DECLASSIFIED
WITH DELETIONS

DOCUMENT NO. HW-62091DEL

MASTER

SERIES AND COPY NO.

DATE _____

October 19, 1959

GENERAL ELECTRIC

HANFORD ATOMIC PRODUCTS OPERATION - RICHLAND, WASHINGTON

X

RESTRICTED DATA

THIS DOCUMENT CONTAINS RESTRICTED DATA AS
DEFINED IN THE ATOMIC ENERGY ACT OF 1954.
ITS TRANSMITTAL OR THE DISCLOSURE OF ITS
CONTENTS IN ANY MANNER TO AN UNAUTHORIZED
PERSON IS PROHIBITED.

TITLE

PLUTONIUM OXALATE DISK FILTER AND FILTER
MEDIA STUDIES

7

OTHER OFFICIAL CLASSIFIED INFORMATION

THIS MATERIAL CONTAINS INFORMATION AFFECTING THE NATIONAL DEFENSE OF THE UNITED STATES WITHIN THE MEANING OF THE ESPIONAGE LAWS, TITLE 18, U. S. C., SECS. 793 AND 794, THE TRANSMISSION OR REVELATION OF WHICH IN ANY MANNER TO AN UNAUTHORIZED PERSON IS PROHIBITED BY LAW.

AUTHOR

G. Rey

ISSUING OFFICE:

THIS DOCUMENT MUST NOT BE LEFT UNATTENDED OR WHERE AN UNAUTHORIZED PERSON MAY HAVE ACCESS TO IT. WHEN NOT IN USE, IT MUST BE STORED IN AN APPROVED LOCKED REPOSITORY WITHIN AN APPROVED GUARDED AREA. WHILE IT IS YOUR POSSESSION AND UNTIL YOU HAVE OBTAINED A SIGNED RECEIPT FROM CLASSIFIED FILES, IT IS YOUR RESPONSIBILITY TO KEEP IT AND ITS CONTENTS WITHIN THE LIMITS OF THIS PROJECT AND FROM ANY UNAUTHORIZED PERSON. ITS TRANSMITTAL TO, AND STORAGE AT YOUR PLACE OF RESIDENCE IS PROHIBITED. IT IS NOT TO BE DUPLICATED. IF ADDITIONAL COPIES ARE REQUIRED, OBTAIN THEM FROM THE RELATED ISSUING FILE. ALL PERSONS READING THIS DOCUMENT ARE REQUESTED TO SIGN IN THE SPACE PROVIDED BELOW.

ROUTE TO:

PAYROLL NO.

LOCATION

FILE	ROUTE	DATE
------	-------	------

SIGNATURE AND DATE

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

DECLASSIFIED
WITH DELETIONS

105-001

105-001

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

HANFORD ATOMIC PRODUCTS OPERATION
RICHLAND, WASHINGTON

NOTICE!

This report was prepared for use within General Electric Company in the course of work under Atomic Energy Commission Contract W-31-109-Eng-52, and any views or opinions expressed in the report are those of the authors only. This report is subject to revision upon collection of additional data.

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission to the extent that such employee or contractor prepares, handles or distributes, or provides access to, any information pursuant to his employment or contract with the Commission.

105-002

SECRET

DECLASSIFIED WITH DELETIONS

HW-62091 **DE2**

This document classified
by R. J. Sloat

This document consists of
27 pages. No. 4 of

27 copies. Series A

PLUTONIUM OXALATE DISK FILTER AND FILTER MEDIA STUDIES

By

George Rey

Process Equipment Development
Chemical Development Operation
Chemical Research and Development
HANFORD LABORATORIES OPERATION

Classification Cancelled (Change to
DECLASSIFIED WITH DELETIONS
.....)

By Authority of C. L. Robinson,

Head Class. Officer,

By W. L. Rains 9-27-61

October 19, 1959

HANFORD ATOMIC PRODUCTS OPERATION

RICHLAND, WASHINGTON

GENERAL ELECTRIC

RESTRICTED DATA

This document contains restricted data as
defined in the Atomic Energy Act of 1954.
transmittal or the disclosure of its con-
tents in any manner to any unauthorized
person is prohibited.

DECLASSIFIED WITH DELETIONS

105-003

SECRET

DECLASSIFIED
WITH DELETIONS

DO NOT PHOTOSTAT

DISTRIBUTION

1. R. E. Burns
2. M. H. Curtis
3. O. F. Hill
4. H. H. Hopkins, Jr.
5. J. W. Kolb
6. E. E. Kurtz
7. D. W. Pearce
8. A. M. Platt
9. W. H. Reas
10. L. C. Schwendiman
11. R. J. Sloat
12. R. E. Smith
13. E. O. Swain
14. L. D. Turner
15. L. L. Zahn
16. G. Rey
17. 300 Files
18. Record Center
- 19-21. Extras

DECLASSIFIED
WITH DELETIONS

SECRET
DECLASSIFIED

-3-

HW-62091

PLUTONIUM OXALATE DISK FILTER AND FILTER MEDIA STUDIES
WITH DELETIONS

INTRODUCTION

Presently rotary drum filters are being used for plutonium oxalate slurry filtration in the 234-5 Building. Changing of the filter cloth used on a rotary drum is a time consuming operation which involves ever increasing radiation exposure to maintenance personnel. Consequently, studies were conducted in the 321 Building on a disk type filter which could be adapted for simple and quick ("one-nut") replacement of the filter medium.

In addition to the tests required to demonstrate the basic operability of the disk type filter, tests were made on several types of filter media. The media tested included rigid porous materials (e.g. porous alumina plate) which could be used only on the disk filter, and various types of filter cloths which could be used with either the disk or the present drum type filters.

In the 321 Building testing it was not possible to use plutonium oxalate. Cerium oxalate and mixtures of filter aid and talc were used as stand-in slurries.

SUMMARY AND CONCLUSIONS

Tests of the 12 inch diameter disk type filter [redacted] have demonstrated the feasibility of the disk type filter for plutonium oxalate slurry filtration. Although the disk filter used in the tests was designed to accommodate a large variety of filter media and not designed for optimum "change" cycles, rigid porous plates could be changed in 3 to 5 minutes. With design improvements it is estimated that media changes could be made in approximately two minutes.

[redacted]

A "scalpel" technique was studied as a possible method of (1) prolonging "blowback" cycles and (2) evening out the delivery of filtered oxalate to subsequent processing steps. The scalpel travels across the face of the filter disk much like a needle crossing a phonograph record. As it passes slowly across the disk face it produces a slit in the filter precoat. With the increased permeability created in the slit, most of the oxalate filters at the groove and builds up for subsequent removal by the doctor knife (see Figure 12). Employing this technique it was found possible to increase the period of continuous operation a factor of two or more before blowback is required. Thus, after establishing a precoat, it may be possible for production to operate continuously for 16 hours or more without using the manually actuated "blowback". In addition the cake discharge rates are much more uniform than those obtained when sections of the drum or disk are blown at suitable intervals to maintain filtering capacity. The scalpel technique involves the continuous delivery of a small increment of cake to subsequent processing, while the sector blowback system involves the delivery of a large "gob" of cake once each revolution of the filter. A relatively uniform and continuous flow of oxalate cake may eliminate many of the processing problems (e.g., unreacted cake, plugging) currently encountered in the calciner-fluorinator complex.

105 -004

DECLASSIFIED
WITH DELETIONS

SECRET

DECLASSIFIED
WITH DELETIONS

Several filter media fulfill the requirements for use on plutonium oxalate slurries. Of the media tested, rigid porous aluminum oxide appears best suited for disk filter operation. It is rigid enough to be held in place and sealed against the filter face with a single center bolt. Another medium, Rigimesh⁽¹⁾ (316 SS) also possesses excellent properties. This medium, however, would be more readily suited for drum filter application. Other media found acceptable were: (1) a KEL-F⁽²⁾ plate, (2) a Carborundum⁽³⁾ plate, (3) a Dynel⁽⁴⁾ cloth, (4) a Dynel felt, (5) a Dacron⁽⁵⁾ felt, and (6) a polyethylene cloth. Of the media tested the Dynel cloth presently in use on the production drum filters had the highest corrosion rate in a nitric-oxalic acid solution (5.8 percent weight loss in 40 days).

Cloudiness was observed in most of the first samples of filtrate taken during filtration tests. However the solids in the first samples accounted for <0.2 per cent of the feeds solids for all media found acceptable. This waste loss drops off very rapidly as the precoat builds up on the filter.

EQUIPMENT

General - A 12-inch diameter (85 sq in. filtering area) disk filter was designed and fabricated for testing.

scaled up in capacity

The disk can be easily while maintaining critically

safe dimensions (i.e. 15 inch diameter). It is conceivable that a disk filter could be constructed to allow "quick" changing of the filter medium. This would result in less exposure to radiation by maintenance personnel now involved in time consuming drum filter cloth changes, and is the prime reason for investigating the disk filter for plutonium oxalate filtration.

Plutonium oxalate is prepared and filtered in the 234-5 Building as shown in Figure 1. The operation involves the precipitation (strike) of plutonium oxalate from a plutonium nitrate-nitric acid solution with oxalic acid. This is done continuously by the addition of both streams at a constant rate into an agitated vessel (precipitator) which is allowed to overflow. The overflow slurry from the precipitator is fed into a filter tank (slurry pan) in which the drum filter rotates partly submerged. Here the slurry is separated into the solid (cake) and liquid (filtrate) components. As the drum rotates and filters, filtrate is continuously discharged to a receiver. Cake is picked up on the drum, washed, dried and discharged to a screw calciner for further processing. The filtration is accomplished using vacuum supplied to the drum filters from the building vacuum header through the filtration receiver to the drum interior. Auxiliaries required for the entire operation include: (1) a cake wash nozzle, (2) cake removing scraper (doctor knife), (3) a drive for rotating the drum and (4) a slurry pan agitator (wig-wag) to keep the heavy precipitate from settling out in the pan.

- (1) Reg. Trademark for the sintered wire cloth manufactured by the Pall Corporation
- (2) Reg. Trademark for the polychlorotrifluoroethylene manufactured by M.W. Kellog Co.
- (3) Reg. Trademark for the SiCarbide refractory manufactured by the Carborundum Co.
- (4) Reg. Trademark for the acrylic fiber manufactured by the DuPont Co.
- (5) Reg. Trademark for the polyester fiber manufactured by the DuPont Co.

DECLASSIFIED
WITH DELETIONS

DECLASSIFIED

-5-

HW-62091

WITH DELETIONS

The basic difference between the disk and drum filter is the location of the filter media with respect to a horizontal rotating axis. The medium on the disk is located perpendicular to the rotating axis while on the drum it is parallel and concentric to the axis. Both types require a rotating axis (shaft).

Disk Filter - The three major components of the disk filter unit were: (1) the slurry pan, (2) the disk proper, and (3) the slide valve assembly. Within the slurry pan the disk proper rotates partly submerged, while vacuum is applied to the rotating disk from a stationary slide valve.

As shown in Figure 2, the face of a 3/4-inch thick steel disk was sectioned into 12 filtering compartments (sectors). Each sector was provided with its own vacuum supply (sector tube) for removing filtrate and wash solution. The tubes passed through and were attached to a four inch pipe shaft. The shaft-disk assembly was rotated by a bicycle chain drive attached to a variable speed motor. The disk face was constructed such that rigid or semi-rigid filter media could be accommodated. Rigid media, such as a porous aluminum oxide plates were held in place and sealed against the disk with a single center bolt. Nonrigid media, such as filter cloths, were held in place by the center bolt and sealed against the outer ridge by the use of a snug fitting rim which was fastened to the disk using four screws. A backing screen was used with the cloths to provide additional support and to prevent their buckling into the sectors.

In order to allow the disk to rotate freely and still maintain the disk sectors under vacuum, the slide valve assembly shown in Figure 3 was used. This consisted of a wear plate which rotates against a stationary slide valve. The wear plate houses the sector tubes from the disk sectors and the slide valve has connected to it the filtering and drying vacuum and air backflush (blowback). The plate and valve faces were closely fit to form a seal. With the seal, vacuum could be transmitted to one portion of the filter disk and pressure could be simultaneously transmitted to another portion. The valve had three slots to which the filtrate suction line, a wash outlet, and air inlet were connected. Consequently as the disk and wear plate rotate together each sector tube aligns once per revolution with each of the three stationary slots in the valve.

Equipment Assembly - Figure 4 is a photograph of the assembly of equipment used to test the disk filter and various media. A slurry was prepared continuously in the precipitator and allowed to overflow into the slurry pan. The slurry used (generally a powder and water mixture described under stand-in materials) was made by feeding dry powder from a hopper via a screw conveyor to the precipitator. Water was also metered into the precipitator. The feed produced was introduced to the slurry pan below the doctor knife. Filtrate was sucked through the disk by vacuum applied to the filtrate catch tank. The disk was rotated by a variable speed drive connected through a bicycle chain and sprocket system to the filter shaft. To facilitate the installation of the filter assembly and allow sufficient working room a long filter shaft was used. The pressure drop across the filter disk was continuously recorded. The filtrate line had a sampler. Provisions were made for testing cake washing and blowback mechanisms.

FILTER OPERATION

General - Both the drum and disk filter can be operated in an identical manner. The present method of operating the production drum filters can be classified

105 - 006

DECLASSIFIED
WITH DELETIONS

SECRET

DECLASSIFIED

-6-

HW-62091

WITH DELETIONS

as a modified precoat operation. The operation consists of: (1) setting the doctor knife at some previously established optimum distance from the filter drum (1/4-inch is presently used), (2) allowing the cake to build up on the drum until it reaches the knife (this is a precoat), and (3) scraping away the excess cake as product. The precoat so established serves as a buffer layer of cake between the media and knife and also does the actual filtering job shortly after it is established. However after several hours of such operation the precoat becomes plugged (blind) because of the penetration and "void filling" of the precoat by the smaller particles in the slurry. Consequently, in order to maintain the desired filtering rate it becomes necessary to regenerate the filter periodically. Presently this is done by 234-5 Building operations in two ways: (1) removing all the plugged precoat and starting over or (2) removing a portion (one section) at a time and doing so frequently. The removal of the precoat is accomplished by backflushing with air (blowback) a section at a time as the blow slot in the slide valve aligns with a section tube. Both types of filter regeneration require operator attention and are referred to later as the "long" or "short" cycle filter operation.

It becomes apparent that it would be beneficial to lengthen the "long" or "short" cycle if it could be done simply and economically. Also, and perhaps more important, it would be better for subsequent processing equipment (calciners, etc.) if the flow of cake from the filter was continuous and not bumpy as produced during "short" cycle operation where the freshly regenerated section of drum does most of the filtering (picking up and discharging a large "gob" of cake) each revolution. A device called a scalpel (described in detail in section Disk Continuous Operation) was made and tested for possible application for either the drum or disk filter. Use of the scalpel extends the "long" cycle time and supplies an even and continuous cake to the calciner.

Regardless of the method of operation, after several days the filter media itself becomes plugged. When this occurs it becomes necessary to change the medium, thus another cycle (media "change" cycle) is introduced into the over-all operation of the filter. With the drum filter this operation at present requires up to one hour to accomplish, with as many as two maintenance men required.

Test Filter Operation - The disk filter was tested using both the "long" and "short" cycle method of operation described above. Evaluation of the filtering characteristics of various media was made "on the disk" operating on a "long" cycle. After testing the various media the most promising one was selected for further testing which included some 30 hours of "short" cycle operation. Figure 5 shows the disk filter in operation during a typical run.

STAND-IN MATERIALS

General - Because of the 321 Building limitations it was not possible to use plutonium oxalate for testing the disk filter and the various media. Instead two slurries, one a powder-water mixture, and the other a precipitated cerium oxalate, were used. The use of the cerium precipitate was limited to two short observation runs because of the expense of the material and safety hazards involved in handling the acidic solutions necessary for its preparation. The powder-water slurry on the other hand was easily handled, prepared, and was used to evaluate the various media by comparing their filtering characteristics against similar studies with a standard media. The standard used was a Dynel cloth which is presently in use on the production drum filters.

105 -007

DECLASSIFIED
WITH DELETIONS

Cerium Oxalate - Cerium oxalate when precipitated like plutonium oxalate is known to be a good stand-in for plutonium oxalate. Figure 6 is a flow diagram showing the strike conditions used to prepare the cerium oxalate. For reasons mentioned previously the use of cerium was held to a minimum. Also, evaluation of the various filter media would have been very difficult using the cerium oxalate because of post precipitation (precipitation after filtering). Also, a more elaborate wig-wag would have been required to maintain the heavy oxalate well agitated in the slurry pan.

Powder-Water Slurry - A slurry made from Dicalite⁽¹⁾ 4200 and talc powders (3 parts by weight of Dicalite to 1 part talc) was used for most of the testing. The filter cake obtained from this slurry visually resembled the plutonium oxalate cake except for color. Particle size distributions for the powder mixture and plutonium oxalate have been determined and are shown in Figure 7. The distributions show the mass mean particle diameter of the powder to be less by a factor of two than the oxalate. Thus the absolute values of the rate of solids penetration (waste losses) through the various media tested (Figure 9) can be considered to be greater than those which would be encountered if plutonium oxalate had been used for the tests.

Assuming a precoat composed of rigid spherical particles of uniform diameters, it is theoretically impossible for the precoat to blind when it is used to filter like material. The porosity of the precoat will remain constant. When the particles are not of uniform diameter, as generally is the case, the precoat will blind when used as the filter medium. The finer particles being filtered will penetrate into the precoat, decreasing its porosity, until the precoat is blind. Nonuniformity of particle size may therefore be considered as an index of how quickly a precoat layer would blind, in the method of precoat operation used in the 234-5 Building. From Figure 7 it can be noted that the plutonium oxalate particles are more uniform than the powder slurry used. To what degree this affects the interpretation of the data obtained can only be estimated. We know from the production line that "long" cycles run about 8 hours, and this compares to a 5 hour "long" cycle obtained on the same cloth using the disk. Assuming the disk and drum filter have similar filtering characteristics this would indicate that "long" and "short" cycle data obtained in the tests should be multiplied by approximately a factor of 1.6 to estimate the cycle performance of the disk filter operation in filtering plutonium oxalate.

FILTER MEDIA

Properties - The properties of the various filter media considered for testing are listed in Table I of the Appendix. Some of the media were weeded out as unsuitable after visual inspection. The Dynel cloth D-2005 (standard) was used as a standard for evaluating the properties of other media. This media is presently used for plutonium oxalate service.

Corrosion and Radiation Damage - Table II summarizes the corrosion and radiation tests made. For the corrosion test media specimens were suspended in a liter of 4M HNO₃ - 0.1M oxalic acid solution. The solution was at ambient temperature and continuously agitated for forty days. The radiation damage tests were made in the 234-5 development lab. Specimens were suspended in a 0.42 M Pu(NO₃)₄ solution for 60 days.

(1) Reg. Trademark for the Silicious filter aid manufactured by the Great Lakes Carbon Corporation

DECLASSIFIED -8-

HW-62091

The standard cloth was found to have the highest corrosion rate of all the media tested. Dynel fabrics were discolored from a light cream color to a rich reddish-brown color (plutonium oxalate has a similar color). The Rigimesh wire cloth had a low corrosion rate, considering its wire size (2 mil wire). Kel-F had no detectible corrosion rate.

Radiation damage was not apparent on any of the media subjected to the plutonium nitrate solution. However the test was limited to visual inspection and handling only.

Pore Size Distribution - Pore size distribution data are summarized in Figure 8. An Aminco-Winslow Porosimeter was used to make the determinations. From the distributions, volumetric mean pore diameters may be obtained. Mean diameters can be used to select media grades for cake filtration. A rule of thumb is to use a medium with a mean pore diameter two to three times larger than the mass mean particle diameter of the solids to be filtered. The Aloxite⁽¹⁾ medium (Grade 2, 36 μ mean pore diameter) was found to be effective (>99.8 per cent efficient) in filtering the powder slurry (17 μ mass mean particle diameter). Considering the value of plutonium oxalate (mass mean diameter 31 μ) selection of a medium on the basis of mean pore size should be made using a mean pore to mean particle diameter ratio of about 2. Thus media with mean pore diameters 60 μ should give effective filtration of the oxalate provided the largest pore does not exceed 90 μ (the largest pore opening can be easily and accurately determined by a bubble point test). In the 234-5 laboratory the oxalate was readily filtered using a Grade 5 Aloxite specimen (45 μ mean pore diameter). The filtrate obtained from the test was clear.

Rate of Particle Penetration (Waste Loss) - Waste loss data was obtained for ten media. The runs were made by testing virgin media under conditions as nearly identical as possible using the "long" cycle mode of operation. The operating conditions were: (1) Disk speed = 0.75 rpm, (2) Slurry rate = 12 l/hr, (3) Full vacuum applied to filter, (4) Wig-wag speed = 40 cycles/min, and (5) a Precoat thickness of 1/4 inch. Run start time was at time of first cake pickup by disk. At least three samples of filtrate were taken at time intervals before maximum pressure drop was achieved. Filtrate samples were filtered through millipore membranes, dried and weighed for solids collected. Samples of the slurry in the make up tank were taken before and after each run. The slurry solids averaged 85 g/l ($\pm$ 10 per cent).

Five media are considered superior to the standard cloth in their relative particle removal performance. Four media were not as good. The data is shown in Figure 9. The media have been assigned performance values (PF) in relation to the standard cloth. Plus values are factors of performance better than, and minus values worse than the standard. A PF of +2 means the waste losses are 1/2 those of the standard cloth. A -2 PF means the waste loss is twice as much as the standard. The values were evaluated using the 1 hour data.

Precoat Blinding Rate - Filter media characteristics affect the rate at which the precoat blinds. This is shown by the data of Figure 10. The data was obtained simultaneously along with waste loss determinations on the virgin media. The time to achieve 20 inches Hg pressure drop for the various media was different. The time difference was as high as a factor of 6 or more. In general the more efficient the medium the quicker the precoat blinds.

(1) Reg. Trademark for the alumina ceramic manufactured by the Carborundum Co.

DECLASSIFIED
WITH DELETIONS

SECRET

DECLASSIFIED

-9-

HW-62091

WITH DELETIONS

The slightest disturbances (sampling, etc.) were enough to give discontinuances in the data. In addition all conditions were not exactly identical from run to run. The data, therefore, should be considered crude, and its use limited.

Critical Pressure - A critical pressure, as applied to cake filtration, is a pressure drop which causes a condition of reconsolidation within the cake. Reconsolidation occurs with the collapse of the particle bridges across the pores of a medium. The strength of a bridge depends upon the characteristics of the particles, (their shape, size, etc.), on the surface of the medium. In addition many of the operating variables also affect this condition. Generally the collapse of a particle bridge across a pore plugs it, and aids in blinding the medium. The determination of the plutonium oxalate critical pressure can only be made after a medium has been selected for use, operating procedure established, and the determination made with plutonium oxalate. Use of critical pressure data is somewhat arbitrary. Generally it is accepted as the upper limit of pressure drop which should be used in the filtering operation for maximum utilization of the power expended (i.e. higher filtering rate per unit of pressure drop), however in many cases this is not practical nor necessary.

Figure 10 shows critical pressures (different for all media) for the media tested. The critical pressure is where the precoat blinding rate curves change in slope. Only one critical pressure is noted for each medium, except the two rigid media, (Aloxite, and Carborundum) which have two critical pressures. The dotted lines can be attributed to reconsolidation effects caused by disturbances such as sampling or a vacuum fluctuation.

Filter Media Blinding - Media replacement requirements ("change" cycles) depend on the rate at which the medium blinds under operating conditions. Assuming identical operating conditions, blinding rate determinations for various media can be made. However, this requires extended periods of operation which may exceed 100 hours in some cases. Since an important variable in media blinding is the compatibility of the particles with the pores, media blinding was not investigated thoroughly. In other words an exact stand-in material would have to be used to obtain useful data on filter media blinding.

A brief media blinding rate determination was made on the Aloxite (Grade 2) medium using the powder slurry. The data are shown in Figure 11. Pressure drops across the cleaned medium were obtained using tap water, after short periods (5-7 hours) of "short" cycle operation. At the end of each period the medium was cleaned by sluicing the precoat off the medium and emptying the trough of slurry. The trough was then continuously flooded with tap water with the liquid level in the pan maintained at the overflow. The pressure drop across the medium was then measured using full vacuum with the filter operating. The data shows that media blinding is a continuous process and can be determined. For the slurry and media used, best estimates are that the Aloxite medium would not require replacement until after at least 100 hours of operation.

Thick media such as the Aloxite (1/2 inches thick) may have a large holdup of solids within it after blinding. After 40 hours of operating time the Aloxite plate was removed and broken apart. Microscopic examination of the pores showed that particle penetration was about 1/16 of an inch. Replaced thick media would therefore contain essentially the same amount of plutonium oxalate as thinner media such as cloths.

DECLASSIFIED
WITH DELETIONS

105 - 010

DECLASSIFIED

-10-

HW-62091

WITH DELETIONS

The Cerium Oxalate Test - Two visual observation runs were made using a cerium oxalate precipitate. The precipitate was prepared according to the flowsheet shown in Figure 6, which is similar to the plutonium oxalate strike. The two most promising media (Aloxite and Rigimesh) were selected for testing. The cerium filtered more readily and with higher efficiency ($< .01$ per cent initial waste loss using the Aloxite Grade 2) than the filter aid-talc mixture. Post precipitation in the filtrate samples collected was observed shortly after sampling. The heavier cerium slurry jammed the wig-wag agitator after 3 to 4 hours of operation. Because of this it was not possible to obtain "long" cycle data on the cerium precipitate. Also observed was a greater tendency for the oxalate to adhere to both media.

MISCELLANEOUS TESTS AND OBSERVATIONS

Feed Entry - Classification of the feed slurry, either by insufficient agitation in the slurry pan, or by cascading the feed over the disk face, results in acceleration of the rate of precoat blinding. Classification occurs when the fine and course particles of a slurry are not homogeneously distributed in the solution. A filter precoat exposed to the finer fraction before filtering the courser fraction will experience greater penetration of the fines into it, then when filtering the course fraction or a homogeneous mixture of both, first.

An optimum location was found for introducing the slurry feed to the slurry pan. To minimize the effects of classification the wig-wag was operated at maximum speed (40 cycles/min). In addition the feed was introduced to the pan at the slurry overflow level, opposite the overflow, under the doctor knife, and directed parallel to the disk face. These conditions allowed filtration of a homogeneous slurry.

When feed was cascaded on the disk face blinding of the precoat layer occurred approximately twice as fast as the preferred method discussed above. With cascade operation the cake was distributed haphazardly (like frozen wax dripping), and was nonuniform in cake quality and resistance (from inner to outer edge). A slurry cascading across a filter medium experiences classification. The fines are the first to filter and the course material, the last.

Cake Removal - Physical disturbances of the precoat by a cake removing device (doctor knife) results in accelerating precoat blinding. This has been shown by the 234-5 Building production line personnel. They use a half hollow ground knife edge to minimize precoat disturbances. Cake disturbance results in reconsolidation (discussed previously).

Tests made on two knives with different edge angles and angles of cut did not show any readily apparent effects on the precoat blinding rate over 3 to 5 hours of operation. Further investigations were not made since the present drum filter knife design appears to be optimum.

Cake Quality (Dryness) - Cake quality in filter operation is used to describe the handling properties of the cake. Thus "wet" cake is difficult to remove from the filter with a doctor knife because of its pastiness. "Dry" cake on the other hand is crumbly and easily removed. The moisture content of the cake is also used to determine heat load requirement for subsequent equipment where further drying occurs.

DECLASSIFIED
WITH DELETIONS

105 - 011

DECLASSIFIED
WITH DELETIONS

-11-

HW-62091

Since the cake dryness is dependent to a large degree upon the characteristics of the material, only one determination was made on the powder slurry. Cake removed from one of the runs was oven-dried and found to have 54 percent moisture on a wet basis. This compares to 30 to 50 percent moisture (wet basis) for plutonium oxalate cake with similar handling properties. The high moisture content of the stand-in cake can be attributed to the hygroscopic nature of the powder (diatomaceous earth and talc) and its much larger surface area/unit of weight than plutonium oxalate.

Cake Wash - Cake washing was not used during the reported runs. However, methods of applying a suitable cake wash were investigated. Dripping wash on to the disk cake was not feasible because the cake is in a vertical position as compared to a horizontal position for drum filters. Deflecting the drippings was very inefficient (<50 per cent). The use of the smallest spray available was adequate but the minimum rate was four times greater than required. The spray combined with a cyclic timer (1 second on, three off), resulted in lowering the cake wash volume to 2 l/hr and adequately (>95 per cent) wet the cake.

Filter Media Replacement - The disk media were quickly changed under non-SWP conditions. The disk however, was not especially designed to accommodate any one type of filter medium. Cloths were changed in 6 to 8 minutes, semi-rigid media in 4 to 6 minutes and rigid media in 3 to 5 minutes. Shorter disk replacement times would result with a disk designed for one specific type of medium.

Variable Area Filter - Run observations showed that at startup and after regeneration, the disk acts as a variable area filter. Since the filter medium is in a vertical plane complete submergence of the filtering medium, during a revolution, cannot be attained unless the slurry in the pan is up to the center of the disk (up to the overflow level). Consequently a low slurry level exposes only the outer portion of the disk to the slurry for filtering. Thus only the filter area that can be submerged during a revolution of the disk is used to maintain capacity. As the filter plugs (loses capacity) the slurry level rises and more filter area is put into play. On the other hand the drum filter exposes all the media to the slurry during a revolution, regardless of the slurry level (submergence).

The effects of the variable area feature upon filter operation were not investigated, however it is evident that after regeneration the disk filter would not "suck the pan dry" to the extreme that the drum filter does.

CONTINUOUS OPERATION

General - When the precoat blinds it must be removed, or doctored in some manner to maintain filtering capacity. Removal of the precoat is done in the 234-5 Building by using the blowback either frequently (about every 45 minutes) on a section of the drum, or less frequently (about once per shift) on all twelve sections at once. These two methods have previously been defined as the "short" and "long" cycles.

During "short" cycle operation the eleven other sections, not blown, containing blind precoat, do little of the filtering during each revolution of the drum. Thus the freshly regenerated section must, and does, essentially all the filtering during each revolution, until it also becomes blind. This produces unequal distribution of cake over the drum surface to the extent that about 80 per cent of the cake discharged during a revolution comes from one section in 1/12 the time it takes to make the revolution. This results in intermittent feed to the

105 -012

DECLASSIFIED
WITH DELETIONS

SECRET

DECLASSIFIED
WITH DELETIONS

-12-

HW-62091

screw calciner. Large chunks of cake so dumped have jammed the screw. Consequently the practice is to manually break them up until they are small enough to be handled by the screw.

The "long" cycle method is less prone to discharge large chunks of cake, rather the cake is discharged evenly but at a slowly decreasing rate until it cannot keep up with the slurry feed. The draw back to this method of operation is that the filter requires complete regeneration which may require a short time off-the-line to accomplish. However if the long cycle could be lengthened appreciably it could possibly be the more desirable mode of operating.

Scalpel Technique - A scouting run was made where the "long" cycle operation was extended by a factor of two or more using a device called a scalpel. The scalpel was a sharp edged instrument like the small blade of a pocket knife. Only the tip of the scalpel was employed to make a slit 1/8-inch deep into the blind precoat. The result was a large increase in filtering capacity in the area of the slit. A continuous ridge of cake (see Figure 12) was produced, and continuously discharged. The scalpel was moved across the face of the disk, and its traveling speed accelerated, as required, to maintain filtering capacity. Radial movement of the scalpel produced a spiral formation of cake on the disk. The slit made by the scalpel increases the average permeability of the blind precoat and delays the necessity for complete blind precoat removal. The test was conducted using the standard cloth, and a slurry feed rate of 24 l/hr (twice the rate used in all other tests). The "long" cycle time with the scalpel employed was identical (5 hrs) to a run made without the scalpel at a 12 l/hr feed rate. Thus twice as much slurry was filtered in the same amount of time. This is a factor of at least two better since the cycle time is proportional to the square of the volume of slurry filtered. Estimates show that the present drum filter, or future disk filter, employing this device can operate with average long cycles of about 16 hours (presently the average drum "long" cycle is 8 hours).

This device would occupy little hood room and can be programmed to operate automatically.

Disk Continuous Operation - At the completion of the shorter evaluation tests made on the media, the most promising medium (Aloxite, grade 2) was selected for prolonged testing. Figure 13 shows the compilation of the total operating time gathered on the medium (40 hours), consisting of one "long" cycle run and six "short" cycle runs. The data show average "short" cycle periods of about 25 minutes for the powder slurry compared to 5 1/2 hours for the first "long" cycle period. Also shown is the clean water pressure drop obtained after each run (this was previously described under filter media blinding, see Figure 11). Also observed was the ability of the medium to filter the cerium oxalate effectively. The shutdown and regeneration after runs 15a through 15e were not necessary, thus the disk filter could have operated indefinitely with the continued use of the "short" cycle blowback.

The successful demonstration of the blowback on the disk indicates that a complete seal between filter sectors is not necessary. Sufficient seal between sectors is required to obtain an instantaneous positive pressure in the sector. With the porous aluminum disk, successful backflushing was achieved even though a portion of the backflushing air leaked laterally through the filter into adjacent sectors.

DECLASSIFIED
WITH DELETIONS

105 -013

TABLE I - PROPERTIES OF FILTER MEDIA CONSIDERED

Filter Media	Material	Cost(\$) Approximate Per Disk	Mean Pore Size(μ)	Porosity Est. Percent	Vendor	Remarks
Aloxite	Al ₂ O ₃	5.00	45 ⁺ 35 ⁺	25-30	Carborundum Corp.	Grade 5 Grade 2
Carborundum	SiC	6.00	42 ⁺	20-25		Grade 1
Alumdum	Al ₂ O ₃	9.00	6 ⁺	25	Norton	Medium Grade
Carbon	Carbon	15.00	27 ⁺	50	National Carbon Co	Activated Carbon
320-DY-V	Dynel	1.00	11 ⁺	50	American Felt Company	Twenty Micron Fibers
AFCO-62-CY-24	Felt	to	~50	>70		
320-DA-V	Dacron		>5	40		
320-DA-B	Felt	5.00	>5	40		
AFCO-62-DA-24			~50	>70		
Rigimesh	316 SS	83.00	45 ⁺	25-30	Pall Filtration Companies	Grade M (2 mil wire)
Sintered SS		35.00	10	50		Grade G
Porous Plastic	Kel-F	55.00	27 ⁺	30-40		1/8 inch thick disc
Multibraid	316 SS	8.50	~100	20-45	Multi-Metal Co	Grade XXD-400 (7 mil wire)
G-201-C	Glass	0.50	35 ⁺	50-70	National	Multi-Filament
G-206-C	Cloth	to	35 ⁺			
D-2004	Dynel	1.00	15 ⁺	40-60	Filter	
D-2005	Cloth		18 ⁺			
X-10059 FB	Linear	↓	>40	60-70	Media Corp.	Mono-Filament
X-10063 FB	Polyethylene		33 ⁺	50-60		
P-2201	Cloth		58 ⁺	25		
Selas	Al ₂ O ₃ - Si	15.00	40	30	Selas Corp.	

+ Determined

DECLASSIFIED
WITH DELETIONS

-13-

HM-62091

105-014

DECLASSIFIED
WITH DELETIONS

DECLASSIFIED
WITH DELETIONS

TABLE II - SUMMARY OF FORTY DAY CORROSION TEST

<u>Filter Media</u>	<u>Material</u>	<u>Per Cent Wt. Loss</u>	<u>Remarks</u>
D-2005 ^t	Dynel Cloth	5.8	Discolored
D-2004	Dynel Cloth	5.62	Discolored
320-Dy*	Dynel Felt	0.62	Discolored
320-DA-B	Dacron Felt	0.77	Slightly Discolored
320-DA-V	Dacron Felt	1.11	
G-206-C	Glass Cloth	4.5	(0.7% Boron)
X-10063-FB*	Polyethylene	0.93	
Porous Plastic*	Kel-F Disc	0.00	
Aloxite	Al ₂ O ₃	0.027	Grade 2
Selas	Al ₂ O ₃ - Si	0.045	Grade XFF (<.1% Boron)
Multibraid	316 SS	0.076	Grade XXD-400 (7 mil wire)
Rigimesh	316 SS	0.028	Grade M (2 mil wire)
Sintered SS	316 SS	0.71	Grade G

Test Solution: 2.4 M HNO₃, 0.1 M Oxalic - 25 C - Agitated

^t - Filter Media presently used for Plutonium Oxalate

* - Immersed for 60 days in Plutonium Nitrate solution

No apparent radiation damage detected

DECLASSIFIED
WITH DELETIONS

DECLASSIFIED
WITH DELETIONS

-15-

HW-62091

FIGURE 1

Plutonium Oxalate Filtration

DECLASSIFIED
WITH DELETIONS

SECRET

FIGURE 2
Disk Filter

UNCLASSIFIED

FIGURE 3

Slide Valve and Wear Plate

UNCLASSIFIED

-17-

HW-62091

105-019

UNCLASSIFIED

FIGURE 4
Equipment Assembly

UNCLASSIFIED

-18-

HW-62091

FIGURE 5

Disk Filter in Operation

FIGURE 6 - Flowsheet For Cerium Oxalate Strike (Precipitation)

FIGURE 7

Particle Size Distribution of Slurries

Cumulative Weight Percent

Particle Size (Microns)

Mass
Mean

○ Plutonium Oxalate Particle Size
(234-5 Lab)

● Dicalite-Talc Mixture Particle Size
(MSA Analyzer & Manufacturer)

Specific Gravities

Plutonium Oxalate = 2.6

Dicalite = 2.3

Talc = 2.7

105-023

DECLASSIFIED
WITH DELETIONS

FIGURE 8

Pore Size Distributions of Filter Media

FIGURE 9

Filter Media Effect on Solids Penetration Rate (Summary)

FIGURE 10

Filter Media Effect on Precoat Blinding Rate (Summary)

DECLASSIFIED
WITH DELETIONS

-25-

HW-62091

FIGURE 11

Blinding Rate of Aloxite Medium

105-026

DECLASSIFIED
WITH DELETIONS

FIGURE 12

Scalpel Effect on Blind Precoat

UNCLASSIFIED

105 - 027

FIGURE 13
CONTINUOUS OPERATION

105-028