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STATUS OF ANALYTICAL PROCEDURES FOR REDOX PRODUCTION PLANT NO. 1

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Summary of Redox Analytical Meetings,
August 30, 31 and September 1, 1949

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Prepared by

G. B. Barton and R. J. Sloat

September 20, 1949

TECHNICAL DIVISIONS
GENERAL ELECTRIC COMPANY
HANFORD WORKS

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STATUS OF ANALYTICAL PROCEDURES FOR REDOX PRODUCTION PLANT NO. 1

Summary of Redox Analytical Meetings, August 30, 31 and September 1, 1949

Present:

Knolls Atomic Power Laboratory

J. F. Flagg

H.W. Analytical Section (Metallurgy and Control)

D. W. Pearce, H. R. Schmidt, T. W. Hauff,
G. B. Barton, R. J. Hale, D. F. Shepard,
J. W. Hall, and W. W. Marshall

H.W. Chemical Research Section (Separations Technology)

F. W. Albaugh, C. M. Slansky

H.W. Redox Development Section (Separations Technology)

R. B. Richards, F. W. Woodfield, R. J. Sloat

I. INTRODUCTION

In order to evaluate the overall status of the Redox Analytical development program in terms of recently firmed-up requirements for Production Plant No. 1 a group discussion was held with the above in attendance. As a basis for discussion, the analytical requirements for Redox Production Plant No. 1 (HW-14142) had been submitted previously by the Redox Development Section to all interested parties. The main purpose of the meetings as stated initially by T. W. Hauff and R. B. Richards was to survey these requirements in light of existing or projected analytical procedures and reach agreement relative to the following:

1. Methods presently satisfactory for production plant application.
2. Methods satisfying accuracy requirements but needing some adaptation for "hot" application.
3. Methods needing minor refinements to attain accuracy requirements and which may or may not need adapting for hot usage.
4. Methods requiring moderate to substantial development to meet accuracy requirements for either hot or cold application.

II. SUMMARY AND CONCLUSIONS

Following a review of the Redox analytical requirements it was felt by those present that the development studies conducted to date at

[redacted]

H.W. and K.A.P.L. have resulted in satisfactory solution for nearly all the analytical problems, and that the work remaining consists primarily of 1) adapting certain satisfactory methods for "hot" application, 2) an evaluation of some already tested methods which are inherently more simple and rapid, 3) refinements to a few existing methods to place them within the accuracy limitations, and 4) moderate development for a few components whose methods do not permit the desired accuracy.

It was generally agreed that a major portion of the analytical research and development work on Redox analytical methods has been completed and that the work remaining is of such a magnitude that it can be accomplished well ahead of the scheduled plant start-up (ca. July 1951). The status of Redox methods as indicated by the discussions was such that, in the opinion of the Redox Development Section, monitoring of a production unit could be attempted very shortly with good control of the key components and satisfactory monitoring of mechanical performance.

By way of presenting an abbreviated outline of the present status of analytical methods, the above-stated purposes of the meeting are treated briefly below in terms of accomplishments and remaining work. Further details are presented in the Discussion.

1. Methods generally satisfactory for immediate application to production control are as follows:

Can Now be Employed "Hot"

Uranium (fluorimeter)

Density (falling drop for aqueous, and gravitometer for hexone samples)

HNO_3 (greater than 0.05 M)

Pu (all valence states)-except for accuracy in IAW, IIAW, IIIAW, Plutonium Concentrate and Rework Adjustment Tank.

pH

Gross beta and gamma

$SO_4^=$

Need be "Cold" Only

Solvent impurities (MIBC, mesityl oxide, organic acids, reducing normality, dinitroisobutane, methyl isopropyl diketone, oxidizing normaltiy)

Spectrographic analysis, final products.

All requested aqueous makeup analyses:

Density

HNO_3 (IBX)

ANN (IBX)

Na

Can Now be Employed "Hot"	Need be "Cold" Only
	Cr (IAS supply and IIAS Supply)
	Fe (30% $Fe(H_2NSO_3)_2$, IIDS and IIIDS Supply, IBX Supply)
	H_2NSO_3H (same as Fe)
	$NaNO_3$ (IIDS and IIIDS Supply)
	Normal H_2O assay

2. Satisfactory methods requiring further adaptation for hot usuage consist of the following:

Fe^{+++}

$Al(NO_3)_3 \cdot 9H_2O$

Uranium (Chromous reduction-ferric oxidation)

Uranium (Polarograph)

Sodium (Flame Photometer)

Cr (all valence states)

$PO_4^{\equiv}$ (in the absence of SiO_2)

3. Methods requiring only further refinement to meet the stipulated accuracy requirements consist of the following:

Hot Methods	Cold Methods
HNO_3 (acid deficient system) -0.3 to + 0.05 M	HNO_3 (acid deficient system)
Pu in IAW (above 0.001 g.Pu/l.; i.e. 1% Pu loss)	
Fission Products (Ru,Cs,Ce,Zr,Cb)*,I**	
Americium	
Curium	

---

* Accuracy limitation to be determined. **Probably not essential to operation of plant.

4. Determinations requiring further development of existing methods or development of new methods to meet accuracy requirements, and requiring adaptation to routine control consist of the following:

   Pu in IAW (below 0.001 g.Pu/l.; i.e., 1% Pu loss).

   $PO_4^{\equiv}$ and Si mixtures.

   Silica (in the absence of PO_4^{---}; see Discussion)

   Pu on Superfiltrol

   Neptunium (accuracy limitation to be determined)

For certain analytical methods currently meeting the accuracy requirements, it was agreed that greater economy would be realized by the use of less involved and more rapid substitutes. Accordingly, a rapid evaluation will be made of the data (some of which are now on hand) showing the feasibility of carrying out the following substitutions.

1. The use of pH for HNO_3 determinations.

2. The use of hydrometers instead of pycnometers for Sp.Gr. of cold solutions.

3. The use of density relationships for the determination of uranium.
   Note: Much of the basic data relative to the above have already been accumulated. Such data combined with a few appropriate experimental studies should rapidly indicate the feasibility of these methods.

The X-ray photometer was discussed as another means for shortening the analytical time for uranium. However, much development work would be required to adapt this instrument for "hot" use. In the event that the density method for estimating uranium should prove satisfactory it is the opinion of the Redox Development Section that the need for a short-cut method would be satisfied.

Although it is recognized that existing analytical methods for fission products (Ru, Zr, Ce, Cb, Cs, I) are unusually long, (generally 2 to 6 hours) the expected frequency of routine analyses for control of these components in the production plant is low. In the opinion of the Redox Development Section adequate control of plant operation should be possible with the present methods for fission products, with moderate refinements to be expected within the next two years before the plant starts up. Although a long range analytical development program for determination of individual fission products may well be justified on the basis of overall plant requirements, such a program is not considered necessary on the basis of the Redox production plant.

Further details relative to the exact breakdown of future work are discussed below.

III. DISCUSSION

Each analysis requested in HW-14142 was examined and the following conclusions resulted:

Density. The existing falling drop, gravitometer, and pycnometer methods can be adapted for use in the production plant. Since the pycnometer utilizes relatively large sample volumes (100 microliters or greater) it could not be used for hot (IAF, IAW, etc.) determinations unless adequate shielding and manipulating devices were provided. K.A.P.L. is to determine whether a hydrometer can be used for many of the density analyses. Both Hanford Works and Schenectady agreed that a laboratory assistant could determine approximately 10 to 15 samples per shift (falling drop method). Gravitometer determinations require about 30 minutes each while pycnometer determinations require about 15 minutes if run as a group, or about 25 to 30 minutes for a single sample.

Since the gravitometer and falling-drop methods have been demonstrated primarily at sites other than H.W., these methods must be written up and tested at H.W. under routine control laboratory conditions.

Uranium. The Jones reductor-ceric titration (for cold samples) and chromous sulfate-ferric titration methods (at present the latter is being scaled down for hot sample determinations) should be satisfactory for high concentration uranium streams. Present experience with the X-ray photometer indicates its applicability to most high uranium concentration streams. Additional development work on shielding, manipulating devices, and decreased cell size would be necessary to adapt the X-ray photometer method to hot sample work. Many of the high uranium concentrations may be calculated within the required limits from density equations. The Redox Development Section will determine those analyses which can be calculated from equations and incorporate the results in the revised issue of HW-14142. It is possible that calculation of uranium concentrations from density equations may satisfy the need for a short-cut method for uranium determinations in certain process streams.

The fluorimeter method for low UNH concentrations is satisfactory for "hot" solutions, such as IAW, up to approximately 2 g.UNH/l., and may possibly be extended up to approximately 5 g./l.

Although the polarograph is satisfactory for the range 0.2 to 10 g.UNH/l., the current method requires a 1 ml. sample and further work is required either to scale-down the sample size or to develop adequate shielding and manipulating devices for "hot" samples.

Nitric Acid. In the forthcoming revision of HW-14142, required accuracies for HNO_3 analyses will be revised to specify ± 3 grams HNO_3/liter for solutions from -0.3 M to + 0.05 M and ± 2 grams/liter for greater positive HNO_3 concentrations. Accuracies for aqueous makeup solutions are to remain as originally requested.

Using present analytical methods the ± 2 grams/liter variation for positive HNO_3 concentrations can be obtained, but additional refinements will be required to obtain the ± 3 grams/liter for the "acid-deficient" to approximately neutral samples. K.A.P.L. has recently developed a new method for determination of acid-deficient HNO_3. This new method will be tested further by both K.A.P.L. and H.W.

Hanford Works is to investigate the possibility of using pH measurements for obtaining nitric acid concentrations. Complete charts on the relationship between pH and free HNO_3 in process solutions have been obtained on a research basis but they remain to be checked for precision and accuracy and application to routine use.

The Redox Development Section is to reevaluate the need for each HNO_3 analysis. The results of this reevaluation are to appear on the reissue of HW-14142.

Aluminum Nitrate. The present acidimetric titration method should prove adaptable for all requested ANN analyses. Since the method at present requires approximately 0.1 ml. of sample, additional effort must be spent to reduce the required sample size for hot solutions or to design equipment with adequate shielding and manipulating devices.

Chromium (VI) and Total Chromium. Present methods are satisfactory for all requested samples except that the method must be scaled-down to use micro sample sizes for "hot" streams.

Ferrous Iron and Sulfamic Acid. Present methods are satisfactory.

Gross Beta and Gamma. The Shonka (ionization) chamber should be used for all gamma determinations. Present beta methods should prove satisfactory.

Other Chemical Analyses. Until further information is available concerning the allowable "trace component" concentrations in the Plutonium Concentrate and Decontaminated UNH Storage it was decided to remove the "overall accuracy" requirements for the trace components. It appears feasible to use spectrographic methods now available, or already under development for other processes, for determinations of these components.

At present no method for determining the clarity of hot solutions has been developed. Since the requirements for the analysis have not been definitely fixed, it was decided to defer further development for at least a year.

Mesityl oxide and oxidizing normality should be added to the determinations requested for the organic sampler and treatment tank. Present methods should prove adequate for all the requested determinations of hexone impurities, although a small amount of work will be required to adapt the methods to routine control.

The determination of sodium, sulfate, and phosphate all appear feasible by present methods. Although the literature indicates that high phosphate concentrations interfere with the accurate determination of sodium, no quantitative data on the magnitude of this interference for any given mole ratio of sodium/phosphate appear to be available. It will be necessary to determine, for a Na/PO_4 mole ratio of 160 or greater expected in the ISF (HW No. 2 Flowsheet), whether phosphate causes a significant interference with the sodium assay. Use of the flame photometer for sodium determinations on hot samples will require development work on equipment and materials handling methods. Requests for $PO_4^{\equiv}$ will be deleted from the Rework Adjustment Tank analyses on the reissue of HW-14142 but $PO_4^{\equiv}$ will still be requested in the ISF Feed Makeup Tank and Underground Waste samples.

The necessity for routine silica determination was discussed and it was decided that this requirement will depend on further process development.

Plutonium. Discussion of the current status of plutonium determinations brought out the following points:

(1) It is desirable to determine plutonium alone rather than total alpha activity for those streams containing appreciable quantities of americium and curium (IAF, IAW, ISF, and ISW).

(2) The TTA method offers the advantage of determining only plutonium (exclusive of americium and curium) and at the same time gives sample disks for counting which are much lower in beta and gamma activity than disks resulting from either the LaF_3 or hydroxide precipitation methods.

(3) Increasing the g./T. above the present level increases the proportions of Am and Cm very rapidly. These elements follow Pu in the LaF_3 analysis thus distorting the plutonium distribution picture.

(4) Increasing the g./T. level will also increase the ratio of Pu^{240} to Pu^{239}. Due to the low Pu concentration and high gross radioactivity in dissolver solution, the only available analytical method for Pu in the IAF stream is by radio assay (counting alpha-radiation). The final plutonium product (plutonium concentrate) will be analyzed for Pu by chemical assay. Since Pu^{240} is a more active alpha-emitter than Pu^{239}, the factors for converting from the initial radio assays for Pu to the final chemical assays will change as the ratio of Pu^{240}/Pu^{239} increases with increasing g./T. Further development work is

required to extend the conversion factors (i.e., between radio and chemical assays) to a higher g./T. level, keeping in step with increases in g./T. level for pile operation.

(5) Present analytical methods for plutonium are not as accurate as desired for accountability purposes, but accuracies required for accountability have not been firmly stated. Without firm accuracy and precision goals, it is not possible to decide how much development work on plutonium assay methods is justified in order to improve the accuracy of accountability data.

(6) While analytical accuracies required for plutonium accountability purposes have not been stated firmly, it is possible to estimate the accuracies required for monitoring plutonium extraction in the production plant to ensure that accumulated waste losses do not average more than 1% of the plutonium production, and to permit material balances for plutonium to be within 98 to 102%. The ± 2% accuracy stipulated in document HW-14142 for the more concentrated plutonium streams (dissolver solution, IAP, IBP, 2BP, Pu Concentrate) may be slightly better than has been demonstrated by existing methods (± 2.3% precision, 98.5% recovery). However it is believed that the existing methods, with minor improvements to be expected in the two years before plant startup, will be suitable for plant control and plutonium-balance purposes. Cross-checking of the TTA method by H.W. and the hydroxide precipitation method by K.A.P.L. will be undertaken in the near future. Accuracies required for plutonium in the waste streams are discussed below.

(7) Some discussion of Pu (IV) polymer and its distribution brought out that it would go to waste in IAW and its rapid and accurate determination would require a new procedure. An approximate method for determining Pu (IV) polymer involving determination of "unextractable" Pu (IV) has been used by O.R.N.L. and presumably could be relied upon on a non-routine basis for any necessary plant assistance studies in starting up the plant. In view of present process uncertainties regarding the need for a routine method, it is considered that no analytical development work on an assay for Pu (IV) polymer need be undertaken for at least a year, pending new information on the properties of Pu (IV) polymer now being studies by process research chemists at K.A.P.L.

It was decided that plutonium analytical requirements should be specified on a g./l. basis and discussed further before a decision was reached as to the suitability of presently available methods. The following table lists the accuracies (for total Pu assays) considered necessary in order to operate the plant at or below the Flowsheet Pu losses.

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Sampling Point	Estimated Plutonium Concentrations g./l.	Limits g./l.
Centrifuge slurry hold tank	0.0007	± 0.0003
IAW	up to 0.001	± 0.0001
	0.001 to 0.005	± 0.0005
	0.005 to 0.03	± 0.001
ISU	0.00003	± 0.0001
ISW	0.0005	± 0.0003
IBU	0.00004	± 0.00002
ICU	0.0001	± 0.00005
IIDW	0.0002	± 0.0001
IIIEU Conc. Sampler and Decontaminated UNH Storage	1 part in 10^8 parts of uranium	± 5 parts in 10^9 parts of uranium
IIAW and IIIAW	0.0004	± 0.0002
IIBW	0.00009	± 0.0001
IIIBW	0.0001	± 0.0001
Pu condensate receiver	0.000005	± 2.5 x 10^{-6}
IHP	up to 0.0003	± 0.00015
	0.003	± 0.001
IIW	0.000005	± 0.000005
Waste Concentrate	0.0004	± 0.0003
Rework Adjustment	0.0004	± 0.0003

Present methods for assaying plutonium are considered satisfactory to meet the limits set for all of the above streams except IAW, IIAW, IIIAW, Waste Concentrate, and Rework Adjustment. In these waste streams the high aluminum concentration is expected to interfere with determining the extremely low concentrations of plutonium (i.e., low number of alpha counts) necessitating further refinement of methods.

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Some development is necessary to demonstrate a satisfactory assay method for plutonium absorbed on the Superfiltrol scavenging agent.

The existing TTA and "Hydroxide" methods are to be compared at Hanford Works and K.A.P.L. to decide which method is the preferred one for each Redox stream. The TTA method may prove more satisfactory for those streams containing americium and curium (IAF, IAW, ISF, ISW) while the "Hydroxide" method may prove more satisfactory for other Pu determinations.

Fission Products. Decontamination in the various columns of the plant will be followed by gross beta and gamma measurements. When the desired decontamination is not achieved, then analyses for specific fission products will be requested. The accuracy and limits of the presently available methods are not sufficiently well known and require further investigation. Additional work is desirable to develop more rapid methods. During the next year an effort will be made to state the accuracy required for assaying the various fission products in the process streams. As a first approximation, it is expected that determining the concentrations of individual fission products within a factor of 2 will be satisfactory.

The present electrolytic and distillation methods for Ru should prove satisfactory although time-consuming. The presence of cesium, as a major component of the beta activity, would be an indicator of misoperation of the columns. Hence development of an assay method for Cs is desirable.

Allowable gross beta and gross gamma radioactivities due to fission products in recovered uranium product, as set up by H.I., could be determined analytically to within ± 100%. The present H.I. specifications for allowable curies of gross beta and gross gamma radioactivities per gram of recovered uranium are based on approximately 2 m.e.v. beta radiation and 0.8 m.e.v. gamma radiation. Residual fission products in recovered (Redox process) uranium are expected to be primarily 43-day Ru^{103}, 330-day Ru^{106}, 65-day Zr^{95}, and 35-day Cb^{95}. As a first rough approximation, on the order of 10 to 20% of the beta-curies due to these fission products are expected to be 2 m.e.v. or stronger, with the balance ranging primarily from 0.2 to 1 m.e.v. On the order of 80% of the beta-curies due to these fission products are expected to be in the range from approximately 0.5 to 0.8 m.e.v., with a smaller fraction as low as approximately 0.2 m.e.v. While the present H.I. specifications represent a satisfactory base line, it is considered desirable to expand these specifications taking into account the actual energy spectra expected from the residual fission products.

An approximate method for the determination of radio-iodine is available at H.W., although the precision and accuracy attainable from the method have not been established. Since the analysis will probably not be used unless the presence of iodine in feed solutions proves objectionable during the initial period of plant operation, no development work on this method is considered necessary for Redox at present.

Further Discussion:

Data were presented for comparison of results obtained at K.A.P.L. and Hanford Works on a group of standard samples. Good agreement was found on all analyses. These data will be published later in a report.

Before the final session J. F. Flagg and H. R. Schmidt drew up a list of the problems which appeared to require further work (list is appended as Exhibit A). Indicated on Exhibit A is a brief discussion of the more important analytical methods requiring further refinement or development, and an assignment of this further work between K.A.P.L. and H.W.

The question was brought up as to whether or not the need for mass spectroscopy had been considered with relation to the program. It was pointed out that if the U product were sold to Oak Ridge for 235 separation then isotope accountability comparable to present plutonium accountability would become necessary. U^{235} can be determined by fission counting of raw uranium to within 1 part in 70, while by mass spectroscopy it can be determined to within 1 part in 700. It was also brought out that two instruments would be needed for a given determination and range. Should isotope distribution in Pu become more important and require mass-spectographic determination, additional mass spectrographs (to be used only for plutonium and never for uranium) will be needed to avoid analytical inaccuracies caused by cross-contamination with uranium in the determination of isotope distribution in Pu. The cost was presented as about $100,000 for an initial set-up of two instruments.

Other minor changes (deletions, changes of accuracy requirements, etc.) which will have no significant effect on the analytical development program will be incorporated in the reissue of HW-14142.

G B Barton
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ANALYTICAL SECTION

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REDOX DEVELOPMENT SECTI

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EXHIBIT A

PROBLEMS TO BE INVESTIGATED

1. The use of pH for HNO_3 determinations (H.W.)

    a) Evaluate the feasibility of rapidly determining HNO_3 concentrations within the desired accuracies from pH measurements.

2. Use of hydrometers for Sp.Gr. (K.A.P.L.)

    a) Determine whether hydrometers may be used on "cold" streams for rapid determinations of specific gravity within the required accuracies.

3. Use of Sp.Gr. for UNH

    a) Falling drop (H.W. and K.A.P.L.)

    b) Gravitometer (K.A.P.L.)

    c. Determine precisions and accuracies of these methods to enable calculation of the accuracies with which UNH concentrations may be calculated from specific gravity data for concentrated uranium streams.

4. Uranium in IAW

    a) Fluorimeter (K.A.P.L. and H.W.)

        1. Satisfactory for "hot" IAW samples up to ca. 2 g.UNH/l. Further testing may increase this range up to ca. 5 g./l.

    b) Polarograph (H.W.)

        1. Presently satisfactory in the range 0.2 to 10 g.UNH/l., except for adaptation to "hot" samples.

5. X-ray photometer study (H.W.)

    a) Develop a time-saving method for UNH in "hot", high uranium solutions. Need for this study for Redox solutions would be minimized if density provides an adequate quick method for high UNH.

6. Spectroscopic analysis of finished uranium and plutonium (H.W.)

    a) Apply spectroscopic techniques (currently used for impurities in uranium metal) to finished (Redox process) uranium and plutonium to determine the suitability of this method for plant control.

7. Plutonium

   a) TTA and Hydroxide methods (K.A.P.L. and H.W.)

      1. Cross-check the two methods between the two sites.

   b) Determine Pu absorbed on Superfiltrol.

      1. Requires development of a method.

8. Fission Products

   a) Zirconium - to be completed as soon as possible (est. ca. 2 mos.).

      1. Mandelic acid (H.W.)

      2. TTA (K.A.P.L.)

      3. Complete the current evaluations of each of the above methods and decide which is the better of the two.

   b) Radioactive iodine (H.W.).

      1. Re-examine present method.

   c) Ru, Ce, Cb, Cs - to be considered after Zr work (K.A.P.L. and H.W.).

      1. Evaluate precisions and accuracies of existing methods.

9. Neptunium (K.A.P.L.)

   a) Develop a satisfactory method.

10. Americium and Curium (H.W. Chemical Research Section)

   a) The Chemical Research Section is currently studying the analyses of $BiPO_4$ wastes for Am and Cm.

11. Standards

   a) Hanford Works to prepare and distribute IAF and IAW standards which will contain Pu. Only total Pu will be determined.

12. Sodium (H.W.)

   a) Develop flame photometer for "hot" samples.

13. <u>Silicon (H.W.)</u>

a) Evaluate silico-molybdate method against fluoride distillation method as a standard. Investigate interference from $PO_4^{\equiv}$ in the concentrations expected in Redox solutions.

14. <u>PO_4^{---}, SO_4^{--} (H.W.)</u>

a) Evaluate present methods.