27/
8-1-78
256 NTIS

The Fluorophotometric Determination of Uranium: An Automated Sintering Furnace and Factors Affecting Precision

J. E. Strain

MASTER

OAK RIDGE NATIONAL LABORATORY
OPERATED BY UNION CARBIDE CORPORATION · FOR THE DEPARTMENT OF ENERGY

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

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.

Printed in the United States of America. Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road, Springfield, Virginia 22161
Price: Printed Copy \$4.50 ; Microfiche \$3.00

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, contractors, subcontractors, or their employees, makes any warranty, express or implied, nor assumes any legal liability or responsibility for any third party's use or the results of such use of any information, apparatus, product or process disclosed in this report, nor represents that its use by such third party would not infringe privately owned rights.

ORNL/TM-6431

Contract NO. W-7405-eng-26

ANALYTICAL CHEMISTRY DIVISION

MASTER

THE FLUOROPHOTOMETRIC DETERMINATION OF URANIUM:
AN AUTOMATED SINTERING FURNACE AND
FACTORS AFFECTING PRECISION

J. E. Strain

NOTICE
This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or 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.

Date Published: July 1978

NOTICE This document contains information of a preliminary nature. It is subject to revision or correction and therefore does not represent a final report.

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37830
operated by
UNION CARBIDE CORPORATION
for the
DEPARTMENT OF ENERGY

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

Reg

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT.....	1
1. INTRODUCTION.....	2
2. DESIGN AND OPERATION OF AN AUTOMATIC FUSION FURNACE...	3
3. FACTORS AFFECTING PRECISION.....	14
3.1 LiF-NaF Ratio Variation.....	15
3.2 Variations of Pellet Mass.....	16
3.3 Variations in Uranium Loading on the Pellet.....	16
3.4 Quenching Due to Dissolution of Metals from the Sintering Dish.....	20
3.5 The Effect of the Uranium Oxidation State on Fluorescent Yield.....	22
4. SUMMARY AND CONCLUSIONS.....	24
REFERENCES.....	25
APPENDIX.....	26

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

LIST OF FIGURES

	<u>Page</u>
Figure 1. Automated Sintering Furnace for Fluorometric Uranium Determination.....	4
Figure 2. Fusion Furnace Construction Detail.....	5
Figure 3. Fusion Furnace Construction Detail.....	6
Figure 4. Fusion Furnace Construction Detail.....	7
Figure 5. Fusion Furnace Construction Detail.....	8
Figure 6. Fusion Furnace Construction Detail.....	9
Figure 7. Fusion Furnace Construction Detail.....	10
Figure 8. Temperature Monitor and Control Circuit.....	11

LIST OF TABLES

	<u>Page</u>
Table I. Effect of Thorough Mixing of Flux on Precision...	15
Table II. Effect of Pellet Mass on Fluorescent Yield.....	16
Table III. Precision of Sample Loading and Fluorescent Yield.	17
Table IV. Fluorometric Reading in Nanograms.....	21
Table V. The Fluorescent Yield of U^{+6} and U^{+4}	23

THE FLUOROPHOTOMETRIC DETERMINATION OF URANIUM:
AN AUTOMATED SINTERING FURNACE AND
FACTORS AFFECTING PRECISION

J. E. Strain

ABSTRACT

The fluorometric method for the determination of uranium has been in use for many years and is one of the more sensitive methods. The major disadvantage of the method is its relatively poor precision ($\sim \pm 20\%$). The development of a new, high precision, fluorometer in 1973 did not materially improve the overall precision. This prompted the design of an automated fusion furnace and a systematic investigation into the cause of the poor analytical precision.

The furnace design is detailed in the first section of this report. The fusion furnace consists of four individually controlled, slotted-tube furnaces that automatically dry, sinter and anneal the fluoride or carbonate pellet used in the fluorometric determination of uranium. The furnace operates in air and prepares approximately 90 pellets per hour for fluorometric measurement.

The factors that were thought to affect the precision of the method were investigated. The two factors that seem to be the most influential are 1) the manner in which the sample is loaded onto the pellet; and 2) the surface characteristics of the platinum dish in which the pellet is sintered and measured fluorometrically.

1. INTRODUCTION

The fluorometric determination of uranium was used at the Oak Ridge National Laboratory almost unchanged from the late 1940's until 1973. The procedure consisted of making the sample about 4 N in nitric acid and quantitatively extracting the uranium into an equal volume of 20% tri-n-octylphosphine oxide (TOPO) in hexane. An aliquot of this organic phase containing between 10 to 1000 nanograms of uranium was pipetted onto a 300 mg pellet of 98% NaF-2% LiF. This loaded pellet was then fused on a three-burner apparatus that held a platinum dish. After fusion the dish and pellet were placed in a fluorometer that utilized two 100 watt mercury lamps, a phototube and a galvanometer to measure the fluorescence. Although the fluorometric method has been in use since 1935 (1) and is extremely sensitive, it has not been generally applied due to the poor precision ($\sim \pm 20\%$). The problems and controversy encountered in the method are reviewed in two papers published in the late 1940's (2, 3). We had assumed that the poor precision was due primarily to the instrumentation and the poor temperature control of flame fusion of the NaF pellets. In 1973, T. R. Mueller and H. H. Ross designed a totally new fluorometer for use in the determination of uranium (4). The new instrument uses solid state circuitry with an internally generated calibration curve, a direct digital read-out in nanograms of uranium, and an internal precision of 1%. Unfortunately, the use of this new fluorometer with the three-burner, flame fusion device produced no significant improvement in the $\pm 20\%$ standard deviation that had been accepted for the past three decades.

This lack of improvement prompted the development of the automated fusion furnace and the subsequent investigation of the causes of the observed imprecision.

2. DESIGN AND OPERATION OF AN AUTOMATIC FUSION FURNACE

The conflicting reports and the controversy between proponents of flame fusion and those who preferred furnace fusion (1) induced us to approach the design of a continuous fusion furnace with uncertainty. We, therefore, designed a slotted tubular furnace with the maximum flexibility. The furnaces could be moved within the housing to vary the heat-cool-anneal cycle; the rate of travel of the pellets through the furnace was variable from 1 cm/min to 25 cm/min by means of a variable speed motor.

By trial and error we established the optimum temperatures, speed, and furnace position to produce a fluorescent yield in the 98% NaF-2% LiF flux that was equal to that obtained in the three-burner fusion device (ORNL Q-1187-7). Surprisingly, the optimum fluorescent yield was produced by a pellet that was not melted but sintered to the point that its volume is reduced to about half of its original size. Any melting of the pellet reduced the fluorescent yield. The optimum transport speed through the furnace was found to be 6 cm/min.

The final furnace design is shown in Figure 1 and its construction is detailed in Figures 2 through 7. A parts list is contained in the Appendix. The furnace consists of four separately-controlled, slotted tube furnaces. The first furnace serves to remove the organic solvent

Figure 1. Automated Sintering Furnace for Fluorometric Uranium Determination.

DECK SUPPORT
(PAGE 3 OF 6)

Figure 2. Fusion Furnace Construction Detail.

Figure 3. Fusion Furnace Construction Detail.

TOP DECK
(1/4 THICK ALUMINUM)

ALL DIMENSIONS IN INCHES

Figure 4. Fusion Furnace Construction Detail.

SPROCKET SUPPORT SHAFT
(2 REQ'D.)

FABRICATE FROM MILD STEEL
PRESS FIT SHAFT INTO HUB

NOTE: DRILL $\frac{7}{8}$ HOLE ONLY PARTWAY
THROUGH $\frac{1}{4}$ PLATE TO LEAVE A
BEARING SUPPORT LIP

DIMENSIONS ARE IN INCHES

DRIVE GEAR HOUSING

Figure 5. Fusion Furnace Construction Detail.

NOTE : DIMENSIONS ARE IN INCHES

Figure 6. Fusion Furnace Construction Detail.

Figure 7. Fusion Furnace Construction Detail.

NOTE:

1. ADJ. OUTPUT OF T.C. AMP μ A 741 TO 0.100 VOLTS WITH 100 k TRIMPOT AT ROOM TEMPERATURE - 25°C
2. $\square$ 22 PIN CONNECTOR

Figure 8. Temperature Monitor and Control Circuit.

from the pellet. The second and third furnaces are the actual sintering sections and the fourth unit anneals the pellet to increase the fluorescent yield. The pellets are moved at the rate of 6 cm/sec through the furnaces by means of a 1 rpm reversible Bodine motor.

The temperature control for each furnace is accomplished by the solid state circuit shown in Figure 8. The temperature is set by means of a ten-turn potentiometer located on the front panel. The temperature sensing elements are chromel-alumel (Type K), stainless steel clad thermocouples. Two thermocouples are installed in each furnace section. One thermocouple is read directly on the front panel meter while the other is connected into the control amplifier. The sensitivity and linearity are adjusted so that each pair of thermocouples indicate the same temperature on the panel meter and the set temperature corresponds to the dial reading on the potentiometers. In all cases, the direct reading thermocouple is considered the true temperature.

The operating temperatures are dependent upon the type of flux used in the analysis. The standard flux in use at ORNL is the 98% NaF-2% LiF. Therefore, the temperatures were adjusted using this mix. Experimentation with times and temperature established the following optimum temperatures:

Drying furnace (#1) = 575°C

Fusion furnace (#2) = 990°C

Fusion furnace (#3) = 960°C

Annealing furnace (#4) = 870°C.

These temperatures do not melt the pellet but sinter it and cause a 50% reduction in pellet volume. If the temperatures are raised to produce a completely melted pellet, the fluorescent yield is reduced ~50% and the melt adheres tightly to the platinum dish. There is no improvement in precision if the pellet is melted completely.

The carbonate flux (10% NaF-45% Na_2CO_3 -45% K_2CO_3) can be used in the furnace by decreasing the temperature settings to:

Drying furnace (#1) = 575°C

Fusion furnace (#2) = 640°C

Fusion furnace (#3) = 645°C

Annealing furnace (#4) = 575°C.

The results obtained with the carbonate flux are comparable in precision but sensitivity is reduced by a factor of three. This reduced yield can be compensated for by increasing the photo-multiplier gain of the fluorophotometer. The lowered operating temperatures used in the carbonate fusion will also help prolong the life of the heating elements. The optimum temperatures to be used for the carbonate flux have not been determined and perhaps a sintered pellet might produce a higher fluorescent yield than the melted pellet. This possibility was not investigated because of our routine use of the 98% NaF-2% LiF melt.

The use of the automatic furnace reduced the relative standard deviation to about $\leq 10\%$. Failure to achieve more precise results prompted an investigation into the cause of the variation.

3. FACTORS AFFECTING PRECISION

To evaluate the factors affecting the analytical precision it was necessary to standardize the sample preparation and treatment so that single variable effects could be evaluated. To this end, the following equipment and procedure was used in all cases except where noted.

Equipment: ORNL Model 5198 fluorometer, the automated fusion furnace, platinum dishes for fusion and reading of pellets, a pellet press, hand operated to produce a 10 mm diam x 4 mm thick pellet and a fusion mixture consisting of 98% NaF-2% LiF.

The analytical procedure was as follows.

1. A sample containing between 1 and 1000 nanograms of uranium is diluted with nitric acid to a volume of ~10 ml and ~4N in HNO_3 in a 30 ml vial.
2. Add 10 ml of 20% trioctylphosphine oxide (TOPO) in hexane and extract by vigorous shaking for 5 minutes.
3. Allow the phases to separate and pipet 50 to 150 microliters of the organic phase onto a 300 mg NaF-LiF pellet (1 cm diam x .4 cm high) in a platinum dish.
4. Place the dish and pellet on the fusion furnace support rings.
5. When the furnace has attained the proper temperature (#1 575°C, #2 990°C, #3 960°C, #4 870°C) start the drive motor.
6. When the sample has exited the final annealing furnace and cooled to room temperature, it is read on the calibrated fluorometer.

7. The displayed value is the number of nanograms of uranium on the pellet.

The samples are normally analyzed in triplicate and known standards are run to verify the fluorometer calibration.

The possible causes of the poor precision were investigated with the following results.

3.1 LiF-NaF Ratio Variation

Because the LiF and NaF are blended as dry powders, it was thought that variation in LiF concentration between pellets might be a factor in the poor precision. Therefore, a series of 10 pellets were pressed from the "as blended" mixture while another ten pellets were pressed from the same mixture after an additional 1.5 hours of vigorous blending in a laboratory ball mill. The results presented in Table 7 show that there was no improvement in precision (the ball mill contaminated the fluoride mixture so as to result in a 55% quenching of the measured fluorescence).

TABLE I
Effect of Thorough Mixing of Flux on Precision

Apparent Nanograms U Per Pellet	
<u>Std. Mixture</u>	<u>Blended Mixture</u>
640, 636, 595, 614	294, 294, 287, 272
622, 633, 637, 636	286, 265, 278, 274
616, 622	257, 297
Ave. 625 $\pm$ 2.3%*	280 $\pm$ 4.6%*

*Rel. Std. Dev.

3.2 Variations of Pellet Mass

The manual pellet preparation was known to produce pellets that varied in mass by as much as 10%. To evaluate this effect, ten pellets each of three different sizes, were produced and loaded with 0.100 ml of the organic extract containing 105 ng of uranium. After loading, the pellets were dried, sintered, and measured in platinum dishes. Table II shows the results.

TABLE II
Effect of Pellet Mass on Fluorescent Yield

<u>Pellet Diameters (mm)</u>	<u>Height (mm)</u>	<u>Apparent U (ng)</u>	<u>Rel. Std. Dev.</u>
3	3	76.5	$\pm 6.4\%$
6	4	100.5	$\pm 6.3\%$
10	4	114.0	$\pm 6.1\%$

The low value of the 3 mm diameter pellet probably reflects the failure of the pellet to absorb all of the 0.1 ml sample. The small change in fluorescent yield between the 6 mm diameter pellet and the 10 mm diameter pellet indicates that a 10% change in mass would have no significant effect on the precision.

3.3 Variations in Uranium Loading on the Pellet

To evaluate the magnitude of pipetting errors the uranium standard was spiked with ^{237}U so that gamma-ray counting could be used to measure the quantity of uranium on the sintered pellet. The ^{237}U was prepared by irradiation of 1 mg of ^{236}U in a thermal neutron flux of

$\sim 10^{15}$ n sec⁻¹ cm⁻² for ten minutes. After a 24-hour decay period, the uranium was dissolved in nitric acid and purified by repeated TTA extractions and a final TOPO extraction. The product was examined by gamma-ray pulse height analysis and was found to be free from fission product contamination. Sufficient natural uranium was then added to produce a specific activity of 7 counts per second per nanogram of uranium when counted in a 5 cm diameter well-type NaI detector. Six standard pellets (10 mm diam x 4 mm high) pellets were loaded with 0.100 ml of the organic extract containing 150 nanograms of uranium; they were sintered and measured in the fluorometer and also assayed by gamma-ray counting. The results are summarized in Table III.

TABLE III

Precision of Sample Loading and Fluorescent Yield

<u>Sample No.</u>	<u>Fluorometer reading</u>	<u>Gamma count/ 300 sec</u>
1	150	22,055
2	164	22,508
3	158	21,681
4	184	22,066
5	161	21,497
6	152	21,690
Ave. 156 $\pm$ 3.2%*		21,916 $\pm$ 1.7%*

---

* Rel. Std. Dev.

Table III not only shows that the fluorescent yield is less precise than the loading, but also indicates that there is no apparent correlation between the gamma count and the measured number of nanograms indicated by the fluorometer.

A qualitative examination of the pellets under ultra-violet light revealed that there was a significant difference in the distribution of uranium on the pellet. In every case, the uranium was concentrated in the upper portion of the pellet. The diameter of the fluorescent spot varied from a minimum of ~ 2 mm diameter to nearly uniform coverage of the top of the sintered pellet. The uranium seems to be extracted and fixed in place in the pellet very rapidly. The size of the deposited uranium is controlled by the speed of sample loading. A slow delivery onto the pellet produces a very small spot of high fluorescent intensity. The small spot results in low fluorescent yield, probably due to self absorption. Rapid sample loading on the pellet produces a more uniform loading of the upper surface of the pellet and the highest fluorescent yield. One series of pellets containing the same amount of uranium deliberately non-uniformly loaded produced the following readings relative to the uranium loading pattern:

U concentrated in center ~ 1 mm diam ~ 1 mm deep--177 nanograms

U deposit partially covering upper face--192 nanograms

U uniformly deposited on the upper face--251 nanograms.

These observations clearly indicate that the distribution of the uranium in the pellet can seriously affect the precision of the measurement. The vertical distribution can be illustrated by comparin

the specific radioactivity of the ^{237}U tagged pellets found in the upper and lower surface of a sintered pellet (a pellet weighing 299 mg and loaded with 300 nanograms of natural U + ^{237}U). After sintering, 11.5 mg of NaF was milled off the upper face and 20.3 mg were milled from the lower face. The specific activity of the material recovered from the upper face was 32.5 times higher than the material from the lower face and represented approximately 12% of the total uranium added. It was further noted that if uranium loaded pellets were inverted after sintering and read on the fluorometer in the inverted position, the fluorescent yield decreased by 48%, almost a factor of two.

The clear indication is that the technique of placing the sample on the pellet is a major factor in achieving good precision. In an attempt to minimize this effect, we investigated the possibility of loading the uranium onto the pellet in a strong HF (7 Normal) solution. Back extraction of the TOPO organic phase with 7N HF quantitatively transferred the uranium to the aqueous phase and this was loaded onto pellets in the usual manner. The uranium again precipitated on contact and was concentrated at the point of loading. As in the case of organic samples the distribution of uranium in the upper surface of the pellet was dependent on the rate that the sample was placed on the pellet. Attempts to use an aqueous medium other than HF (i.e., HCl, H_2SO_4 , or HNO_3) resulted in a quenching of the fluorescence, lowering of the melting point (HCl) or swelling of the pellet (HNO_3). No solution has been found to minimize the variability observed in manual sample loading.

3.4 Quenching Due to Dissolution of Metals from the Sintering Dish

The concentration of uranium on the upper surface of the pellet casts doubt on the assumption that diffusion of platinum into the pellet during fusion could cause quenching of the fluorescence. If quenching is not a real problem, the use of materials other than platinum can be used to contain the samples during sintering. Graphite was used to contain the pellet in a first series of experiments because it is non-contaminating and might provide a protective atmosphere of CO_2 around the pellet. The pellets sintered and measured in graphite had the same appearance as those sintered in platinum but the measured fluorescent yield was about half the value observed when measured in a platinum dish. When the pellet sintered on the graphite dish was transferred to a platinum dish for measurement, the expected fluorescent yield was obtained. Subsequent experiments showed that 42% of the measured fluorescence is reflected from the surface of the platinum dish. This finding means that the surface of the platinum dish is also important to the analytical precision. To verify these conclusions, a series of experiments were run. 1) Nine randomly selected platinum dishes were loaded with standard pellets and 300 nanograms of uranium. 2) Nine platinum dishes were selected, reshaped and polished to a smooth surface. They were then loaded with standard pellets and 300 nanograms of uranium. 3) A single platinum dish was blackened with lamp black and each of the sintered pellets from (2) above were measured for fluorescence in the single dish. The results are summarized in Table IV.

TABLE IV
Effect of Surface Reflection on Fluorometric Precision

Fluorometric Reading in Nanograms		
<u>Random Dishes</u>	<u>Polished Dishes</u>	<u>Single Blackened Dish</u>
294, 294, 287	302, 293, 304	180, 177, 178
272, 286, 265	286, 297, 299	177, 179, 177
278, 272, 259	295, 286, 297	174, 175, 170
Ave. 284 $\pm$ 4.6%*	296 $\pm$ 2.9%*	176 $\pm$ 1.7%*
*Rel. Std. Dev.		

In a related experiment, a single sintered pellet was read in twenty-one different platinum dishes with no surface preparation of the dishes. The average value measured was 303.6 with a relative standard deviation of $\pm$ 3.8%. The maximum reading recorded was 323 nanograms and a minimum reading was 286 nanograms.

The above experiments indicate that replacement of the platinum dish with a one-time-use disposable dish of uniform reflectance could eliminate the large platinum inventory now required and would improve the analytical precision. The most practical material found was a high-fired MgO or Al₂O₃ ceramic dish. These show no visible reaction with the NaF pellet, do not cause quenching and do not adhere to the pellet during sintering. The only problem noted was a variable blank apparently due to uranium contamination corresponding to 5 ± 3 nanograms of uranium per dish. This source of error can be eliminated by

modification of the sample holder of the fluorometer so that the samples are measured after removal from the sintering dish. The limited number of ceramic dishes available and the present sample load precludes an extensive investigation into the use of ceramic dishes at this time. The study will be resumed at a future date.

3.5 The Effect of the Uranium Oxidation State on Fluorescent Yield

Uranium is often determined in aqueous solutions by direct loading of the sample on the NaF pellets without separation of the uranium by solvent extraction. Since the composition of the uranium fluoride compound producing fluorescence is uncertain it was assumed that the oxidation state of the uranium could affect the fluorescent yield. To answer this question, two standard solutions were prepared. One contained U^{+6} obtained by extracting $UO_2(NO_3)_2$ from 4N HNO_3 into TOPO and back extracting into 7N HF. The other contained U^{+4} obtained by the direct dissolution of UF_4 in 7N HF. Twenty standard pellets were prepared and loaded with uranium. Ten contained 640 nanograms of U^{+6} and ten contained 530 nanograms of U^{+4} . After sintering in platinum dishes the fluorescence was measured on the fluorometer using an internal calibration curve calculated from U^{+6} data. The results are presented in Table V.

TABLE V
The Fluorescent Yield of U^{+6} and U^{+4}

Fluorometric Reading in Nanograms	
U^{+6} (640 nanograms)	U^{+4} (530 nanograms)
635, 678, 629, 633	496, 513, 554, 545
620, 651, 664, 680	636 ¹ , 544, 498, 542
580 ¹ , 665	506, 483
Ave. $643.5 \pm 4.8\%^*$	$531.7 \pm 8.3^*$

¹These extreme values would normally be discarded but in this case are retained to illustrate the problem frequently encountered in the fluorescent method.

*Rel. Std. Dev.

From the above data, it appears that there is no significant difference in the fluorescent yield between U^{+6} and U^{+4} . No bias will be introduced by the oxidation state of the uranium when aqueous samples are loaded directly on the NaF pellet. It should be pointed out, however, that additions of any ions other than uranyl and fluoride or volatile solvents produce quenching to some degree. If solvent extraction is not used to eliminate foreign ions, a method of standard additions should be used to compensate for the decrease in fluorescent yield due to quenching.

4. SUMMARY AND CONCLUSIONS

During the course of this investigation it has become evident that there are two major factors that contribute to the poor precisions of the method. These are:

1. Non-uniform loading of uranium on the pellet; and
2. The variation in reflected fluorescence caused by surface irregularities in the platinum dishes used for sintering and measurement.

The second source of variation can be eliminated simply by transferring the sintered pellet into a holder of uniform surface, either non-reflecting or totally reflecting, prior to measuring the fluorescence.

The first problem is more difficult and will require either an automated sample loader to uniformly load a fixed sample volume at a constant rate, or a coating for the pellet such that the sample after loading will slowly diffuse onto the surface of the pellet. The latter approach would seem to be the more attractive from the standpoint of simplicity and cost. A cup-shaped semipermeable material that would contain the sample during diffusion and then be vaporized in the furnace would be ideal. The combination of a diffusion layer with a preformed standard pellet would be a valuable adjunct to the fluorometric determination of uranium.

Future work will concentrate on the development of a volatile diffusion coating and expanded use of the ceramic sintering dishes.

REFERENCES

1. Hernegger, F. and Kurlih, B. Sitzber. Akad. Wiss. Wien, Math. Naturw. Klasse Abt. IIa. 144, p. 217 (1925).
2. Price, G. R., Ferretti, R. J. and Schwartz, S., "The Fluorophotometric Determination of Uranium," ANL-4002 (1947).
3. Barton, C. J., "The Precision of the Fluorometric Determination of Uranium and Some Optical Properties of Phosphorous," TID-271 part 6 (1949).
4. T. R. Muller and Ross, H. H. (to be published).

APPENDIX

Fusion Furnace Parts List

ELECTRONIC

Resistors

<u>Quantity</u>	<u>Value</u>	<u>Type</u>
4	2M	Metal film
8	390K	Metal film
8	50	Metal Film
4	25K	Metal Film
4	20K	Metal Film
4	10K	Metal Film
8	50K	Carbon
4	500K	Ser. 76 helitrim
4	100K	Ser. 76 helitrim
4	50K	Ser. 76 helitrim
4	20K	Ser. 76 helitrim
4	5K	10 turn helipots plus dials
4	2K	Carbon $\pm$ 10%
8	5K	1%

Capacitors

<u>Quantity</u>	<u>Value</u>	<u>Type</u>
4	.05 uf	Silvered mica
6	.01 uf	Silvered mica
4	0.1 uf	Silvered mica

Simiconductors

<u>Quantity</u>	<u>Source</u>	<u>Type</u>
4	Fairchild	μ A727
4	Fairchild	μ A741
4	Fairchild	μ A741
4	Fairchild	μ A747
1	Texas Inst.	SN 7407 Hex-buffer
1	Texas Inst.	SN 7408 Quad AND gate

DC Power Supply

+ 15v DC at 200ma Analog Devices Mod. 920; + 5v regulator (μ a 7805)

Solid State Relays

4 teledyne relay mod. 61A-21A2-12Z, contact rating 21 amps

Pilot Lights

7 indicating postlite, neon, 125v mod. 105, Drake Company

Panel Meter

Indicating 0 to 1500 deg. C with chromel-alumel thermocouple,
mod. -4- 421-806R2, Assembly Products

MECHANICAL

Sprockets

Two 8.5 inch diameter bicycle sprockets with a 1" diameter hub

Chain

Approximately 6' of compatible bicycle chain, 0.5" links, 5/16"
thick

Drive Motor

1 rpm Bodine motor

Drive Gears

8 tooth brass gear, 1/4" shaft

136 tooth brass gear, 3/8" shaft

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

INTERNAL DISTRIBUTION

1. D. A. Costanzo
2. J. H. Cooper
3. W. S. Lyon
4. R. J. McElhaney
5. R. R. Rickard
6. H. H. Ross
7. W. D. Shults
8. J. E. Strain
- 9-10. Central Research Library
11. Document Reference Library
- 12-14. Laboratory Records Dept.
15. LRD-RC
16. ORNL Patent Office

EXTERNAL DISTRIBUTION

17. Barry Nicholson, Atlantic Richfield Hanford Company,
Richland, Washington 99352
18. T. R. Herold, E. I. DuPont Co., Savannah River Labs.,
Aiken, South Carolina 29801
19. Research and Technical Support Division, DOE-ORO
20. Anne Gibbs, E. I. DuPont Co., Savannah River Plant,
Aiken, South Carolina 29801
- 21-47. Technical Information Center, P.O. Box 62, Oak Ridge,
Tennessee 37830