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PROJECT 9536

File

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Route List

1. 1005
2.
3.
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5.

Date 10-7-44

Subject Waste Process Acid.

To S.J. Bugbee

From J.E. Sally

Copy No. [redacted] Bugbee.

BEFORE READING THIS DOCUMENT, SIGN AND DATE BELOW: ~~Attachments: 10-4-44 - 4533~~
~~10-4-44 - 4534~~ [redacted]

10/11	INV. 9-47
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DJ BROWN
4-4-02

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No. [redacted]

WAR DEPARTMENT
UNITED STATES ENGINEER OFFICE
HANFORD ENGINEER WORKS
P. O. BOX 550
PASCO, WASHINGTON

IN EIDMPC 410.2
REFER TO

7 October 1944

[redacted]

325

E. I. du Pont de Nemours & Co. [redacted]
Hanford Engineer Works
Richland, Washington

INV. 9-47

Attention: Mr. S. J. Bugbee

Gentlemen:

Further to the recent discussion between Madison Square Area officers and 300 Area personnel on the difficulties being experienced in the filtration of the precipitate formed by the neutralization of waste process acid, Captain Sturges has forwarded the enclosed reports on the precipitation of the peroxide from sulphate and flouride solutions, on the assumption that some of the factors evaluated therein may be of assistance in the solution of your problem.

He draws attention to the use of $Al_2(SO_4)_3$ in the ratio of about three times the quantity necessary to form the complex of AlF_6^{---} with the flouride ion present to give a peroxide precipitate of low flouride content. This method has been proven in plant scale operations.

Experience in the precipitation of sodium uranate from acid solutions indicates that different pH conditions may be required depending on the concentration of the flouride ion present; the presence of flouride ion makes it necessary to raise the pH to 10-10.5 to get complete precipitation of the uranium and to get a readily filterable product, whereas in the absence of flouride ion, essentially complete precipitation can be obtained at a pH of 6-7. However, even in the absence of flouride, there is some indication that a more readily filterable precipitate can be obtained at the higher pH.

In either case it is desirable to carry out the precipitation at 90-95°C adding the caustic very slowly rather than all at once, to agitate during the course of the precipitation and to digest the precipitate with agitation at 90-95°C for at least one hour prior to filtration. Of course, the concentration of uranium present in the acid solution and the concentration of the caustic used may have some effect on the physical nature of the precipitate, but it is believed that the general conditions outlined above will give the desired type of product.

For the Area Engineer:

J. F. SALLY
Major, Corps of Engineers
Chief of Production

2 Incls:
#1 - Memorandum and Notes
#2 - Report #A-1262

[redacted]

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DATE [illegible]
For The Atomic Energy Commission
Chief, Declassification Branch

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WAR DEPARTMENT
UNITED STATES ENGINEER OFFICE
HANFORD ENGINEER WORKS
P. O. BOX 890
PASCO, WASHINGTON

[redacted]

IN REPLY REFER TO EIDMPC 410.2

7 October 1944

E. I. du Pont de Nemours & Co.
Hanford Engineer Works
Richland, Washington

Attention: Mr. S. J. Hughes

Gentlemen:

Further to the recent discussion between Madison Square Area officers and 200 area personnel on the difficulties being experienced in the filtration of the precipitate formed by the neutralization of waste process acid, Captain Sturges has forwarded the enclosed reports on the precipitation of the peroxide from sulphate and flouride solutions, on the assumption that some of the factors evaluated therein may be of assistance in the solution of your problem.

He draws attention to the use of $Al_2(SO_4)_3$ in the ratio of about three times the quantity necessary to form the complex of AlF_6^{---} with the flouride ion present to give a peroxide precipitate of low flouride content. This method has been proven in plant scale operations.

Experience in the precipitation of sodium uranate from acid solutions indicates that different pH conditions may be required depending on the concentration of the flouride ion present; the presence of flouride ion makes it necessary to raise the pH to 10-10.5 to get complete precipitation of the uranium and to get a readily filterable product, whereas in the absence of flouride ion, essentially complete precipitation can be obtained at a pH of 6-7. However, even in the absence of flouride, there is some indication that a more readily filterable precipitate can be obtained at the higher pH.

In either case it is desirable to carry out the precipitation at 80-85°C adding the caustic very slowly rather than all at once, to agitate during the course of the precipitation and to digest the precipitate with agitation at 80-85°C for at least one hour prior to filtration. Of course, the concentration of uranium present in the acid solution and the concentration of the caustic used may have some effect on the physical nature of the precipitate, but it is believed that the general conditions outlined above will give the desired [illegible]

For the Area Engineer:

J. P. SALLY
Major, Corps of Engineers
Chief of Production

2 Incls:
#1 - Memorandum and Notes
#2 - Report #A-1082

[redacted]

CLASSIFICATION CANCELLED
DATE 4-24-[illegible]
For The Atomic Energy Commission
H. R. Carroll
Chief, Declassification Branch

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6-5-02

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Report A-1042

Metal Reclamation

MADISON SQUARE AREA
MANHATTAN DISTRICT
NEW YORK, N.Y.

17D1

THE PRECIPITATION OF URANIUM PEROXIDE FROM FLUORIDE SOLUTIONS

by

[illegible]ward J. King, Herbert M. Clark, T/4 H. R. [illegible]rownell, T/5 A. L. Grunewald, and T/5 E. G. Robles

(Sterling Chemistry Laboratory, Yale University)

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The purpose of these experiments was to find a way of reducing the fluoride content of the uranium peroxide precipitate obtained in the recovery of uranium from the bomb operation slags. The effect of the presence of ferric and aluminum ions on the fluoride content of the precipitate was investigated. A method of analysis for fluoride in the peroxide precipitates was developed.

I. The Analytical Method

by

H. M. Clark

The following analytical methods for fluoride were investigated: (a) titration with ferric chloride using thiocyanate as indicator; (b) titration with thorium nitrate using alizarin as indicator; (c) titration with aluminum chloride using methyl red as indicator. The aluminum chloride method seemed to be the most satisfactory, and was used for all analyses. Some of the advantages of aluminum chloride are treated by R. Geyer (1), in a general discussion of fluoride analysis.

The pH of the fluoride solution was adjusted before titration with aluminum chloride [illegible] the solution was just basic to methyl red. The solution became [illegible] methyl red at the completion of the reaction for the formation of AlF_6^{---}. It was found that the addition of sodium nitrate and ethyl alcohol produced a sharper end-point. The titration was carried out at room temperature. An N/4 aluminum chloride solution was standardized against pure sodium fluoride. Twenty grams of sodium nitrate, 25 ml of 95% ethyl [illegible] a few [illegible] of methyl red were added to a solution of sodium fluoride in [illegible] ml of water. After adjustment of the acidity, aluminum chloride was added until a reproducible indicator color change was obtained. A permanent color standard for reference was prepared by blending sodium dichromate and cobalt chloride solutions. The accuracy and precision [illegible] with the N/4 aluminum chloride solution were comparable with those

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[illegible] titrations involving solutions of similar concentration.

Before titration of unknowns, the fluoride content was quantitatively separated from the samples by distillation of hydrofluosilicic acid according to the method of Willard and Winter (2). The added step of the distillation caused some loss in precision and increased the volume of the solution before titration. At the same time a more dilute (N/16) solution of aluminum chloride was needed for the small fluoride samples encountered. Neutralization of the distillate with dilute sodium hydroxide was found to be a tedious process at room temperature, due to the low rate of reaction which resulted in only temporary methyl red end-points. Rapid adjustment of acidity was possible at about 50°C. The amounts of sodium nitrate and alcohol were increased when large volumes were necessary, and new color standards were prepared. End-points for the aluminum chloride titration were not quite so sharp, but results were still satisfactory in view of the size of the samples. Careful and consistent control of amounts of all reagents was required, of course, for reliable results.

II. The Experiments

Experiments I and II followed procedures suggested in a letter (EIDM 0-221-Z-MS) of May 23, 1944. The other experiments were devised in this laboratory.

Experiment I.

A solution was prepared which was 2.5 molar in magnesium sulfate, 0.13 molar in uranium sulfate, 2 molar in sulfuric acid, and 0.13 molar in sodium fluoride. This was heated to 50°C and 1.5 equivalents of calcium hydroxide was added for each liter of solution. The pH was then raised to 3.5 with 20% sodium hydroxide solution. One-half of this mixture was filtered immediately for Experiment I-A, while the remainder was kept at 50°C for 24 hours and then filtered for Experiment I-B. The fluoride contents of filtrates A and B were, respectively, 0.162 and 0.157 gram of fluorine per 100 ml. After standing for 10 hours at 50°C, samples of both filtrates were refiltered and analyzed. Both samples showed a 6.7% decrease in fluoride content. This may be attributed in part to slow precipitation of insoluble fluorides during the standing, and in part to attack of the glass vessels by the fluorides.

From other portions of filtrates A and B the uranium peroxide was precipitated at a pH between 3.0 and 3.5 in the presence of ammonium sulfate. Part of this precipitate was filtered off immediately; the remainder was allowed to stand for 10 hours at room temperature before filtering. The precipitates which were filtered off immediately were 0.82% and 0.42% fluorine for Experiments I-A and I-B respectively, whereas those which were filtered after standing were 1.93% and 0.61%. In the course of several hours or a day, white precipitates developed in all of the filtrates from the peroxide precipitates.

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Thus it is seen that the peroxide precipitates take up fluoride upon standing, and that better results are obtained when the insoluble fluorides of calcium and magnesium are given time to come out of solution before precipitation of the peroxide.

Experiment II.

The purpose of this experiment was to investigate the effect of the presence of ferric ions on the fluoride content of the precipitates. A solution was prepared which was 2.5 molar in magnesium sulfate, 0.13 molar in uranyl sulfate, and 0.0852 molar in sodium fluoride. This was heated to 50°C and its pH was raised to 3.5 with 20% sodium hydroxide solution. The resulting solution was divided into 3 parts; II-C, II-D, and II-E.

One sample of II-C was kept at 50°C for 10 hours. No precipitate developed. From another sample the peroxide was precipitated under the conditions described in Experiments I. That part of the precipitate which was filtered off immediately was 0.39% fluorine, while that which stood for 10 hours before filtration was 0.52% fluorine.

Solution II-D was treated with enough ferric sulfate to complex all of the fluoride as $FeF_6^{\equiv}$. A sample which stood for 10 Hours at 50°C developed a precipitate which was mainly ferric hydroxide. The peroxide precipitate was 0.34% fluorine if it was filtered off immediately, while if it stood for 10 hours it was 0.41% fluorine.

Solution II-E was treated with three times the stoichiometric amount of ferric sulfate necessary to complex the fluoride (in other words, the mole ratio of iron to fluorine was 1:2). The large amount of ferric hydroxide which formed was filtered off before proceeding. Another large precipitate of ferric hydroxide developed in a sample of the filtrate during 10 hours standing at 50°C. The uranium peroxide from a second portion of the filtrate was 0.46% fluorine if filtered off immediately, and 0.71% fluorine if filtered after 10 hours. A third portion of the filtrate was treated with sodium hydroxide until the pH rose to 5.1. The precipitate, which consisted largely of ferric hydroxide along with a small amount of uranium, was removed by filtration. A portion of the filtrate developed a slight precipitate during 10 hours standing at 50°C. The peroxide precipitate which stood for 10 hours was 0.50% fluorine; the one which was filtered immediately was lost.

These experiments show that the presence of ferric ion does not help to reduce the fluoride content of the peroxide. Apparently, the complex ferric fluoride ion is not sufficiently stable, and an [illegible] ferric ion cannot be maintained in a solution having a pH be[illegible] and 3.5. It is also evident that even in the absence of calcium, [illegible] peroxide precipitates pick up fluoride upon standing.

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Experiment III.

This experiment was an investigation of the effect of aluminum ions on the fluoride content of the peroxide. Aluminum was more promising than iron for two reasons; the complex aluminum fluoride ion (AlF_6^{---}) is more stable than the corresponding ferric complex, and aluminum hydroxide does not start to precipitate until the pH is about four.

The peroxide was precipitated from 125 ml portions of a solution which was 2.0 molar in magnesium sulfate, 0.10 molar in uranyl sulfate, and 0.762 molar in sodium fluoride. Each portion (except in III-G) also contained 10 grams of ammonium sulfate and varying amounts of aluminum sulfate. The results are given in Table I. The term "stoichiometric aluminum" means that quantity of aluminum ion or aluminum sulfate necessary to complex the fluoride as AlF_6^{---}.

Table I.

Expt. No.	Description	% F in ppt.
III-A	No aluminum	0.98
III-B	Stoichiometric aluminum	.60
III-C	2 x " "	.23
III-D	3 x " "	.08
III-E	6 x " "	.06
III-F	3 x " Iron	.75
III-G	3 x " aluminum and only 3 grams of ammonium sulfate	.10

Ten grams of ammonium sulfate correspond to about 3 grams of salt to 1 gram of uranium metal, but Experiment III-G shows that a reduction of the salt to [illegible] a one to one ratio as directed for Experiments I and II causes no significant change in the fluoride content of the precipitate. White precipitates developed in the filtrates from the peroxide precipitations of III-A, B, C, and F, that in III-F being relatively large. No precipitates were obtained in the filtrates of III-D, E, and G. This illustrates the effectiveness of excess aluminum in keeping fluoride in solution.

The peroxide precipitated in the presence of three times stoichiometric aluminum was analyzed for aluminum. Various macro and semi-micro methods gave negative results. The alizarin spot test described by Feigl (3) appeared to show the presence of several hundredths of a per cent probably associated with the fluoride in the precipitate.

The results of this experiment indicate that the optimum quantity of aluminum is three times the stoichiometric amount. The same amount of ferric ion, under the same conditions, is much less effective. As a matter of fact, a good deal of the iron precipitated as the hydroxide and had to be filtered off before the peroxide precipitation.

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Experiment IV.

This experiment was similar to III, but the peroxides stood for 10 hours before filtering. When no aluminum was present the precipitate was 0.30% fluorine, but in the presence of three-times the stoichiometric amount of aluminum the percentage was 0.12.

Experiments V and VI.

In order to determine the influence of the presence of calcium on the effectiveness of aluminum, Experiment I was repeated in a modified form. The preparation of the initial solution of magnesium sulfate, uranyl sulfate, sulfuric acid, and sodium fluoride, and the addition of calcium hydroxide followed the procedure described for Experiment I. One portion of the mixture was filtered for Experiment V; the remainder was kept at 50°C for 14 hours and then filtered for Experiment VI.

The filtrates were analyzed and found to contain 0.151 and 0.153 gram of fluorine per 100 ml for V and VI respectively. On the basis of these analyses aluminum sulfate was added to 200 ml aliquots of the filtrates to give the required aluminum to fluorine ratios. A white precipitate gradually formed in the filtrate of Experiment V before the addition of the aluminum sulfate. This was filtered off before proceeding. Six grams of ammonium sulfate were added to each solution, and uranium peroxide was precipitated as usual. The results are summarized in Table II.

Table II

Expt. No.	Mole ratio Al:F	Remarks	% F in ppt.
V-A	1:6	Ppt. filtered off immediately	0.41
V-B	1:6	Ppt. filtered off after 10 hrs.	2.32
V-C	3:6	Ppt. filtered off immediately	0.062
VI-A	3:6	Ppt. filtered off immediately	0.056
VI-B	3:6	Ppt. filtered off after 10 hrs.	0.056

The high result of V-B can probably be attributed to post-precipitation of calcium fluoride. It is seen that the presence of calcium in general aids in reducing the amount of fluoride in the precipitates.

III. Conclusions

The fluoride content of uranium peroxide precipitates can be reduced to 0.06% to 0.08% by precipitation of the peroxide in the presence of at least a three-fold excess of aluminum ion. It is desirable to allow the [illegible] obtained by adding calcium and magnesium liner materials to the retort discharge to stand for several hours in order to give time for slow precipitation of insoluble fluorides before filtration and subsequent precipitation of the peroxide.

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Literature Cited

(1) R. Geyer, Z. Anorg. Chem., [illegible], 42-55 (1943).
(2) Willard and Winter, Ind. Eng. Chem., Anal. Ed., 5, 7 ([illegible]).
(3) Feigl, Spot Tests, Nordemann Publishing Co., 1937, p. 116.

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PERURANIC ACID FROM URANYL SULFATE SOLUTION

Principle and Reactions:

Uranyl sulfate solution is heated to 40°C. ([illegible] deg.) and is reacted at that temperature with 27.5 - 30% hydrogen peroxide. [illegible] acid is precipitated. Complete precipitation is assured by [illegible] caustic soda solution to a PH of 2.8 - 3.5. The precipitate is [illegible] by filtration or centrifuging depending on the type of crystal obtained. It is purified from water soluble impurities by washing with distilled water. The reactions involved may be graphically presented as follows:

$$(1)\quad XO_2SO_4 + H_2O_2 \xrightarrow{H_2O} XO_4 \cdot 2H_2O + H_2SO_4$$

$$(2)\quad H_2SO_4 + 2NaOH \longrightarrow Na_2SO_4 + H_2O$$

Yield = 99.6 - 100% of theory (based on metal content).

Discussion

Tolerable Impurities

The above precipitation has been successfully effected on sulfate solutions containing the following metals, [illegible] in solution: calcium, magnesium, iron, sodium, lithium, boron, [illegible], mercury, lanthanum, chlorides and fluorides. The approximate amounts of these impurities observed to be tolerable to date are:

	Pts/pt. Uranium metal
Calcium	0.0084
Fluoride	0.0060
Magnesium	0.2900
Iron	0.0150
Mercury	0.0200
Sodium	0.0100
Lithium	0.0200
[redacted]	0.0400
[redacted]	0.0200

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	Ppm/gm. Uranium Metal
Lithium	0.0800
Chloride	0.0100
Lanthanum	0.0200

Variations in Concentration of Uranium Metal

Successful precipitations have been made from solutions containing from 3.5 - 10.0% of uranium metal.

Effect of Variations in Temperature on Crystal Size and Settling Rate of Peruranic Acid

The effect of temperature upon crystal grouth of the peruranic acid obtained from the recovery of slag and dross (containing calcium, magnesium, fluorides and iron as impurities as indicated above) is shown in the following prints (752 diameters) which show crystal structure for precipitations at (I) 5 - 10°C., (II) 20 - 25°C., (III) 30 - 35°C., and (IV) 40 - 45°C. Each small division on the scale represents 1 micron. (See note 1)

All types could be filtered on a Buchner Funnel in the laboratory. Type (I) filtered slowly, however, and required return of filtrates because they came through milky. Types II, III and IV filtered rapidly and showed rapid settling rates in centrifuge operations. Type I showed a very poor settling rate.

Sodium uranate dissolved in sulfuric acid does not, however, show a similar crystal grouth as indicated above. Fine circular particles less than 1 micron in diameter were obtained at temperatures from 5° to 45° C. These particles were difficult to filter, and had very poor settling rates, which improved, however, to a slight extent as the precipitating temperature ascended because of aglomeration of the crystals.

Addition of magnesium sulfate to the sulfate solution of sodium uranate, however, gave needles of Types II and III upon precipitation at 40° - 45°C. These crystals filtered good by gravity, settled rapidly and gave good results in the centrifuge, although they did not filter well on a Buchner Funnel. Further study of addition agents other than $MgSO_4$ to improve crystal formation and filtering properties, is in progress. (See note 2)

Quality of Peruranic Acid Produced

(Recovered from slag and dross)
(1000 g. sample $XO_4 \cdot 2H_2O$)

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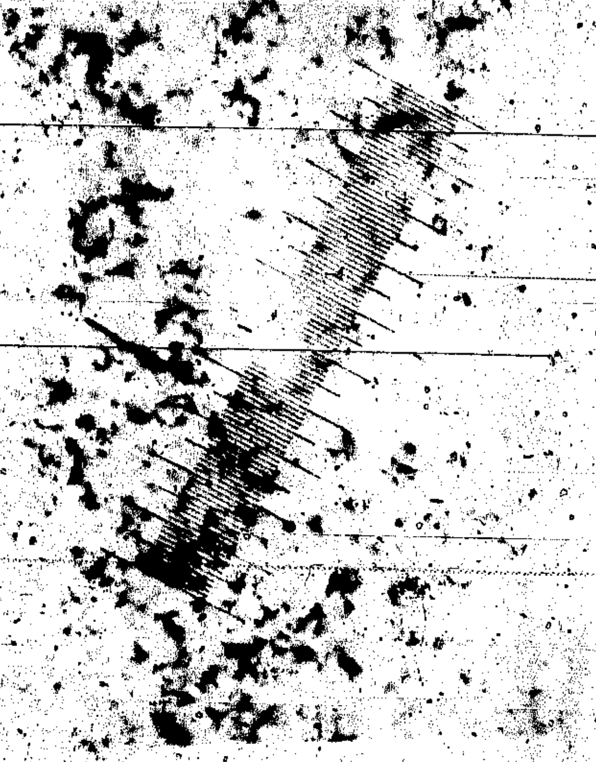
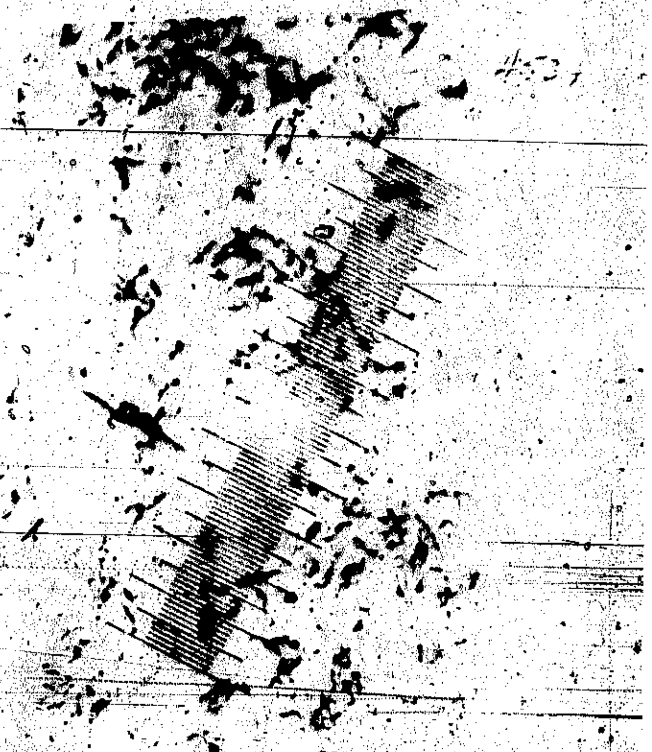
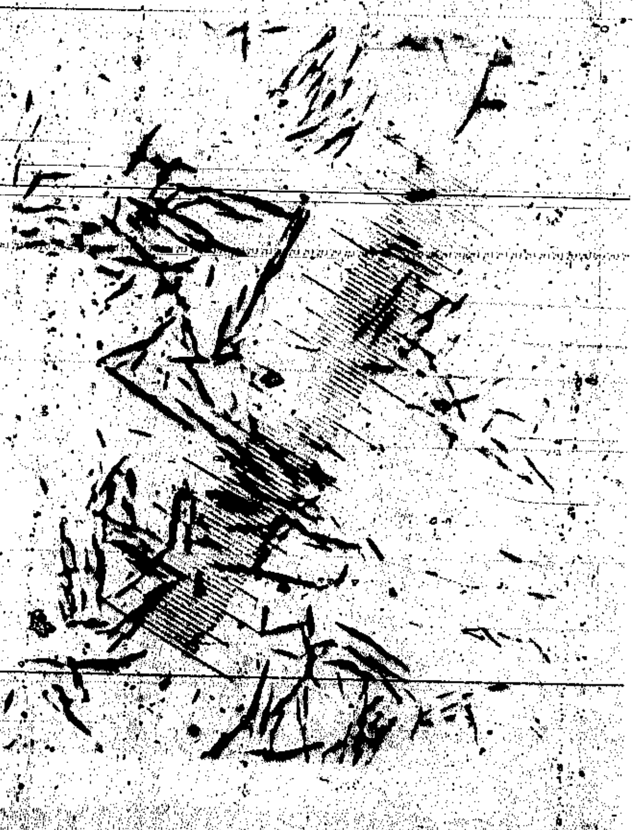


I - 5-10°C.



II - 20-25°C.

III - 30-35°C.



IV - 40-45°C.

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Rare earths	Not detected on 100 g. sample
Chlorides	Less than 0.001
Heavy metals	0.002
Boron	Not detected
S	0.057
Nitric insoluble	0.009
Si	0.001
Fe	0.002

Spectrographic Analysis

- - Not detected	(p.p.m. indicated)	
Ag V.W	Cd - -	Li - -
As - -	Co L 10	Mg W
Au - -	Cr - -	Mn - -
B - -	C L 10	Mo - -
Bi - -	Ge - -	N L 50
Be - -	Fe V.W	Na Weak
Ca 2.5	In - -	Pb L 50
Sb - -	Si L 100	Tl - -
U - -	Zn - -	

The above quality is better than required for D-1 specification on Black Oxide.

Adjustment of Sulfate Solution Prior to Reaction with Hydrogen Peroxide

Excess of sulfuric acid which may be employed for dissolving uranium bearing materials should be neutralised at 90 - 95° C. with finely powdered calcium carbonate to a pH of 3.0 - 3.5. Calcium sulfate is precipitated. This is separated by filtration. Prior to pH adjustment, however, any four valent uranium present should be oxidized to the six valent form by bubbling with air, or by addition of hydrogen peroxide. This valence change takes place with a change in the color of the solution from green to yellow. + Sodium uranate, and limited amounts of caustic soda have also been employed for this pH adjustment without detrimental results. This is done to avied precipitation of basic uranium (IV) sulfate to the pH employed.

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[illegible] [redacted] [redacted]

Rate of Addition of Reagents, and Concentration of Reagents.

No detailed study has been made on rates of addition and concentration of reagents. The hydrogen peroxide has been generally added over a period of 1/2 to 1-hour. The caustic soda has been added over a period of 1-1/2 to 2-1/2 hours. The rate of addition is adjusted to the speed of agitation and the strength of caustic soda solution employed to avoid local precipitation of impurities such as magnesium or iron. Concentrations of hydrogen peroxide from 10 - 30% strength, and caustic soda from 5-50% strength have been employed without any appreciable variations in results.

A 20 - 30% excess of hydrogen peroxide over theory requirements has been usually employed.

Reaction Controls Employed.

Excess of hydrogen peroxide has been determined by spot test with KI starch papers. A blue coloration is obtained when excess of hydrogen peroxide is present. pH determinations have been made by spot with Congo Red papers (range 3.0 - 5.0) and check with a Beckman pH meter. Completeness of precipitation has been determined by spot tests with potassium ferrocyanide - a brown coloration is noted if the reaction is incomplete - in which latter case the reaction mass is stirred longer, and more hydrogen peroxide is added if test with KI starch papers indicates that this is necessary. When the reaction is complete, this test invariably gives a blue rim because of iron present in the liquors.

Procedure:

Heat 250 cc of uranyl sulfate solution pH 3.0 - 3.5 (6.68 g. 100% uranium per 100 cc). (= 16.7 g. uranium 0.07 mol M.W. = 238) to 40° C. Agitate and add dropwise 11 cc 27.5% H_2O_2 (3.0 g. 100% H_2O_2, M.W. = 34 = 0.088 mole) over a period of 30 - 45 minutes. Agitate for 3/4 - 1 hour. The pH of the reaction mass drops to 1.45 - 1.50. Raise the pH to 2.8 - 3.5 by dropwise addition of 32% by weight caustic soda (11 - 12 cc are usually required). This should be done slowly (time 1 - 2½ hours) with good and rapid agitation to avoid any local basification which will precipitate such impurities as iron and magnesium. The reaction temperature is held at 40 - 45° C. until the precipitation is complete as indicated by spot test with potassium ferrocyanide. Agitation for 3/4 - 1 hour is usually necessary after all of the caustic soda has been added, the pH is adjusted, and the excess of hydrogen peroxide is assured. Adjustment of pH can be made with

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[illegible] if too much alkali has been added. The peruranic acid [illegible] as a creamy yellow colored body. The product is isolated by [illegible] filtration or centrifuging after cooling to 30 - 35° C. * The wet paste is washed with distilled water until sulfate free.

Volume filtrate = 220 cc   Uranium content = nil
Volume of wash waters = 500 - 1000 cc
Weight of wet peruranic acid cake = 60 gm.
Weight dry peruranic acid (dried at 75-80°C. - 24 hours)
= 24.1 g. (69.4% uranium)
Weight 100% $UO_4.2H_2O$ (70.4% U) = 23.7 g. or 100% of theory

* In small scale experiments the reaction mass was filtered without cooling. On large scale operation, it may be desirable to cool to 30°C. to avoid decomposition or solution of the peruranic acid due to loss of hydrogen peroxide on prolonged holding at 45°C. while isolating the material.

Final page - Memo. on Prepn. of
Peruranic Acid from Uranyl Sulfate Solns.

Louis Spiegler,
Jackson Laboratory
4-30-43 - ahs

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NOTES ON MEMORANDUM ON PREPARATION OF PERURANIC ACID FROM URANYL SULFATE SOLUTIONS

Note 1: Later work indicated that prolonged heating at 60-95°C showed no appreciable improvement in growth of crystals already formed.

Note 2: The addition of 75-125 parts of ammonium sulfate per 100 parts of uranium in solution was later found to be most desirable. With this preparation of ammonium sulfate, crystals of peroxide formed slowly giving fast settling precipitates and granular dense press cakes.