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Chemistry

AEC Research and Development Report

EXTRACTION OF THORIUM NITRATE FROM NITRIC ACID BY TBP - "ULTRASENE"

by

T. H. Siddall, III

Separations Chemistry Division

October 1956

E. I. du Pont de Nemours & Co.
Explosives Department - Atomic Energy Division
Technical Division - Savannah River Laboratory

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DP - 181

CHEMISTRY

EXTRACTION OF THORIUM NITRATE FROM NITRIC ACID BY TBP - "ULTRASENE"

by

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ABSTRACT

Equilibrium data are presented for the distribution of thorium nitrate and nitric acid between aqueous solutions and tri-n-butyl phosphate diluted with kerosene.

External Distribution according to
TID-4500 (12th Ed.)

TABLE OF CONTENTS

LIST OF TABLES AND FIGURES

EXTRACTION OF THORIUM NITRATE FROM NITRIC ACID BY TBP - "ULTRASENE"

INTRODUCTION

Thorium can effectively be separated from uranium and fission products by liquid-liquid extraction processes using tri-n-butyl phosphate (TBP) as the extractant. Processes for this separation have been described[1], but detailed equilibrium data have not been available. This report is a presentation of such data for the distribution of thorium between aqueous solutions of thorium nitrate - nitric acid and a solution of TBP in "Ultrasene".

SUMMARY

The distribution of thorium nitrate and nitric acid between aqueous solutions and TBP - "Ultrasene" was determined at 30° and 50°C for 30 per cent and at 30°C for 42.5 per cent TBP in the solvent. The data were obtained from 144 batch equilibrations and are presented in the form of tables and three equilibrium diagrams. The concentrations of thorium nitrate and nitric acid in the aqueous phase cover the range from 0.01 to 0.7 mol per liter and from 0 to 4 mols per liter, respectively. Data for the distribution of nitric acid in the absence of thorium are also presented.

As has been reported[1], a second organic phase exists at high concentrations of thorium. The combinations of thorium and acid concentrations and temperature required to form this second organic phase were determined for 30 per cent TBP in "Ultrasene". Lines separating the two- and three-phase regions are shown in the two diagrams for 30 per cent TBP. No second organic phase was observed in the concentration ranges shown in the diagram for 42.5 per cent TBP, but such a phase was observed in qualitative tests at higher concentrations of thorium than those used in the distribution studies.

EXPERIMENTAL

REAGENTS AND PREPARATION OF MATERIALS

C. P. thorium nitrate and nitric acid were used throughout the work.

The TBP was "Butanol Free" grade and was washed with dichromate, caustic, and water before use. After this treatment the TBP was dried under vacuum at room temperature.

"Ultrasene", a product of the Atlantic Refining Company, is a mixture of normal, iso-, and cycloparaffins with a very low content of olefins and aromatics. The average molecular weight is 175 (between C_{12} and C_{13}). The "Ultrasene" was washed with sulfuric acid to remove traces of olefins or aromatics.

The TBP - "Ultrasene" solutions were prepared by weighing the dried TBP into volumetric flasks and adding the diluent. These solutions were washed with caustic and water to remove dibutyl phosphate immediately before use.

EXPERIMENTAL METHOD

The equilibrium data were obtained by batch contacts of portions of the aqueous and organic phases. TBP - "Ultrasene" and appropriate aqueous phases containing the thorium nitrate and nitric acid were placed in centrifuge cones. The centrifuge cones were then placed in a water bath which was controlled to within ±0.2°C of the desired temperature. After the cones and contents had reached thermal equilibrium, the phases were mixed thoroughly. The resulting emulsions were allowed to separate. The cones were centrifuged, and samples of both final phases were analyzed.

ANALYTICAL METHODS

"Versene" Method for Thorium

Most of the thorium analyses were done by the volumetric method of Fritz and Ford[2]. This method is based on the titration of thorium with a standard "Versene" (ethylenediamine tetraacetic acid) solution at pH 2.8, with Alizarin Red S as the indicator. Solutions were buffered with acetic acid - ammonium acetate. The "Versene" was standardized by titration against a thorium nitrate solution which had been standardized by conversion to oxide.

Samples of the organic phase were analyzed directly without preliminary stripping of the thorium into an aqueous medium. Some of the indicator was extracted into the organic phase, but enough remained in the aqueous phase to provide a sharp end point.

The method was found to be fast and accurate. An average deviation of 0.5 per cent between duplicates was easily obtainable.

"Thoron" Method for Thorium

The colorimetric method for thorium described by Thomason, Perry, and Byerly[3] was used for samples which contained less than 0.01 mol of thorium per liter. This method was satisfactory but the thorium in organic samples had to be stripped into water prior to analysis. This extra step was time consuming and a potential source of error.

Determination of Acid in the Presence of Thorium

Acid was determined potentiometrically with a Beckman titrimeter. Thorium interference was prevented by adding potassium oxalate to complex the thorium. From the shape of the titration curve pH 7.2 was taken as the end point for aqueous samples. For organic samples the titration curve was shifted slightly, and pH 7.6 was taken as the end point. Organic samples were titrated directly without preliminary stripping.

The method was found to be very rapid and quite accurate. Average deviations between duplicates were less than one per cent for acidities above 0.2 mol per liter. At lower acidities, end points became progressively less certain, down to about 0.05 mol per liter at which point direct determinations could no longer be made.

The concentrations in some of the solvent samples were in the very low range. These values were determined by titrating acid in the aqueous phase before and after contact with the organic phase. By using large volumes of organic phase the concentration could be calculated from the amount of acid transferred.

DISCUSSION

DISTRIBUTION DATA

The following symbols are used in the diagrams, tables, and text:

- Th_a - concentration of thorium nitrate in the aqueous phase at equilibrium
- Th_o - concentration of thorium nitrate in the organic phase at equilibrium
- H_a - concentration of nitric acid in the aqueous phase at equilibrium
- H_o - concentration of nitric acid in the organic phase at equilibrium
- H_o' - concentration of nitric acid in the organic phase in equilibrium with aqueous nitric acid only

All concentrations are expressed as mols of solute per liter of solution at 23°C. Concentrations may be converted to a weight basis, if desired, by using the density equations:

$$\text{Aqueous density } (23^\circ C) = 1.001 + 0.401\ Th_a + 0.030\ H_a$$

$$\text{Solvent density (30 per cent TBP, } 23^\circ C) = 0.836 + 0.402\ Th_o + 0.026\ H_o$$

A density equation was not determined for 42.5 per cent TBP. The molar density coefficients should, however, remain unchanged. Work with uranyl nitrate and nitric acid has shown that molar density coefficients change very little with changes in TBP concentration. The constant term in the equation will increase from 0.836 to 0.861 because of the higher TBP content.

The solid lines in Figures 1, 2, and 3 are lines of constant H_a. The dotted lines are lines of constant H_o. The heavy solid lines in Figures 1 and 2 define the boundary between two- and three-phase regions. Below the line there is a single organic phase and above it there are two.

The lines of constant H_a were constructed by graphical interpolation of the data given in Tables I, II, and III and in Figure 4.

The lines of constant H_o were more difficult to construct. Fortunately a guide was available in the form of a relation discovered by Dr. W. O. Haas at Knolls Atomic Power Laboratory, for the extraction of uranium into TBP. Haas found that the ratio of H_o in the presence of uranium to H_o' in the absence of uranium both at the same H_a is a linear function of U_o over a wide range of composition. This ratio may be plotted against U_o to give a single straight line for a wide range of concentrations. This relationship may or may not have fundamental significance, but in any event, it is of great assistance in constructing equilibrium diagrams.

A modified relationship held well for the thorium system when H_a was greater than one mol per liter. The ratio of H_o to H_o' was found to depend on H_a as well as on Th_o, and it was necessary to plot a family of lines at various constant values of H_a. The change in slope of the lines is fairly insensitive to H_a, however; points which vary as much as 20 per cent in H_a fit to the same line. A typical plot of this sort is illustrated in Figure 5, a plot of the data for 30 per cent TBP at 50°C. For H_a less than one mol per liter the relationship is no longer linear. Instead, the curve goes through a maximum with Th_o.

The data for 30 per cent TBP are more precise than those for 42.5 per cent TBP, primarily because the 30 per cent data were measured later, after analytical methods had been refined in practice. No rigorous analysis of errors was made. However, duplicate experiments and analyses, together with the fit to smooth curves, suggests that the typical average deviations are two per cent or less for the data for the 30 per cent TBP system.

SECOND ORGANIC PHASE

As the thorium content of 30 per cent TBP was increased to about 0.2 mol per liter a second organic phase appeared. This second organic phase appeared first as cloudiness in the organic layer. As conditions favoring the appearance of the phase were intensified the volume of the phase increased and it separated as a discrete layer immediately above the aqueous layer. Either increasing the nitric acid concentration of the organic phase or decreasing the temperature caused the second organic phase to appear at lower thorium concentration.

No experiments were done specifically to explain the nature of the second organic phase in the TBP - "Ultrasene" system or why it

occurs. However, the data suggest that the mechanism is very simple. Depending on temperature and the concentrations of thorium and acid, the thorium complex with TBP in the organic phase exceeds its solubility in the two-phase system and a third phase forms. The minimum thorium concentrations for the formation of the second organic phase are indicated for various acid concentrations and temperatures in Figure 6.

Qualitative tests showed that the temperature and composition at which the second organic phase occurs with thorium is a function of the nature of the diluent and the extractant. When the diluent was a hydrocarbon of lower molecular weight such as n-octane, it was much more difficult to form the second organic phase. If tri-isoamyl phosphate was substituted for TBP it was also more difficult to form the extra phase. Both of these observations support the proposition that appearance of the second organic phase is controlled by the solubility of the thorium complex in the original organic phase. It must be that the thorium - TBP complex is more soluble in octane than it is in "Ultrasene". The thorium-tri-isoamyl phosphate complex must be more soluble in "Ultrasene" than is the TBP complex.

The data defining the boundary between the region of a single organic phase and the region of two organic phases are given in Table IV and Figure 6. The data were obtained by equilibrating aqueous and organic phases while slowly decreasing the temperature. The temperatures recorded in the table are those at which the second organic phase first manifested itself by faint cloudiness in the organic layer. The temperature was then increased one or two degrees and both phases were sampled. This sampling technique introduced small errors since pipettes were not calibrated for elevated temperature. Otherwise the technique was quite satisfactory. The points at which the second organic phase appeared were easily fixed.

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2. Fritz, J. J. and Ford, J. J. "Titrimetric Determination of Thorium" Anal. Chem. 25 1640 (1953).

3. Thomason, P. F., Perry, M. A., and Byerly, W. M. "Determination of Microgram Amounts of Thorium" Anal. Chem. 21 1239 (1949).

TABLE I

THORIUM NITRATE - NITRIC ACID - 30 PER CENT TBP - "ULTRASENE" AT 30°C

H_a	H_o	Th_a	Th_o
0.0 (Th_a/H_a(excess) = 100/3) for this series.		0.636	0.235
		0.480	0.179
		0.426	0.157
		0.352	0.117
		0.333	0.108
		0.283	0.079
		0.240	0.0550
		0.200	0.0339
		0.179	0.0243
		0.136	0.0110
		0.085	0.00235
0.30	--	0.532	0.211
0.29	--	0.318	0.126
0.29	--	0.201	0.0661
0.28	--	0.122	0.0263
0.28	--	0.078	0.0110
0.28	--	0.0586	0.0069
0.29	--	0.0460	0.0048
0.370	0.066	0.281	0.113
0.358	0.063	0.230	0.086
0.315	0.056	0.172	0.0526
0.319	0.055	0.127	0.0327
0.319	0.044	0.0489	0.0057
0.58	0.081	0.519	0.218
0.56	0.093	0.316	0.141
0.54	0.098	0.185	0.080
0.54	0.098	0.108	0.0389
0.54	0.095	0.0674	0.0202
0.54	--	0.0549	0.0155
0.54	--	0.0398	0.0110
0.54	0.089	0.0239	0.0053
1.10	0.145	0.397	0.195
1.12	0.162	0.335	0.182
1.11	0.174	0.254	0.148
1.08	0.189	0.172	0.112
1.07	0.189	0.138	0.095
1.07	0.198	0.103	0.0683
1.05	0.201	0.094	0.0646
1.06	0.206	0.0524	0.0360
1.06	0.211	0.0267	0.0184
1.07	0.215	0.0102	0.0066

TABLE I (Continued)

H_a	H_o	Th_a	Th_o
1.43	3rd phase	0.376	3rd phase
1.57	0.196	0.363	0.197
1.52	0.211	0.308	0.186
1.53	0.229	0.221	0.153
1.51	0.250	0.162	0.126
1.53	0.268	0.107	0.095
1.49	0.291	0.083	0.076
1.54	0.293	0.0418	0.0432
1.46	0.318	0.0254	0.0271
1.45	0.319	0.0079	0.0090
2.03	0.266	0.234	0.170
2.02	0.276	0.194	0.154
1.97	0.303	0.150	0.132
3.05	0.386	0.202	0.178
3.15	0.408	0.181	0.174
3.09	0.430	0.176	0.169
3.12	0.447	0.122	0.146
3.06	0.471	0.118	0.141
3.08	0.494	0.0675	0.105
3.01	0.52	0.0545	0.092
3.00	0.56	0.0282	0.0589
2.95	0.60	0.099	0.0249
2.98	0.64	0.0463	0.0129
4.50	0.488	0.140	0.165*
4.55	0.55	0.129	0.161
4.51	0.57	0.090	0.140
4.48	0.60	0.0631	0.113
4.40	0.70	0.0313	0.081
4.31	0.72	0.0131	0.0432
4.28	0.78	0.00482	0.0224

* No third phase at 30°C. Third phase appears between 30° and 25°C The next point gave no third phase when the organic phase was brought to 25°C.

TABLE II

THORIUM NITRATE - NITRIC ACID - 30 PER CENT TBP - "ULTRASENE" AT 50°C

H_a	H_o	Th_a	Th_o
0.0 (Th_a/H_a(excess) = 100/3) for this series.		0.670	0.229
		0.504	0.171
		0.450	0.149
		0.378	0.111
		0.350	0.099
		0.305	0.073
		0.208	0.0272
		0.087	0.0006
0.28	--	0.509	0.189
0.27	--	0.313	0.106
0.27	--	0.202	0.0527
0.27	--	0.127	0.0204
0.28	--	0.080	0.0061
0.29	--	0.0280	0.0016
0.58	0.086	0.532	0.209
0.56	0.094	0.315	0.131
0.56	0.094	0.191	0.074
0.55	0.091	0.113	0.0347
0.55	0.086	0.071	0.0175
0.56	0.079	0.0249	0.0046
1.55	0.220	0.388	0.200
1.53	0.228	0.320	0.178
1.53	0.238	0.231	0.146
1.54	0.261	0.172	0.118
1.52	0.225	0.115	0.091
1.53	0.289	0.086	0.071
1.49	0.290	0.0458	0.0396
1.49	0.300	0.0268	0.0250
1.48	0.311	0.0086	0.0084
3.06	0.465	0.194	0.159
3.05	0.50	0.133	0.130
2.98	0.53	0.0608	0.083
3.02	0.59	0.0321	0.0545
3.00	0.67	0.0111	0.0228
2.99	0.65	0.00517	0.0116
4.66	0.60	0.149	0.144
4.59	0.64	0.108	0.124
4.54	0.68	0.0716	0.101
4.50	0.73	0.0408	0.0736
4.40	0.79	0.0170	0.0390
4.37	0.84	0.0056	0.0153

TABLE III

THORIUM NITRATE - NITRIC ACID - 42.5 PER CENT TBP - "ULTRASENE" AT 30°C

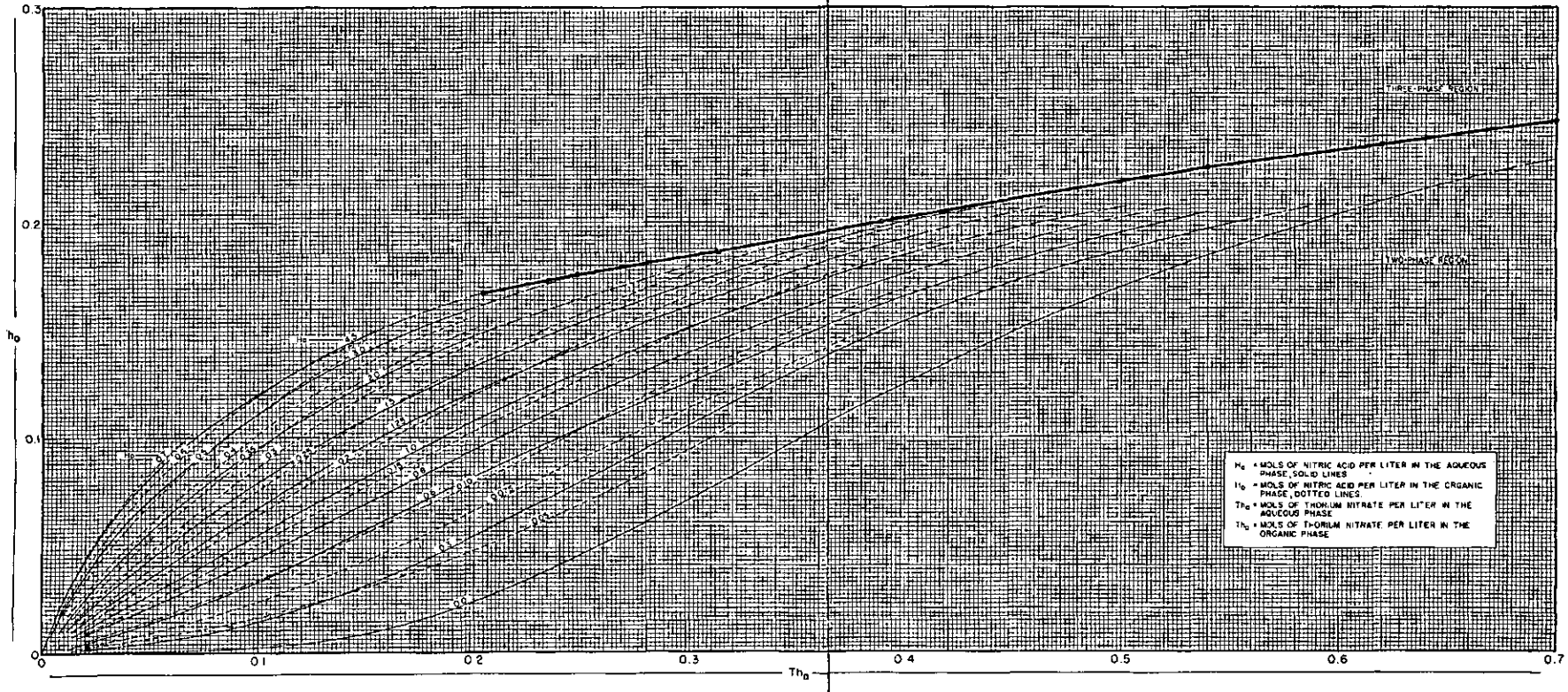
H_a	H_o	Th_a	Th_o
0.0	0.0	0.617	0.336
0.0	0.0	0.510	0.293
0.0	0.0	0.388	0.216
0.0	0.0	0.281	0.122
0.0	0.0	0.173	0.0307
0.0	0.0	0.0944	0.0074
0.47	0.10	0.518	0.308
0.47	0.10	0.367	0.242
0.44	0.14	0.254	0.156
0.44	0.12	0.146	0.058
0.45	0.10	0.0775	0.0243
0.93	0.17	0.433	0.298
0.94	0.19	0.341	0.254
0.85	0.19	0.210	0.171
0.86	0.23	0.112	0.092
0.86	0.25	0.055	0.047
1.89	0.30	0.305	0.294
1.83	0.36	0.180	0.230
1.74	0.42	0.0814	0.122
1.66	0.46	0.0348	0.0670
2.65	0.44	0.194	0.248
2.86	0.47	0.162	0.232
2.69	0.60	0.0468	0.136
2.66	0.67	0.0222	0.077
3.08	0.525	0.157	0.249
3.10	0.609	0.091	0.198
3.08	0.650	0.0663	0.165
3.07	0.701	0.0439	0.128
3.05	0.762	0.0253	0.090
3.06	0.830	0.0108	0.0467
3.54	0.74	0.0497	0.146
3.56	0.85	0.0222	0.0785

TABLE IV

LIMITING CONDITIONS FOR SECOND ORGANIC PHASE

Temperature (°C)	H_a	H_o	Th_a	Th_o
68	None		1.18	0.355
49	None		0.96	0.328
32	None		0.75	0.272
16	None		0.536	0.237
63	0.98	0.15	1.00	0.268
49	0.94	0.14	0.80	0.262
30	0.91	0.11	0.542	0.218
15	0.89	0.13	0.331	0.173
69	1.94	0.23	0.99	0.259
56	1.82	0.22	0.79	0.253
42	1.87	0.22	0.558	0.229
26	1.89	0.24	0.316	0.191
65	2.88	0.26	0.79	0.243
47	2.84	0.30	0.548	0.230
31	2.78	0.30	0.309	0.199
25	2.68	0.32	0.222	0.180

FIGURE 2



EQUILIBRIUM DIAGRAM : THORIUM NITRATE – NITRIC ACID – 30% TBP – "ULTRASENE" AT 50° C

4

FIGURE 1

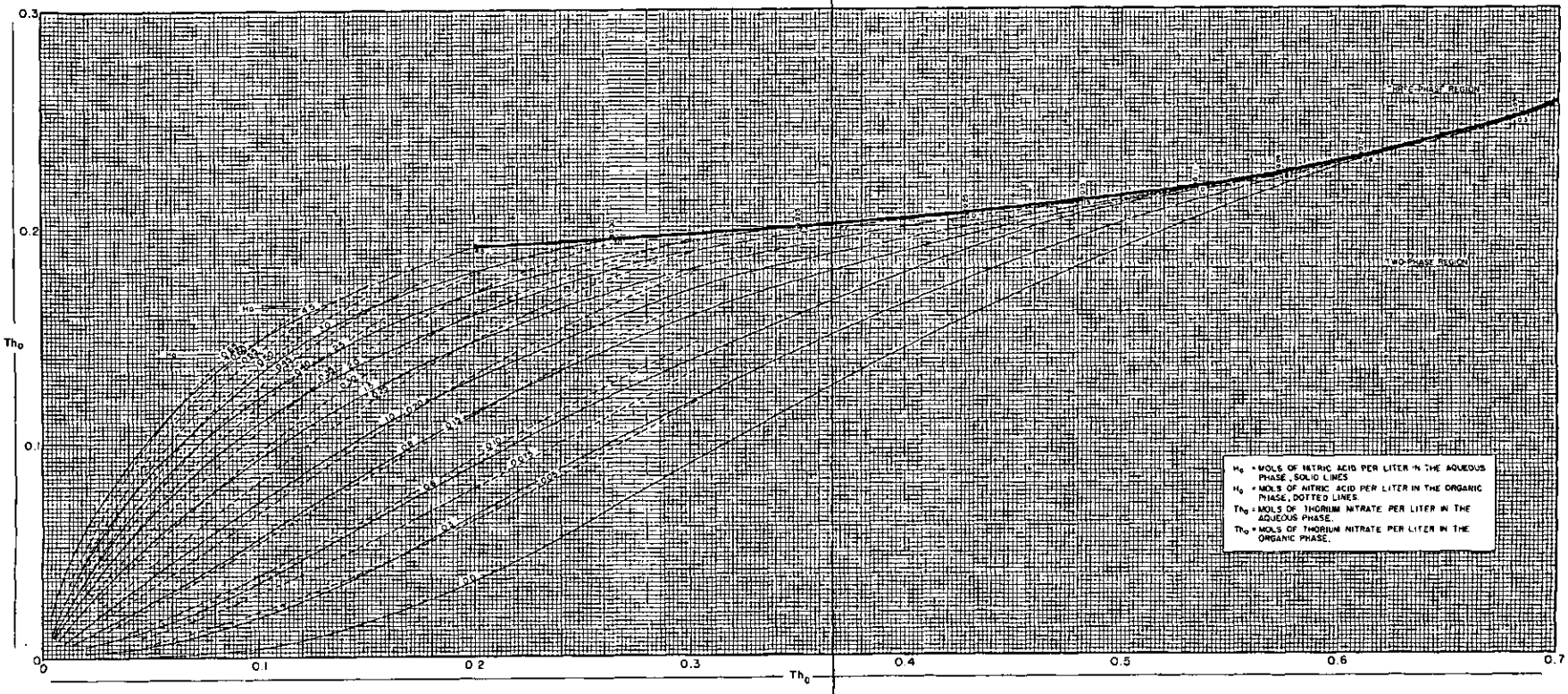


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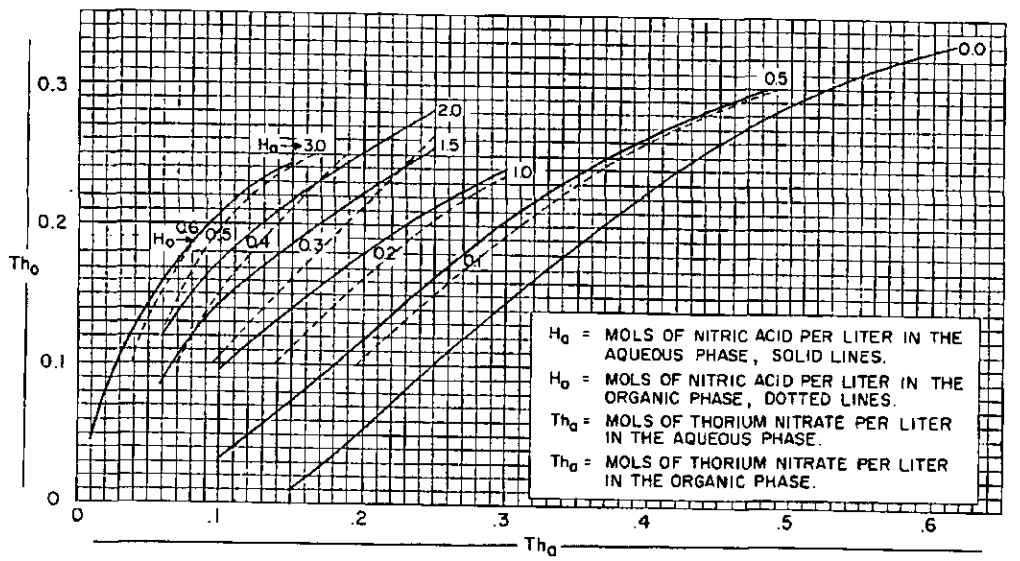
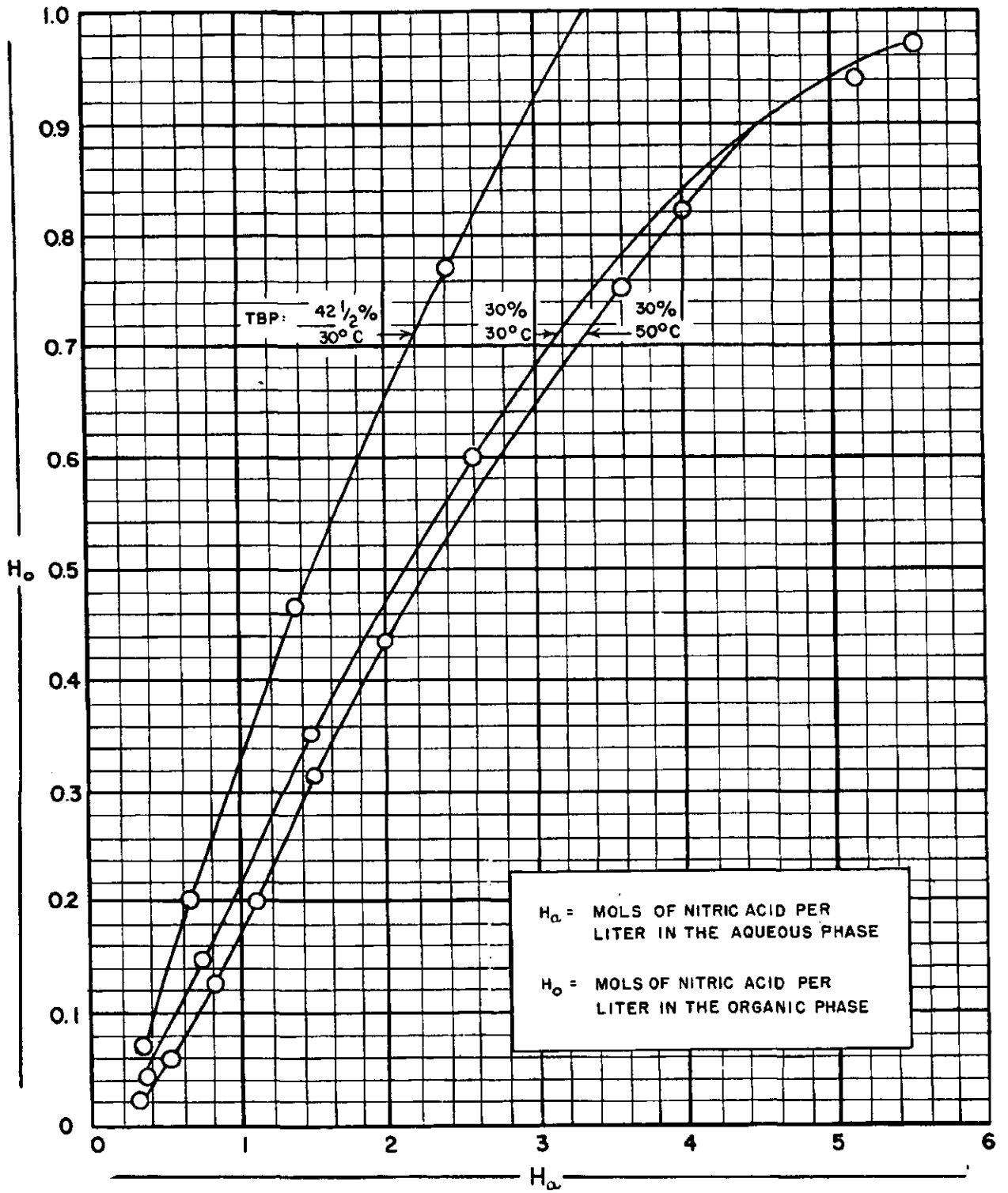
FIGURE 3



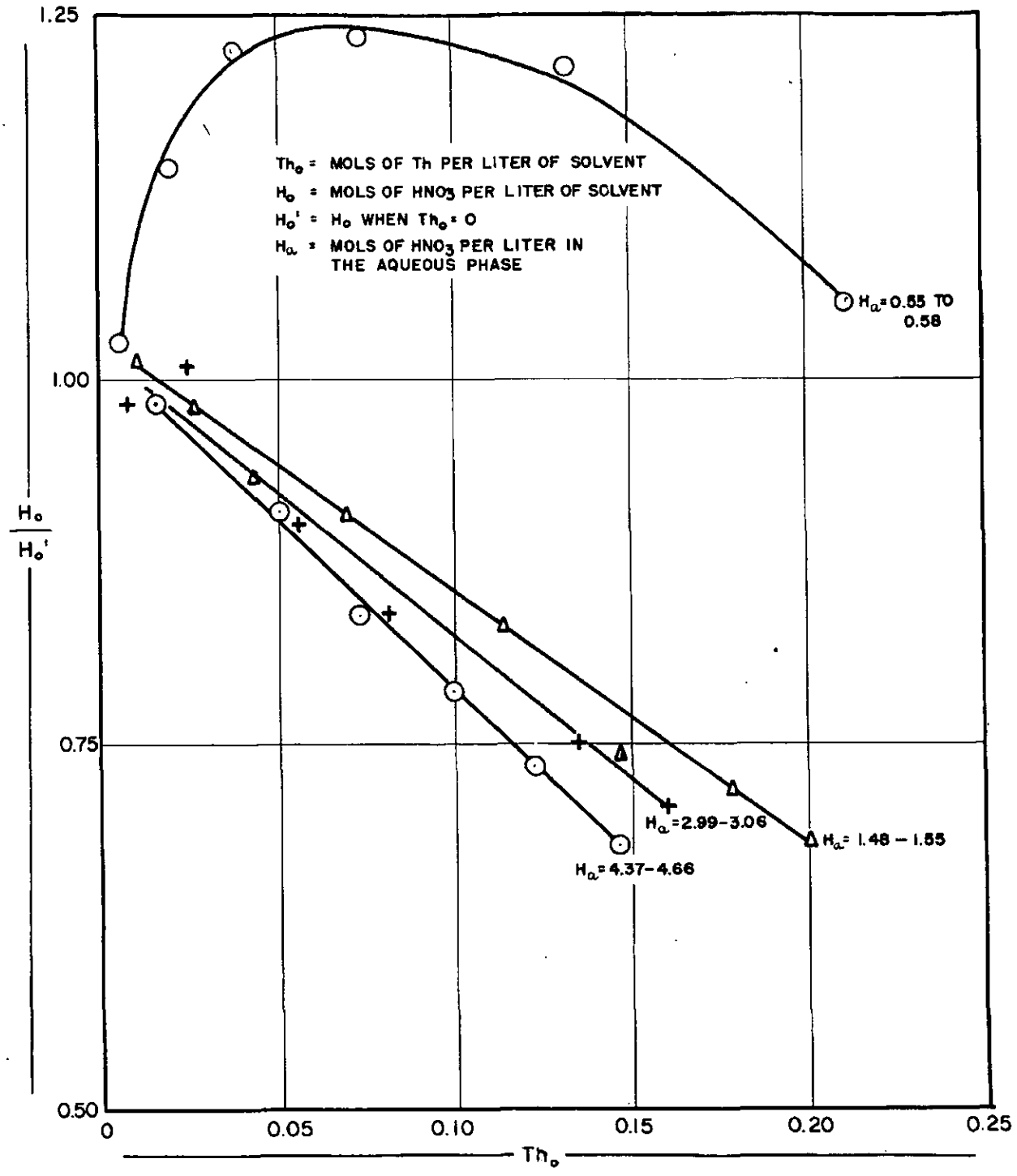
EQUILIBRIUM DIAGRAM : THORIUM NITRATE - NITRIC ACID - 42.5% TBP - "ULTRASENE" AT 30° C

FIGURE 4



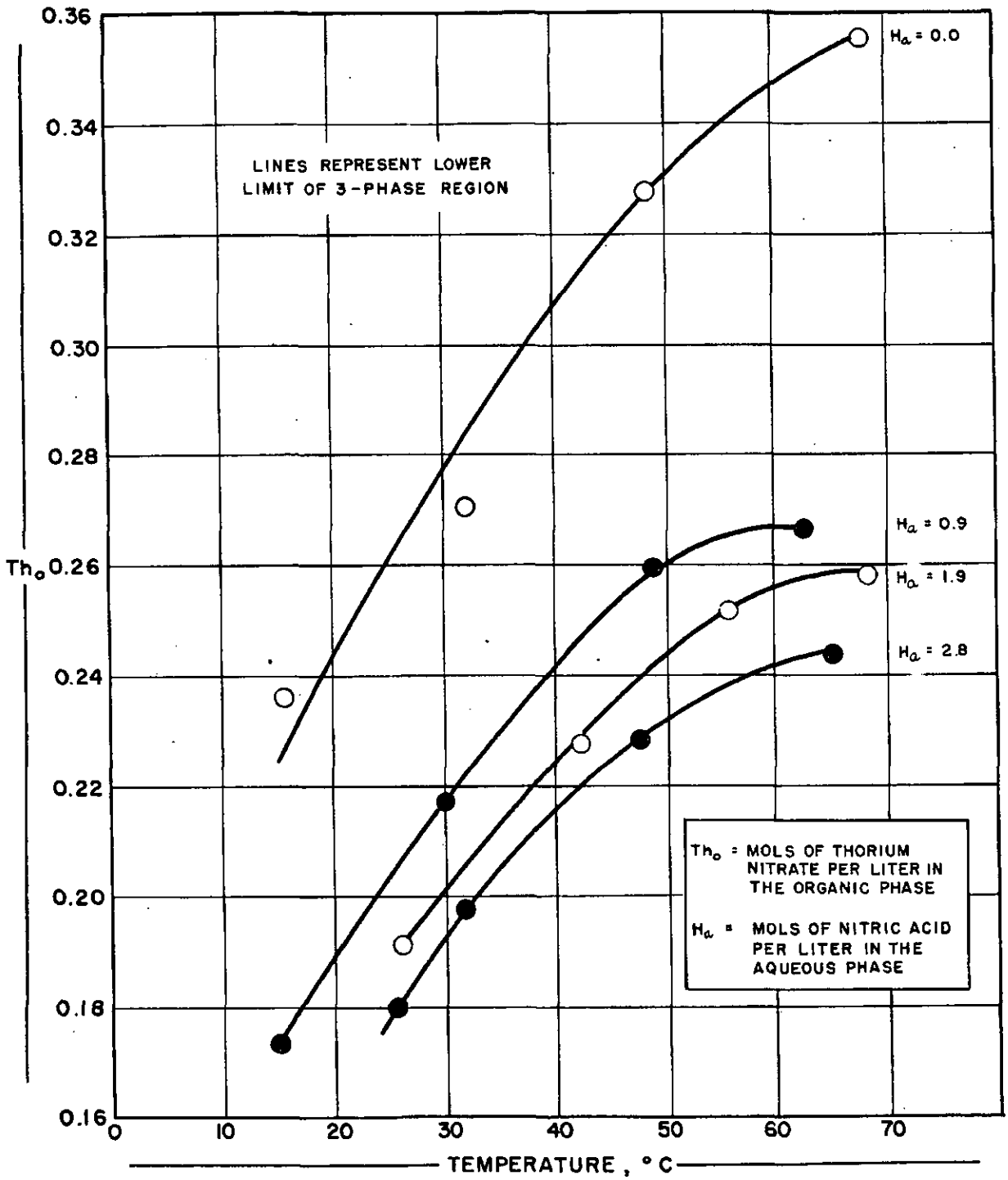
NITRIC ACID DISTRIBUTION BETWEEN AQUEOUS AND ORGANIC PHASES

FIGURE 5



H_o / H_o' VS. Th_o – 30% TBP AT 50°C

FIGURE 6



LIMITS FOR THE EXISTENCE OF A SECOND ORGANIC PHASE

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April 23, 1957

cc. JCA

Mr. Hood Worthington, Director
Technical Division - AED
Explosives Department
E. I. du Pont de Nemours & Company
Wilmington, Delaware

Dear Mr. Worthington:

The following unclassified report transmitted by your letter of December 21, 1956, has been reviewed as to its classification and patent matters. We agree that it may be released as written.

DP-181 "Extraction of Thorium Nitrate From Nitric Acid by TBP - 'Ultrasene'" by T. H. Siddall, III

Dr. Wahl's office has been notified of this action by telephone.

Very truly yours,

J. V. Levy, Director
Industrial & Technical
Services Division

CC: Dr. M. H. Wahl (2)

E. I. DU PONT DE NEMOURS & COMPANY
INCORPORATED
SAVANNAH RIVER PLANT
AIKEN, SOUTH CAROLINA
(TEL. & TEL. ADDRESS, AUGUSTA, GA.)

EXPLOSIVES DEPARTMENT
ATOMIC ENERGY DIVISION

REC-D15--15

CC: F. A. Robertson - SROO
S. A. McKnight
L. C. Evans - H. Worthington
M. H. Wahl - J. W. Morris -
J. Q. Andrews - TIS File
J. O. Morrison

December 21, 1956

Mr. Joel V. Levy (2)
Assistant Director for Technical
Technical and Production Division
Savannah River Operations Office
U. S. Atomic Energy Commission
Post Office Box A
Aiken, South Carolina

Dear Mr. Levy:

PROPOSED PUBLICATION - DP-181

Attached for review as to classification and patent matters are two copies of the following unclassified report:

Extraction of Thorium Nitrate
from Nitric Acid by TBP-"Ultrasene"

by T. H. Siddall

which we propose to release for standard external distribution.

To facilitate the release of this report, it would be appreciated if you would telephone your comments to M. H. Wahl's office and send a confirming letter to me with a copy to M. H. Wahl. The report will be released when approval is received, but not until after 14 days from the date shown above.

If further clarification or technical information are needed to aid in your patent review, we suggest you contact

J. O. Morrison
Separations Chemistry Division
Savannah River Laboratory

If there is a continued interest in pursuing the patentability of a development, I would be happy to supply the additional information required, such as appropriate references and the name of the person responsible for the development.

ATOMIC ENERGY DIVISION

Hood Worthington /sec

Hood Worthington, Director
Technical Division

HW:eef

U. S. Atomic Energy Commission

Document No.
DP-181

PUBLICATION FORM

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1. Document Title
Extraction of Thorium Nitrate from Nitric Acid by TBP-"Ultrasene"

2. Author(s)
T. H. Siddall, III

3. Contract Number under which document was prepared
AT(07-2)-1

4. Document Date
October 1956

5. Document is (check one)
[X] Unclassified [ ] Submitted for declassification

6. If information in this document is also contained in a project report, indicate number. _____
Is this information identical? [ ] Yes [ ] No

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13. Submitted by:
J. W. Hill (Name) — Savannah River Laboratory (Installation)

5-8-57 (Date)

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TECHNICAL DIVISION
SAVANNAH RIVER LABORATORY

Report Data Sheet

Report Number DP-181 S C (U) Approved by [signature]

Author T. H. SIDDALL, III 2385

Title EXTRACTION OF THORIUM NITRATE FROM NITRIC ACID BY TBP - "ULTRASENE"

Category Chemistry Approved by [signature]

Abstract _____ Approved by [signature]

	Author	Supv.	Div. Hd.	Sec. Dir.	Lab. Dir.
Manuscript Approval	THSiddall	_____	[signature]	~~[signature] 10/11/56~~	[signature] 10/12
Master Approval	THS	.....	[signature] 11/5	~~[signature] 11/6/56~~	
Final Copy Approval	THS	.....	✓ [signature] 11/5	.....	.....
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181 (U) Extraction of Thorium Nitrate from Nitric Acid by TBP-Ultrasene by T. H. Siddall, III

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BCC: Hood Worthington - L. C. Evans
J. W. Morris - J. C. Andrews

E. I. DU PONT DE NEMOURS & COMPANY
INCORPORATED
WILMINGTON, DELAWARE

EXPLOSIVES DEPARTMENT

January 7, 1957

Mr. J. V. Levy, Director (2)
Industrial and Technical Services Division
Savannah River Operations Office
United States Atomic Energy Commission
Aiken, South Carolina

Dear Mr. Levy:

CLASSIFICATION - DP-181

The proposed publication "Extraction of Thorium Nitrate from Nitric Acid by TBP - 'Ultrasene'" by T. H. Siddall, III which was submitted to your office by letter of December 21, 1956 from Hood Worthington, has been reviewed from the point of view of classification.

It is recommended that DP-181 be submitted to the Declassification Branch for declassification under topic 1-204 of OD - DOC - 44. The pertinent limiting reference associated with topic 204 is 10-205 but this is not applicable to DP-181 in so far as the Savannah River Plant is concerned. However, we believe that the report should be referred to the Declassification Branch in order to ensure that none of the limiting references associated with 1-204 is applicable from the point of view of other AEC sites.

Very truly yours,

S. A. McKnight
Technical Assistant

SAMcK:EJC

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