limit. However, appreciable differences in transfer rate would undoubtedly be reflected in a loss of HTU, and lengthening of coalescence times by 50 - 100 per cent would be accompanied by a decrease in flooding capacity.

(2) Comparison of Diluents

The data in Table II showed that to obtain an effect upon Shell Spray Base, nitrite ion had to be present. In order to compare the other diluents therefore, 0.01 M $NaNO_2$ was introduced along with 2.25 M HNO_3. The result of exposure of various diluents to this aqueous is shown in Table III.

These data show uranium distribution values for Bayol D and Ultrasene containing 30 per cent TBP which are inconsistently high. Aside from this discrepancy, the data show that Shell Spray Base is a little less stable than the other diluents. Soltrol 170 appears to be about the best. To settle the question, the experiment should be repeated under conditions more severe, as for example, by increasing the nitric acid or nitrite concentration.

(3) Effect of Nitric Acid Concentration

Results of exposure of various solvents to aqueous nitric acid - nitrite mixtures at 71 C is shown in Table IV. These data, though incomplete, show definitely that the rate and extent of Shell Spray Base deterioration increases as the nitric acid concentration is increased. The comparison of Soltrol 170 and Shell Spray Base shows the marked superiority of Soltrol 170.

These data show that the deterioration of Shell Spray Base in 6.0 M HNO_3 - 0.05 M $NaNO_2$ at 70 C is appreciable in as little as six hours. Significant deterioration has also occurred at 4.5 M HNO_3 levels in as little as sixteen hours, indicated by the rapid generation of colored impurities.

Decomposition of 30 per cent TBP - Shell Spray Base is not markedly increased by the periodic additions of nitrite, as opposed to the addition of one aliquot at the start of the reaction.

(4) Effect of Nitrite Inhibitors

The effect of nitrite ion is so severe at temperatures near 71 C, that it seems there is good reason to doubt that the Purex process will operate satisfactorily for long at this temperature unless measures are taken to eliminate nitrites from all aqueous phases in contact with organic. The elimination of nitrites can readily be accomplished by the introduction of either urea or sulfamic acid. The effect of these additions on the stability of solvent is shown in Table V.

From the data in Table V, it is clear that in the absence of nitrites, solvents containing Shell Spray Base are satisfactorily stable in 6.0 M HNO_3 at 71 C. In an instance where no nitrite was added before exposure, the solvent was stable for about 43 hours in 6.0 M HNO_3, but rapidly deteriorated thereafter. This indicates that in the absence of a nitrite inhibitor, nitrites eventually are formed, whereupon the reaction accelerates rapidly. In view of these findings, it is recommended that provision be made to exclude nitrites if the Purex process is to be operated in this temperature range.

(5) Behavior of Aromatics

In a report shortly to be issued (3), it was shown that the aromatic hydrocarbon content of Shell Spray Base is altered after the solvent has cycled through the Uranium Recovery Process, and only trivial amounts of reaction products of an aromatic nature remain in the solvent. This effect seems to be explained by a comparison of the chemical effects of 2.25 M HNO_3 + 0.01 M $NaNO_2$ on Shell Spray Base containing variable amounts of aromatic hydrocarbons.

These data are presented in Table VI and show that the rate of color generation is approximately proportional to the aromatic content indicating that the aromatic hydrocarbons are reacting to produce colored constituents. The E_a^o and uranium transfer rate measurements show no significant differences. This may be taken to mean either that the aromatic reaction products, in themselves, have no effect on solvent quality, or that after carbonate washing (which was done prior to E_a^o or uranium transfer rate measurements) the aromatic reaction products are removed. Were this latter the case, then solvent quality tests would show no differences, and failure to find the aromatic reaction products by analysis of recycled Uranium Recovery Process RCW is explained.

V. SUGGESTIONS FOR FURTHER WORK

In addition to the detailed results reported above, a number of miscellaneous experimental results have been obtained which appear to merit further study:

A. The Effect of Uranyl Nitrate

In one instance wherein 0.8 M UNH was incorporated in an aqueous phase 2.25 M in HNO_3 and 0.01 M in $NaNO_2$ and the aqueous contacted with one-half its volume of 30 per cent TBP in Shell Base at 71 C, the organic phase seemed unduly affected. The color was exceptionally intense (after stripping to remove uranium) and the uranium distribution ($E_a^o = 0.083$) and uranium transfer rate ($R = 0.62$) were indicative of unusually extensive reaction after only 93 hours. This result raises the question as to whether uranyl nitrate has exerted an independent effect. Since uranyl nitrate can displace nitric acid from its complex with TBP and make the latter available in relatively higher concentration to the aqueous phase, the effectiveness of nitric acid may be enhanced. It would seem that this effect might well be investigated further.

B. The Chemical Stability of TBP

Solutions of pure vacuum distilled TBP were contacted at 71 C with:

(a) 6.0 M HNO_3 + 0.05 M $NaNO_2$

(b) 4.5 M HNO_3 + 1.0 M UNH (with light excluded)

In case (a), the TBP immediately complexed all of the nitrous acid and the highly characteristic spectrum of the complex thereafter remained unchanged for a period of 240 hours. There was no evidence of reaction leading to the production of colored substances.

In case (b), a small amount of color appeared within 65 hours and remained approximately constant in intensity thereafter. This color is characterized by a maximum at 326 mμ, a minimum at 308 mμ, and intense absorption at shorter wave lengths. E_a^o values on stripped and carbonate washed samples taken at various times ranged from 0.0078 to 0.0051 (decreasing as the exposure continued).

Unfortunately, the presence of uranium prevents measurement of the color or other quality indices "in situ". These tests could be made only after extensive carbonate washing to remove uranium, and this treatment may have removed much of the substance responsible for the color.

C. Comparison of Diluents

It would appear that the choice of experimental conditions for the comparison of diluents (cf. Table III) was unfortunate. For this purpose, more severe conditions would probably give a more reliable

estimate. This is indicated by the comparison of Soltrol 170 and Shell Spray Base solutions shown in Table IV. It would appear desirable to extend these measurements.

D. Measurements at Lower Temperatures

An organic phase 30 per cent in TBP in a Shell Spray Base diluent adjusted to seven per cent volume in aromatic hydrocarbons proved to be stable for over 330 hours when in contact with an aqueous phase of 2.25 M HNO_3 and 0.1 M $NaNO_2$ at 25 C ± 2 C. "As received" Shell Spray Base alone is likewise stable for a similar period when in contact with this aqueous. On the basis of these measurements, it would appear that radiation effects will alone be responsible for any solvent deterioration which may occur at this temperature and a search for chemical effects need not be extended.

Preliminary measurements at 50 C indicate measurable chemical effects occur. The rate of these reactions appears to be approximately one-half that at 70 C in the few instances studied. Extension of this data is contemplated.

VI. REFERENCES

(1) Maness, R. F., Private Communication.

(2) Brite, D. W., "The Determination of Dibutyl Phosphate," HW-30643 Rev., Confidential, October 15, 1954.

(3) Moore, R. H., "Investigation of Solvent Degradation Products in Recycled Uranium Recovery Plant Solvent," HW-34502, Secret, February, 1955.

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APPENDIX I

OUTLINE OF PROCEDURES FOR SOLVENT QUALITY TESTS

A. Uranium Distribution, E_g^o (dilute region)

The solvent sample is adjusted to 30 per cent in TBP, washed three times with an equal volume of three per cent sodium carbonate and once with water. After centrifuging, it is contacted with an equal volume of an aqueous phase containing 1.0 g/l U (as UNH). The clear organic is then analyzed for uranium fluorimetrically, and the distribution coefficient is calculated.

B. Uranium Transfer Rate

The solvent sample is adjusted to 20 per cent in TBP and washed as in the above procedure. The test is done in an apparatus which automatically performs a series of extractions and analyses of the sample. The organic is contacted with an aqueous phase 2.0 M in HNO_3 and 0.70 M in UNH. The uranium content of the aqueous phase is then determined photometrically after successive contacts (stirring) with the organic until equilibria is reached. The rate at which uranium transfers to the organic phase is determined from calibration data and the rate constant K is compared with that of standard solvent. The ratio is reported as R.

C. Dispersion and Coalescence Time

The solvent is adjusted to 30 per cent in TBP and washed as in (A). 12.5 ml. is carefully added to 12.5 ml. of an aqueous 0.75 M in UNH and 2.0 M in nitric acid contained in a 25.0 ml. graduated cylinder. Agitation is started at 60 ± 3 strokes per minute using a reciprocating perforated plate stirrer. The time in seconds required for a packed dispersion to build up to the 21 ml. mark is taken as the dispersion time.

In similar fashion, the sample is contacted with a solution 0.0075 M in UNH, stirred until dispersion is complete and then for an additional 30 seconds. The stirrer is stopped and the time required for the appearance of any part of a clear interface taken as the coalescence time.

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TABLE I - RESULTS OF REFLUX OF 2.25 M HNO_3 AND DILUENTS

Diluent	Hours Refluxed	Temp. of Reflux, °C	E_a^o, U* before CO_3^-	E_a^o, U* after CO_3^- wash
Shell Spray Base	28	103-105	0.08	0.06
Soltrol 170	20	105	--	0.04
Methylcyclo-hexane	18	99	0.015	0.007

*Samples adjusted to 30% vol. TBP prior to test.

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TABLE II - CHEMICAL EFFECTS AT 71 C, EFFECT OF NITRITE ION

Sample Description	Exposure Time Hrs.	Color Intensity O.D. Units 430 mμ	Color Intensity O.D. Units 320 mμ	E_a^o U	Uranium Transfer Rate K	Uranium Transfer Rate R	Dispersion Time Sec.	Coalescence Time Sec.
"As Received" Shell	45	0.08	0.26	--	--	--	--	--
Spray Base	96	0.02	0.26	--	--	--	--	--
vs.	163	0.02	0.30	0.0051	14.24	1.11	68	19
2.25 M HNO_3								
"As Received" Shell	26	0.21	36.0	--	--	--	--	--
Spray Base	77	0.26	37.0	--	--	--	--	--
vs.	144	0.42	43.0	0.055	9.24	0.70	57	30
2.25 M HNO_3, 0.01 M $NaNO_2$								
"As Received" Shell	27	0.025	0.50	--	--	--	--	--
Spray Base + 30% vol.	67	0.020	0.60	--	--	--	--	--
TBP (CO_3 washed)	139	0.050	0.80	0.008	11.69	0.97	80	46
vs. 2.25 M HNO_3								
"As Received" Shell	20	0.32	17.8	--	--	--	--	--
Spray Base + 30% vol.	67	0.29	23.0	--	--	--	--	--
TBP (CO_3 washed)	139	0.27	26.0	0.028	13.00	1.02	62	53
vs. 2.25 M HNO_3 + 0.01 M NO_2^-								
"As Received" Shell	20	0.39	27.0	--	--	--	--	--
Spray Base + 30% vol.	67	0.46	32.0	--	--	--	--	--
TBP (CO_3 washed)	139	0.69	43.0	0.19	10.01	0.79	45	55
vs. 2.25 M HNO_3, 0.1 M $NaNO_2$								

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TABLE III - CHEMICAL STABILITY OF VARIOUS DILUENTS AT 71 C IN TWO-PHASE SYSTEMS*

Sample Description	Hours Exposure	Color Intensity O.D. Units 430 mμ	320 mμ	E^{o}_{a} U	Uranium Transfer Rate K	R	Dispersion Time Sec.	Coalescence Time Sec.
	26	0.21	36.0					
Shell Spray Base	77	0.26	37.0					
	144	0.42	43.0	0.055	9.24	0.70	57	30
	26	0.035	4.5					
Soltrol - 170	97	0.035	5.3					
	195	0.075	6.9	0.014	11.07	0.84	53	22
	26	0.04	3.2					
Bayol - D	96	0.04	4.8					
	195	0.12	12.2	0.023	10.53	0.88	60	22
	16	0.14	2.4					
Ultrasene	93	0.22	2.7					
	189	0.11	3.1	0.010	14.61	1.16	82	24
	20	0.32	17.8					
Shell Spray Base	67	0.29	23.0					
30% TBP	139	0.27	26.0	0.028	13.00	1.02	62	53
	22	0.09	7.0					
Soltrol 170	92	0.17	7.5					
30% TBP	170	0.17	7.6	0.014	15.06	1.08	76	20
	22	0.07	4.2					
Bayol D	92	0.25	17.5					
30% TBP	170	0.54	24.0	0.14	12.41	0.90	56	32
	22	0.13	5.8					
Ultrasene	92	0.21	14.2					
30% TBP	170	0.46	20.0	0.14	10.45	0.76	62	28

* Aqueous phase comp. - 2.25 M HNO_3 + 0.01 M $NaNO_2$.

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TABLE IV - EFFECT OF NITRIC ACID CONCENTRATION ON CHEMICAL STABILITY AT 71 C

Sample Description	Hours Exposure	Color Intensity O.D. Units 430 mμ	Color Intensity O.D. Units 320 mμ	E_a^o U	Uranium Transfer Rate K	Uranium Transfer Rate R	Dispersion Time Sec.	Coalescence Time Sec.
Shell Spray Base +	20	0.32	17.8					
30% TBP vs. 2.25 M	67	0.29	23.0					
HNO_3 + 0.01 M $NaNO_2$	139	0.27	26.0	0.028	13.00	1.02	62	53
Shell Spray Base +	16	0.52	37.0					
30% TBP vs. 4.5 M	40	0.96	55.0					
HNO_3 + 0.01 M $NaNO_2$	93	2.00	74.0	0.11	7.92	0.66	31	58
Shell Spray Base +	6	0.38	36.0	0.061				
30% TBP vs. 6.0 M	23	0.91	59.0	0.073				
HNO_3 + 0.05 M $NaNO_2$	96	2.60	128.0	0.23				
Soltrol 170 + 30%	6	0.11	6.2	0.029				
TBP vs. 6.0 M HNO_3	23	0.12	12.0	0.034				
+ 0.05 M $NaNO_2$	96	0.80	28.0	0.039				
Shell Spray Base +	23	0.28	26.0					
30% TBP vs. 2.25 M	54	0.57	37.0					
HNO_3 with 0.01 M	96	0.80	42.0					
$NaNO_2$ present initially	240	1.31	61.0					
& added periodically in	310	1.88	74.0	0.059				
0.01 M increments								

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TABLE V - THE EFFECT OF NITRITE INHIBITORS ON SOLVENT STABILITY AT 71 C

Sample Description	Hours Exposure	Color Intensity O.D. Units 430 mμ	320 mμ	E^o_a U
30% TBP in Shell Spray Base vs. 6.0 M HNO_3	25	0.09	1.32	
	43	0.24	3.10	
	163	2.50	104.0	
	238	2.70	118.0	0.30
Same, but with 0.2 M urea in aq. ph.	25	0.02	0.94	
	163	0.27	3.50	
	238	0.38	5.40	
	330	0.38	4.90	0.0097
Same, but with 0.2 M sulfamic acid in place or urea	65	0.07	1.05	
	91	0.10	1.33	
	119	0.12	1.73	
	240	0.36	4.40	0.0073
Shell Spray Base vs. 2.25 M HNO_3	45	0.08	0.26	
	96	0.02	0.26	
	163	0.02	0.30	0.0047
Same, but with addition 0.2 M urea	25	0.02	0.22	
	76	0.02	0.20	
	142	0.02	0.22	0.0040

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TABLE VI - EFFECTS DUE TO AROMATIC CONSTITUENTS AT 71 C

Sample Description	Exposure Time Hours	Color Intensity O.D. Units 430 mμ	320 mμ	E_a^o U	Uranium Transfer Rate K	R
"Aromatic Free" Shell Spray Base vs. 2.25 M HNO_3 + 0.01 M $NaNO_2$	20 70 163	0.02 0.08 0.25	3.0 8.8 24.0	0.064	8.2	0.63
"As Received" Shell Spray Base, containing 2% vol. Aromatics vs. 2.25 M HNO_3 + 0.01 M $NaNO_2$	26 77 144	0.21 0.26 0.42	36.0 37.0 43.0	0.055	9.2	0.70
"As Received" Shell Spray Base + 5% vol. Aromatics (total of 7% vol.) vs. 2.25 M HNO_3 + 0.01 M $NaNO_2$	4 21 72 141	1.65 0.57 0.70 0.78	4.7 27.0 80.0 83.0	0.088	8.75	0.66

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