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ROVER REPORT FOR MARCH 1963

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CONTENTS

Page

Abstract 4

1. Introduction 5

1.1 Brief Survey of the Problem and the Research 5

1.2 Status and Evaluation of the Efforts 7

2. Recovery of Uranium from Spent Rover Fuel by the Combustion-Fluorination Process 9

2.1 Introduction 9

2.2 Status of Development 9

2.3 Current Work 10

2.3.1 Fluorination Run F-2 10

2.3.2 Summary of Both Parts of Run F-2 15

2.4 Breaking Tests on Prototype Fuel 16

2.5 Conclusions 16

3. Laboratory Work in Support of the Combustion-Fluorination Process 16

3.1 Thermogravimetric Fluorination of Partially Fluorinated Ash from Run RO-2: Study to Determine the cause of the Incomplete Fluorination 16

4. Aqueous Processes for Recovering Uranium from Rover Fuel Combustion Ash 19

4.1 Development of the Deline-Burn-Dissolve Flowsheet 19

4.1.1 Recovery of Niobium from Delining Solutions 20

4.1.2 Volatility of Fission Products During Combustion of Irradiated Rover Fuel 20

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ABSTRACT

Laboratory-scale and engineering-scale efforts to recover uranium (and possibly the niobium) from spent Rover fuel elements are reported, and the status of each aspect of the work is presented, including the evaluations.

Basically, the fuel consists of pyrolytic-carbon-coated spheroids of uranium carbide dispersed in a graphite matrix. The coolant channels in the graphite are lined with niobium carbide. For uranium recovery, in essence the fuel element is first oxidized to remove the graphite, leaving an ash that contains principally the oxides of uranium and niobium; then, the uranium is separated from the niobium and nonvolatile fission-products by appropriate methods. Most of the chemical problems are associated with the niobium and the pyrolytic carbon coating.

The Combustion-Fluorination Process represents the main effort, and basically it involves the combustion of the fuel and the fluorination of the ash; the uranium is recovered as UF_6. The method is in the pilot-plant stage of development. This issue includes the following information: the overall status of the development, the procedure, results, and evaluation of a fluorination run (F-2), and the results of tests on the breaking of temperature-cycled prototype fuel rods. In addition, laboratory-support work on the probable cause(s) of incomplete fluorination of the combustion ash in run F-2 (above) is reported.

Alternatives to combustion-fluorination involve the dissolution of the ash with hydrofluoric-nitric acid mixtures, thereby preparing solutions adaptable to the solvent-extraction separation of the niobium, uranium, and fission products. Several processes are based on this system, and they are: the Deline-Burn-Dissolve, the Burn-Dissolve, and the Burn-Leach Processes. Reported in this issue is more on the tentative flowsheet for the Deline-Burn-Dissolve Process, with special reference to the recovery of niobium from the delining solution when used on the new coated-particle fuel. These methods are in the laboratory stage, though some pilot-plant work has been done on the Deline-Burn-Dissolve method.

Other alternatives to combustion-fluorination include the gaseous chlorination of the combustion ash to form the volatile, separable chlorides of niobium and uranium, and the chlorination of the ash with hot hexachloropropene (HCP) followed by appropriate solvent-extraction steps to separate the niobium and uranium. These methods are in the laboratory stage of development. Nothing is reported on these subjects in this issue.

1. INTRODUCTION

1.1 Brief Survey of the Problem and the Research

The studies are being made in order to develop an efficient and economical process for the recovery of uranium from spent Rover fuel, which consists of uranium carbide (coated with pyrolytic carbon) dispersed in graphite, with niobium carbide liners in the coolant channels. The burnup of the elements will be low, and they will not be excessively radioactive. The problems derive principally from the pyrolytic-carbon coating on the uranium carbide spheres and from the niobium carbide liners for the coolant channels.

The coating is very resistant to chemical attack, and a preliminary step in the process necessarily is the removal of the coating, whether or not this step is to be followed by fluorination, chlorination, or aqueous processing. This first step is conveniently done by simple combustion, which also includes the advantage of removing the bulk of the fuel element (the graphite moderator) as carbon dioxide. Then, the ash (oxides of uranium, niobium, and nonvolatile fission products) may be fluorinated, chlorinated, or treated by an aqueous mixture of hydrofluoric and nitric acids in order to recover the uranium. It may even be possible to eliminate hydrofluoric acid, according to a recently introduced variant of the aqueous-processing route. The combustion step, on the other hand, may or may not result in sintered oxides or uranium and niobium, depending on the controllability of the temperature, and dense sinters may make other steps in the recovery more difficult to take. As stated above, the niobium carbide liners and the pyrolytic

carbon coatings on the fuel spheres are presenting the most difficult problems and consequently requiring the most attention, whatever the approach.

The main effort is the Combustion-Fluorination Process, which essentially consists in burning the element in oxygen in order to produce a combustion ash, and then fluorinating the ash in order to separate the uranium (as the volatile hexafluoride) from the fluorides of niobium and the fission products. Some of the fission products are also volatile, as is niobium fluoride, but, due to their physical properties, they can be separated from the uranium by adsorption on beds of sodium fluoride, for example. The Combustion-Fluorination Process is in the pilot-plant stage of development.

The alternative methods for recovering the uranium from the combustion ash are the aqueous nitric-hydrofluoric acid process and the liquid- or gas-phase chlorination processes and their variants. According to the aqueous route, it may be possible to circumvent the sintering of the niobium pentoxide by first dissolving the liners from the elements in order to prevent the appearance of niobium oxide in the combustion ash, thereby improving the process whether the postcombustion steps follow an aqueous or a gaseous route. That development is called the Deline-Burn-Dissolve (DBD) Process and has advanced to the point of a pilot-plant study. Two variations of the DBD Process have also been introduced: the Burn-Dissolve and the Burn-Leach Processes. The Burn-Leach Process, should it be workable, offers the advantage of eliminating hydrofluoric acid from the aqueous processing.

The other alternative to the Combustion-Fluorination Process is one that basically involves the liquid-phase, low-temperature chlorination of the combustion ash with hexachloropropene (HCP), or high-temperature, gas-phase chlorination, plus the useful variants that may develop as the study advances. Liquid- or gas-phase chlorination have so far shown themselves worthy of study because early corrosion results indicate that the nickel reactor will not be attacked severely. Gas-phase chlorination is very similar in many important respects to the fluorination method,

but liquid-phase chlorination with HCP has special advantages. For example, it is run at low temperature (below 200°C), and the product is readily adaptable to extraction by tributyl phosphate--Amsco mixtures. On the other hand, gas-phase chlorination may offer promise as a method not only for separating the uranium in bulk but for purifying it, though this has yet to be demonstrated. The gas-phase work is done in an apparatus called the "Torrefactor."

Laboratory work in support of the processes is continuing to help identify and solve problems as they arise.

The status and evaluation section below is confined to the progress made on the work done during this reporting period. The other progress reports in this series are: ORNL-TM-170, 199, 227, 267, 298, 373, 408, 426, 443, 509, and 520.

1.2 Status and Evaluation of the Efforts

Recovery of Uranium from Spent Rover Fuel by the Combustion-Fluorination Process. The fluorination of the combustion ash from run 0-2 was completed in two steps because of an unforeseen "freezeout" of NbF_5 in the top of the distillation column. The NbF_5 from fluorination runs F-1 and F-2 had built up a loading throughout the still, and this condition, once reached, caused the response of the system to temperature control to become slow. Also, the sensing of temperatures became inadequate, and proper conditions could not be maintained at the still head. The entire system will be studied and revised. In spite of the unexpected difficulty, the uranium recovered or accounted for was 104.8% of the computed charge, which can be compared with the recovery of 107% of the theoretical weight of the oxide. Only 1.8% of the uranium followed a path regarded as a process loss (residue in the reactor), and this loss was higher than expected because of an early shutdown of the run, caused by a plugged line downstream from the reactor.

The following conclusions can be drawn from fluorination run F-2:

1. The fluorination of dense materials of various compositions can be readily accomplished if the reaction zone is kept at 400 to 450°C.

2. The separation system needs study and modification.

Breaking tests on prototype fuel led to the following conclusions: Temperature cycling does not materially alter the graphite properties with respect to the production of fines during breaking to short lengths.

Laboratory Work in Support of the Combustion-Fluorination Process: Thermogravimetric Fluorination of Partially Fluorinated Ash from Run RO-2. Partially fluorinated combustion ash from run F-2 (discussed above) was fluorinated in the thermobalance to determine possible causes for incomplete fluorination in that particular Unit Operations run, in which it was necessary to resort to a two-part fluorination. The conclusion offered is that the laboratory yield of 99.8% achieved in a normal manner indicated that the cause of the incomplete fluorination was not due to the composition or physical form of the combustion ash. No conclusion could be drawn with respect to the effect of refluxing NbF_5 as a possible cause of the difficulty. It was shown that the reaction rate responded well to fluorine control.

Aqueous Process for Recovering Uranium from Rover Fuel Combustion Ash. With respect to the recovery of niobium from delining solution used in Deline-Burn-Dissolve (DBD) Process, it is possible that the delining solution will contain only an insignificant amount of uranium when the new coated-particle fuels are delined. Therefore, recovery of the niobium from the delining solution before it is transferred to the waste tanks may be economically attractive. One method for recovering the niobium from hydrofluoric-nitric acid solutions that contain little or no uranium is extraction with hexone. According to laboratory investigations, niobium can be recovered from delining solutions by the use of multistage extraction, and, furthermore, a reasonably good separation from uranium can be made. For example, in solutions where the hydrofluoric acid concentration is 6 to 7 M, the extraction coefficient for niobium is 0.4, while that for uranium is less than 0.03.

With regard to the volatility of fission products during the combustion of irradiated Rover fuel, the equipment has been assembled and cold-testing started.

2. RECOVERY OF URANIUM FROM SPENT ROVER FUEL BY THE COMBUSTION-FLUORINATION PROCESS

R. W. Horton
R. S. Lowrie
F. G. Kitts
L. E. McNeese

2.1 Introduction

The fuel being used in reactors designed for the Rover project is a dispersion of enriched uranium carbide in graphite. The large quantity of uranium required for ground testing justifies uranium recovery. The use of a protective coating on the uranium particles (pyrolytic carbon), and the use of niobium carbide liners for the propellant-gas passages makes the chemical separation of uranium from graphite very difficult when using conventional aqueous methods. New process methods designed especially for this fuel are being developed. At present, the most feasible methods all use combustion with oxygen to eliminate the graphite and convert the niobium and uranium carbides to oxide. The separation of uranium from other combustion products by aqueous methods (Deline-Burn-Dissolve Process) and by the fluoride volatility process is being studied.

2.2 Status of Development

1. An 8-hr burning cycle, which includes time for charging with broken fuel rods and preheating, has been demonstrated for the 5-in.-diam reactor. The ash produced was a high-density, partially fused clinker with adequate surface for fluorination.

2. A finned, internally water-cooled gas cooler was very efficient in reducing the temperature of combustion gases before they left the reactor.

3. The woven metal filter used has a high resistance after a precoat has been laid down, probably because of the small particle size of the solids. Blowback would be required to keep the filter free and to discharge solids for fluorination.

4. Fluorination of partially fused ash at high rate is easily accomplished, provided that the material is kept at or above about 425°C during reaction. No evidence of local overheating has been noted.

5. Construction of a slab reactor 2.5-in. thick by 24-in. wide is about 75% complete. Plans for installation are proceeding.

6. A corrosion study at Battelle Memorial Institute shows only minor corrosion of nickel when exposed to refluxing NbF_5 for 400 hr.

7. Breaking experiments with rods exposed to 2000°C temperature have been made. No variations as a result of temperature are apparent.

2.3 Current Work

2.3.1 Fluorination Run F-2

The combustion of 50 broken fuel rods (run 0-2, reported in February 1962)[1] produced 4477.4 g of dense (2.4 g/cc), partially fused oxide material for fluorination. The material was too heterogeneous for representative sampling; however, data on the fuel charged indicated that 2.1718 kg of uranium and 1.1360 kg of niobium should have been contained in the charge. This loading, if oxidized to U_3O_8 and Nb_2O_5, would have produced 4186.3 g of mixed oxides. After fluorination, the total uranium recovered or accounted for was 2.274 kg, 104.8% of the computed charge, which can be compared with the recovery of 107% of the theoretical oxide weight. The total uranium accounted for after fluorination was distributed as shown in Table 1. Only item 3 in this group is considered to be a process loss. This loss, 1.8%, is higher than expected because of an early shutdown of the run, caused by a plugged line downstream from the reactor.

Discussion of First Part of Run F-2. The fluorination (run F-2) was done in two parts because of an unforeseen "freezeout." In the first part, the oxides were supported on a plate that rested on the fluorine

[1] F. L. Culler, Rover Report for February 1963, ORNL-TM-520 (in press).

inlet as in the original design. The plate used was nickel rather than alundum, and it was supported by the internal fluorine inlet chamber, which projects 8 in. above the bottom of the reactor. The nickel plate did not contact the heated reactor wall; therefore, all heat transfer to the plate and the material resting directly on it was by radiation. The radiant transfer, plus some conductive transfer to oxide particles that contacted the wall, provided sufficient heat to bring the bed up to the required 400°C at startup. A flow of 10 to 20 slm of 100% fluorine was introduced, and the reaction proceeded at a high rate until more than 50% of the oxides had reacted, then the rate appeared to be sharply decreased. (Estimates of the amount of fluorine reacted are based on relative volumetric flows of input fluorine and output fluorine-oxygen mixture, since no direct monitoring device is available.) The fluorine flow was stopped after 4 hr 15 min fluorinating time, during which time 2288 liters, equivalent to 176% of the stoichiometric requirement, was used.

Table 1. Distribution of Uranium after Fluorination Run F-2

Item	U (g)	% of U Charged, Calculated
1. Samples to various laboratories	30.0	1.4
2. NaF pellets removed from V-104 and V-105 for intermediate sampling	30.5	1.4
3. Residue in reactor after fluorination	38.4	1.8
4. NaF bed in V-104 (NbF_5 trap)	38.1	1.8
5. NaF bed in V-105 (UF_6 product trap)	2137.0	98.4
Totals	2274.0	104.8

When the reactor was opened, a layer, or cake, of solid material was left on the plate. This material was nonhomogeneous, varicolored, very heavy, and contained no apparent voids. Figure 1 is a top view of the largest piece of material, and Fig. 2 is an edge view of the large piece after being broken in two. Various fractions of this residue were examined

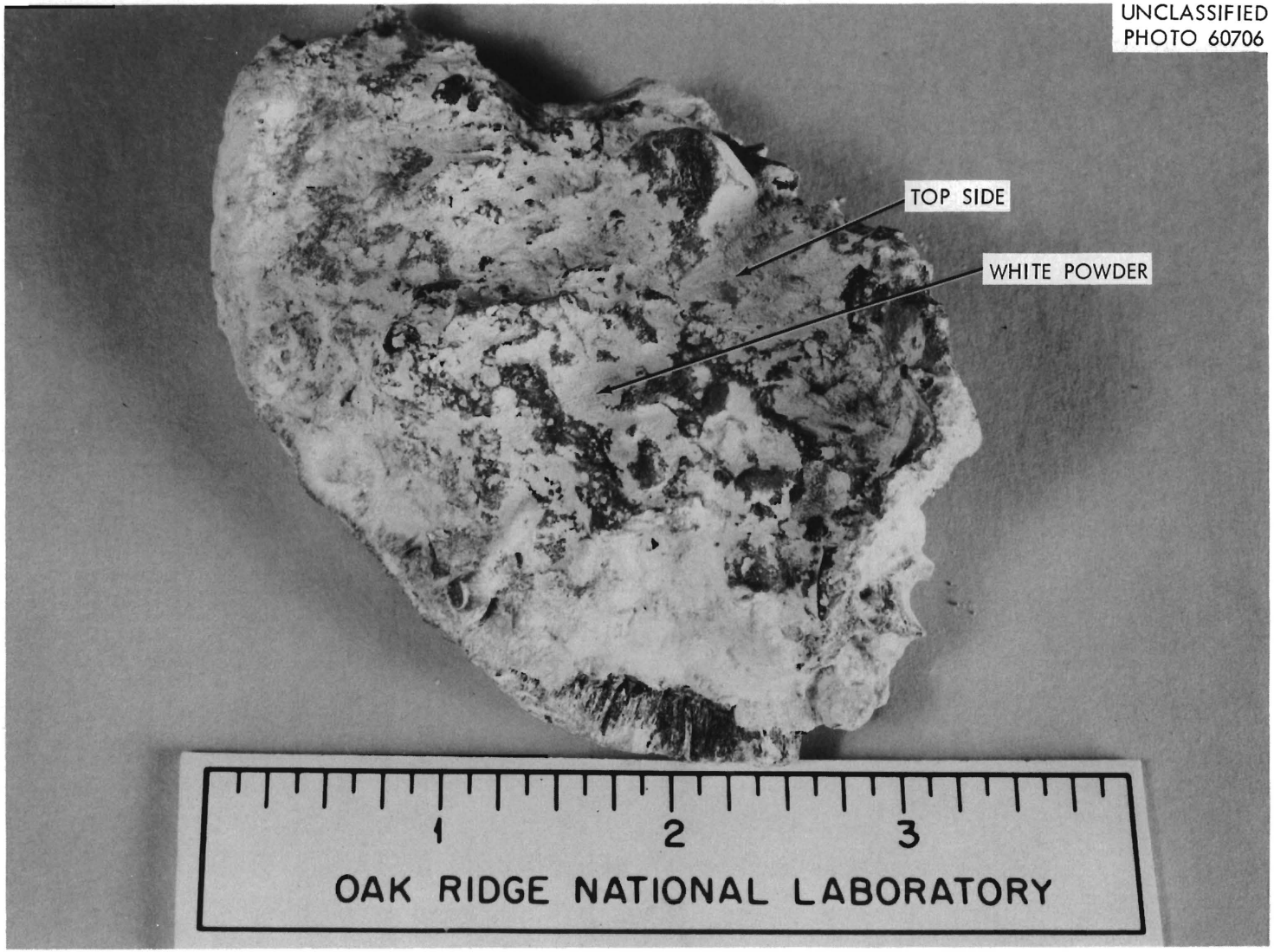

Fig. 1. Large Cake of Residue from Interrupted Fluorination, Run F-2 (Top View).

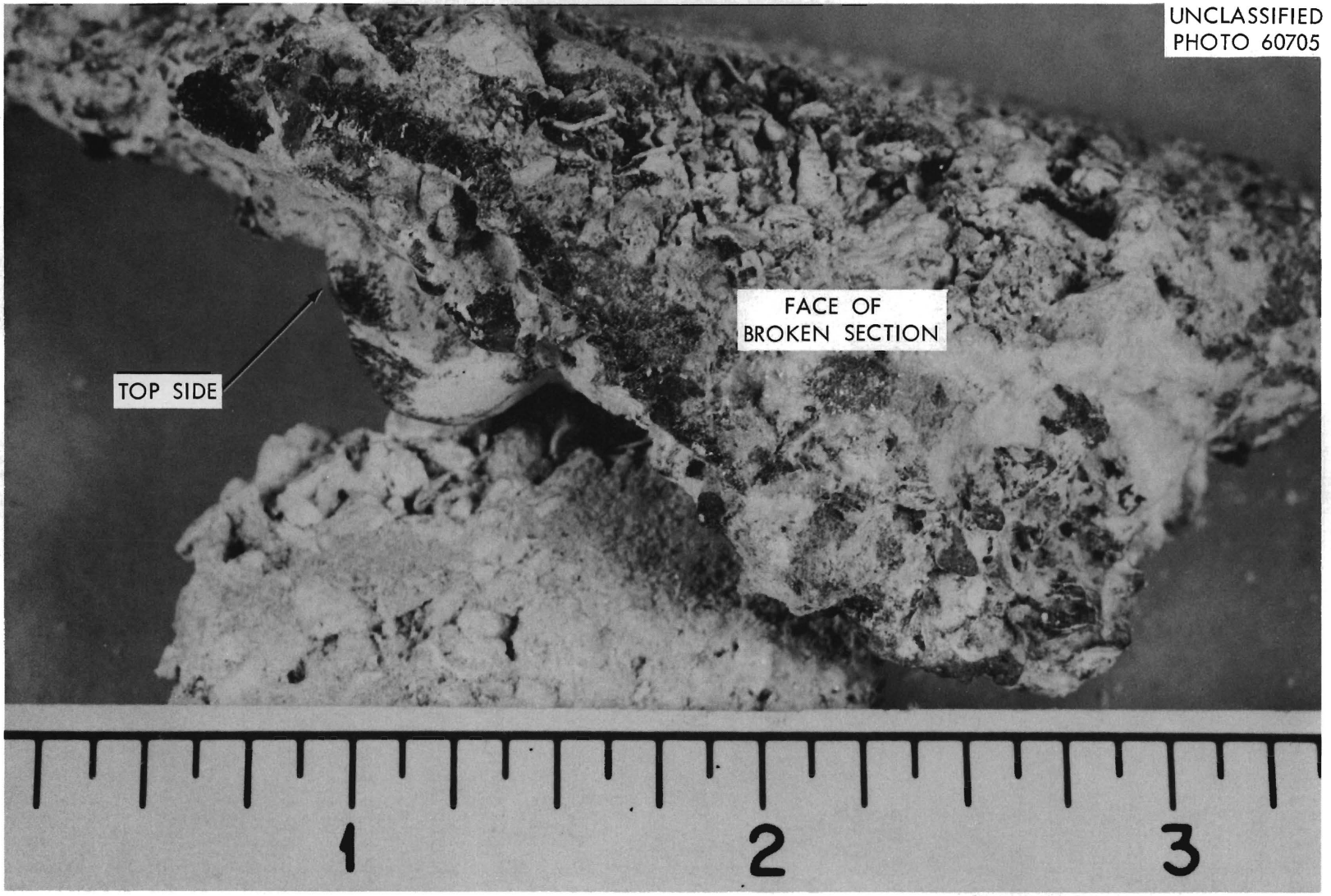

Fig. 2. Edge View of Broken Cake of Residue from Interrupted Fluorination, Run F-2.

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by x-ray diffraction, by wet chemical analysis, and by petrographic methods. It was not possible to define the composition by these methods, and grinding the entire mass to obtain a homogeneous sample was rejected because it was considered to be more important to determine whether or not the massive residue could be fluorinated. The analysis did indicate the presence of NbF_5, which is consistent with the manner in which we believe the residue was formed. Briefly, it is believed that NbF_5 condensed on the gas-cooling unit in the upper part of the reactor and refluxed back into the oxide residue. This reflux continued until enough heat had been removed from the reacting mass to practically stop the reaction, except at a few points where oxides were adjacent to the heated wall. The cementing material that closed the voids in the bed is NbF_5 and the products of reaction between NbF_5 and U_3O_8 and Nb_2O_5. A layer of white powdery crystalline material on top of the bed appeared to be NbF_5. A film of material of the same appearance was present on the fins of the gas cooler, and tracks of liquid droplets were evident in this film. The gas cooler was not being used as a cooling unit during fluorination but could only be heated by radiation from the wall of the vessel. Inspection of temperature recording charts after the run revealed that the bottom section of the cooler (nearest the reaction site) was 100°C below the boiling point of NbF_5 when fluorination started, and that the temperature rose while fluorination was proceeding.

Discussion of Second Part of Run F-2. After examination of the residue, which originally weighed 1450 g, the remaining 1401 g were returned to the reactor. In this part of the experiment, the normal support plate was removed, and the residue was placed directly on the bottom of the reactor. Additional heating was supplied so that the bottom flange could be kept at 425°C. The gas-cooler unit was removed from the reactor for this fluorination. Fluorine was admitted to the heated reactor, at 100% concentration, and the reaction started immediately and continued until the low (estimated) rate and calculations based on stoichiometry both indicated that the run was near completion. It was intended to continue fluorine treatment for a longer time, but a plug in the still column stopped all flows, and the run terminated.

The residue remaining in the reactor was a soft fluffy material with adequate surface for continued reaction. The residue weighed 108 g and contained 38.4 g of uranium. A large part of the remaining uranium could have been recovered if the run had continued.

2.3.2 Summary of Both Parts of Run F-2

A summary of the fluorine flow rates, times, and total quantity for both parts of the run F-2 is shown in Table 2.

Table 2. Log of Fluorine Use, Run F-2

100% Fluorine (no dilution) used throughout

Stoichiometric requirement: 58.0 moles
Total use: 132.7 moles
F_2 utilization efficiency: 43.8%

Date	Time	Period (min)	Rate (liters/min)	Volume Fed (liters/period)	Total Volume Fed (liters)
3/5/63	1145-1158	13	10.5	136.5	
	1158-1208	10	15.3	153.0	289.5
	1208-1216	8	18.8	150.4	439.9
	1216-1221	5	17.0	85.0	524.9
	1221-1228	7	15.0	105.0	629.9
	1228-1309	41	9.9	405.9	1035.8
	1309-1328	19	9.7	184.3	1220.1
	1328-1528	120	8.9	1068.0	2288.1
					(102.15 moles)
3/19/63	1246-1255	9	5.0	45.0	2333.1
	1255-1329	34	6.8	231.2	2564.3
	1329-1355	26	5.7	148.2	2712.5
	1355-1412	17	5.8	98.6	2811.1
	1505-1531	26	6.2	161.2	2972.3
					(Total 132.7 moles)

The stoppage that resulted in shutdown was caused by NbF_5 freezeout in the top of the distillation column. The capacity of the system is such that all the NbF_5 from runs F-1 and F-2 was required to build up an NbF_5 loading throughout the still. When this condition was reached, it was found that the system response to temperature control was slow and that the sensing of temperatures inadequate to maintain proper conditions at the still head. The entire system will be studied and revised.

2.4 Breaking Tests on Prototype Fuel

Breaking tests on temperature-cycled 19-hole hexagonal fuel rods indicated that 0.1 to 0.2% of the rods were reduced to fines, independent of both the blade energy over the range 12 to 40 in.-lb and the number of temperature cycles over the range 1 to 3. The amount of leachable uranium present in the broken fuel increased from 0.1% to 2.4% as the number of 2000°C cycles increased from one to three (Table 3). Evidently a more severe temperature cycle would be required to alter the breaking characteristics. None of the rods was lined.

2.5 Conclusions

1. The fluorination of dense materials of various compositions can be readily accomplished if the reaction zone is kept at 400° to 450°C.

2. The separation system needs study and modification.

3. Temperature cycling does not materially alter the graphite properties with respect to the production of fines in breaking to short lengths.

3. LABORATORY WORK IN SUPPORT OF THE COMBUSTION-FLUORINATION PROCESS

C. E. Schilling G. I. Cathers S. Katz

3.1 Thermogravimetric Fluorination of Partially Fluorinated Ash from Run RO-2: Study to Determine the Cause of the Incomplete Fluorination

Partially fluorinated RO-2 combustion ash was fluorinated in the thermobalance to determine possible causes for incomplete fluorination in the

Table 3. Guillotine Breaking of 19-hole Hexagonal Fuel (unlined) Containing Pyrolytic-Carbon-Coated UC_2 Particles

Temperature cycled rods nominally 54-in. long, reduced to 3/4-in. lengths by a 30° blade.

	Rod Numbers					
	1	2	3	4	5	6
Wt of fines, g	1.04	0.76	1.88	0.99	1.47	0.96
Total U, g	0.137	0.101	0.246	0.133	0.196	0.129
Leachable[a] U, g	0.028	0.025	0.034	0.042	0.034	0.032
Wt of Pieces, g	708	712	688	711	710	709
Total U, g	91.3	96.1	89.4	92.4	92.3	91.5
Leachable[a] U, g	0.067	0.058	0.041	0.052	0.572	2.16
Total U, g	91.4	96.2	89.6	92.5	92.5	91.6
Total leachable U, g	0.095	0.083	0.075	0.094	0.606	2.19
Total leachable U, %	0.10	0.09	0.08	0.10	0.66	2.4
Number of cycles[b]	1	1	1	2	2	3
Blade energy, in.-lb	12	20	40	12	20	20

[a]Leached 8 hr in 15.8 M HNO_3 at 95 to 100°C.

[b]175°C/hr to 2000°C, held at 2000°C for 4 hr. All elements had previously been graphitized at 1800°C for 4 hr.

last Unit Operations run (F-2, discussed on the preceding page). A relatively large sample (2.35 g) was selected to permit stepwise fluorination. Otherwise the method was similar to that described in a previous report.[2]

During the initial heating at 300°C in helium atmosphere, the weight change was less than 1 mg, thus ruling out the presence of NbF_5 (bp 233°C) in the sample tested. Cooling below reaction temperature by refluxing NbF_5 had been suggested as a possible cause for extinguishing the RO-2 reaction.

The fluorination step was initiated at 400°C and, as in previous tests, proceeded normally. The first weight loss occured after 3 min (the time calculated for displacement of the helium) and quickly became too rapid to follow, meanwhile the temperature rose to 425°C due to the heat of the reaction.

To permit examination of the partially fluorinated sample, the fluorine was turned off after 16 min of fluorination, and a helium flush was started. Weight loss continued for an additional 2.5 min, at which point the reaction was 28% complete.

The sample was then removed from the thermobalance and examined at 15X under the microscope. The main chunk appeared unchanged, with large, dark-gray grain fields in a lighter-gray matrix, with the latter showing preferential attack. Light-colored fluffy deposits similar to previously observed ashes were dispersed over the surface of the main piece and the floor of the basket. As noted in all previous fluorinations, the final ash did not glaze over, but spalled off as a loose powder.

The final fluorination was done with variations in fluorine feed rates to demonstrate control of the reaction by this means. The steady-state weight loss rate as given in Table 4 for the corresponding sequential fluorine flows demonstrate the qualitative control obtained.

In the final 4% of the fluorination the temperature was gradually raised to 570°C to overcome the effect of the diminishing surface as the reaction approached completion. It appeared almost complete somewhat below 570°C. The temperature was not held long enough at intermediate

[2] F. L. Culler, Rover Report for November 1962, ORNL-TM-443.

levels to ascertain the lowest temperature to achieve complete fluorination. The data here appears to support the maximum temperature requirement previously reported, 530°C.

Table 4. Control of Reaction Rate by Fluorine Flow

Fluorine Flow (Std cc/min)	Weight Loss (mg/min)
16	7.7
8	3.6
16	8.1
28	13.1

The final fluorination yield of 99.8% on the tested sample was achieved in a normal manner, and indicates that the cause of the incomplete fluorination of RO-2 was not due to its chemical composition. Further, it was shown that the reaction rate responded well to fluorine control.

These experiments clearly indicate that the shutdown of the Unit Operations run (in which a two-part fluorination was required in order to fluorinate the ash) occurred because the temperature had dropped below 400°C. The second conclusion is that temperatures in excess of 500°C are required at the end of the run in order to achieve greater than 99% volatilization of the uranium as the hexafluoride. The third conclusion is that the reaction rate will respond well to control of the fluorine flow, though some time must be allowed for steady-state to be established.

AQUEOUS PROCESSES FOR RECOVERING URANIUM FROM ROVER FUEL COMBUSTION ASH

L. M. Ferris K. S. Warren

.1 Development of the Deline-Burn-Dissolve Flowsheet

The Deline-Burn-Dissolve (DBD) process involves dissolution of the NbC liners in hydrofluoric-nitric acid solution, washing the fuel thoroughly with water, burning the delined and washed fuel in oxygen at 800 to 1000°C, and dissolving the U_3O_8 ash in nitric acid.

4.1.1 Recovery of Niobium from Delining Solutions

With the new coated-particle Rover fuel, it is possible that the delining solution will contain only an insignificant amount of uranium. Should this be so, recovery of the niobium before the bulk of the solution is transferred to the waste tanks might be economically attractive. One method for recovering the niobium from hydrofluoric-nitric acid solutions containing little or no uranium is extraction with hexone.

Solutions, with hydrofluoric acid concentrations varying from 2 to 7 M, and nitric acid concentrations varying from 2 to 8 M, each containing 22 ± 3 g of niobium per liter and less than 3 ± 1 g of uranium per liter were prepared by delining sections of Kiwi-B1A fuel. Each solution was equilibrated at 26°C with an equal volume (5 ml) of hexone (methyl isobutyl ketone) for 2 min before the phases were separated and analyzed. Extraction coefficients (E_a^o = concentration in hexone layer/concentration in aqueous layer) were determined for both niobium and uranium. The data (Table 5 and Fig. 3) show that in solutions where the hydrofluoric acid concentration is 6 to 7 M, the extraction coefficient for niobium is 0.4 to 0.5, while that for the uranium is less than 0.03. These data suggest that niobium can be recovered from delining solutions by the use of multi-stage extraction, and, furthermore, a reasonably good separation from uranium can also be made.

4.1.2 Volatility of Fission Products During Combustion of Irradiated Rover Fuel

Equipment for use in studying the volatility of fission products during the combustion of Rover fuel has been assembled and cold-testing initiated. Under the present schedule, the combustion experiments will be conducted in the low-level cell in Building 4505, starting about May 1, 1963, immediately following Chloride Volatility experiments now in progress.

[redacted]

Table 5. Extraction of Niobium and Uranium from Hydrofluoric-Nitric Acid Delining Solutions with Hexone

Each solution contained 22 ± 3 g of niobium per liter and less than 3 ± 1 g of uranium per liter; 5 ml of hexone and aqueous solution were equilibrated for 2 min before analysis.

Sample	Concn. (M)		E_a^o at 26°C	
	HNO_3	HF	Nb	U
1	2.43	2.32	0.094	0.006
2	6.08	2.62	0.16	0.059
3	7.95	2.55	0.12	0.028
4	2.48	4.38	0.30	0.003
5	4.29	4.42	0.40	0.019
6	6.10	4.50	0.13	0.026
7	7.97	4.63	0.11	0.0008
8	2.58	6.18	0.50	0.036
9	4.26	6.34	0.50	0.022
10	6.05	6.55	0.39	0.027
11	7.82	6.28	0.40	0.002

[redacted]

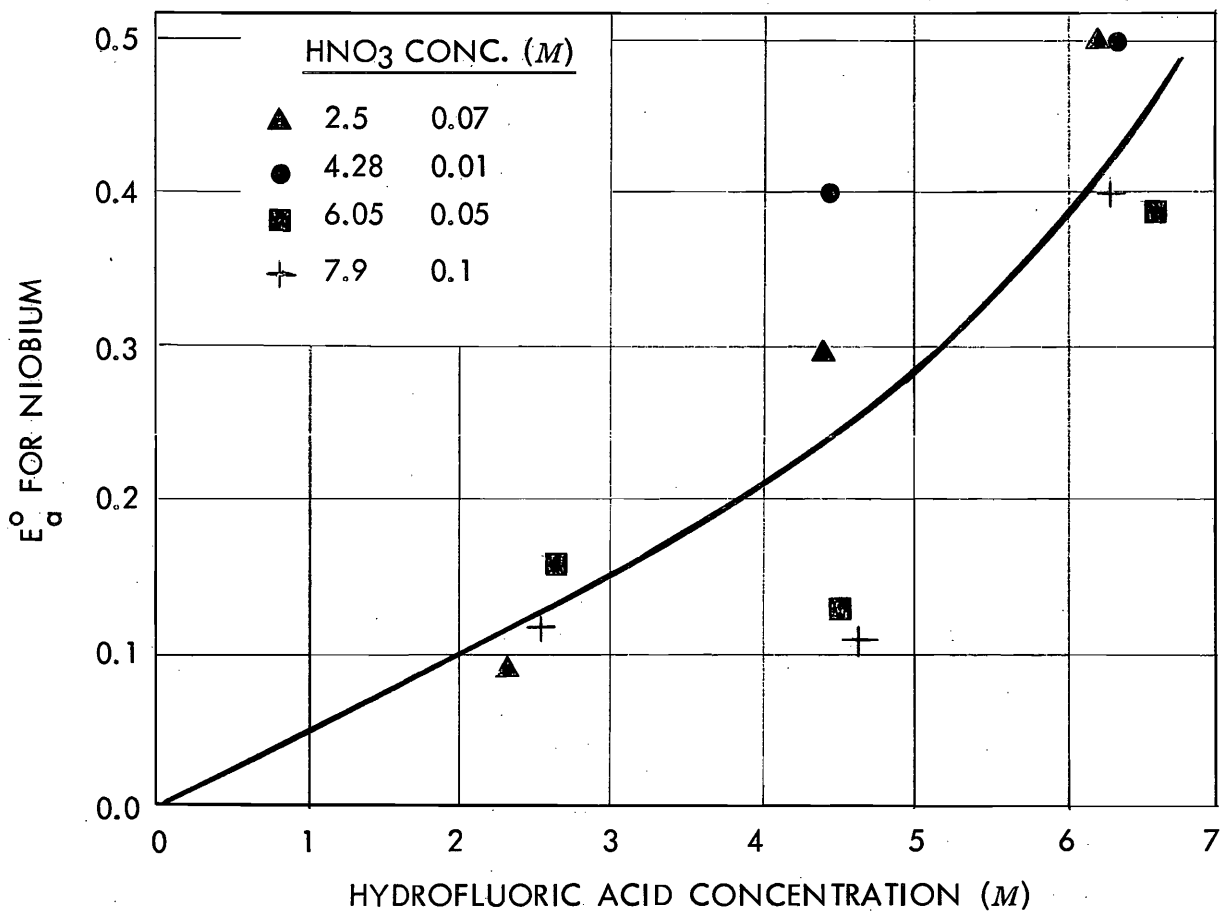

Fig. 3. Effect of Hydrofluoric and Nitric Acid Concentrations on the Extraction of Niobium from Hydrofluoric-Nitric Acid Solutions with Hexone at 26°C. Equal volumes (5 ml) of aqueous and organic phases were equilibrated for 2 min. Each aqueous solution originally contained 22 ± 3 g of niobium per liter and 3 ± 1 g of uranium per liter.