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REPROCESSING OF ROVER AND PLUTO FUELS:
INTERIM PROGRESS REPORT

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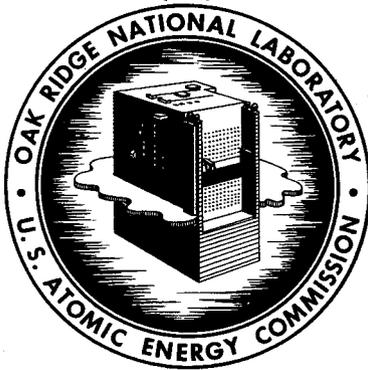
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CHEMICAL TECHNOLOGY DIVISION

REPROCESSING OF ROVER AND PLUTO FUELS: INTERIM PROGRESS REPORT

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Captain, United States Air Force, on loan from
Air Force Institute of Technology

DATE ISSUED

MAY 2 1958

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
operated by
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ABSTRACT

Uranium can be quantitatively removed from U-graphite particles ranging in size from below 0.149 mm to 1.19 mm by leaching with boiling 70% HNO_3 . However, grinding on a plant scale and subsequent handling of the large solid residue precludes recommendation of this method as the sole pre-treatment of irradiated U-graphite for solvent extraction.

Uranium apparently can be quantitatively removed from U-graphite by a combustion- HNO_3 digestion technique. Part of the residue which remains after HNO_3 digestion is soluble in boiling 30% HCl . The insoluble portion of the original material after HNO_3 - HCl digestion amounts to about 0.06%. The solids handling problem is greatly simplified by this technique. However, selection of a material of construction resistant to combustion conditions and boiling HNO_3 and HCl will undoubtedly present the most formidable engineering problem. Complete evaluation of this method is not possible until more quantitative data are obtained.

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1.0 INTRODUCTION

The purpose of this report is to give progress on two potential methods for extracting uranium from spent uranium-graphite fuel elements. The two methods reported herein, "grinding-leaching" and "combustion-digestion," were originally outlined in a previous memo.¹ Neither of these methods can be considered unique since variations of both have been employed in the past to reclaim uranium from salvaged graphite crucibles and other forms of scrap.^{2,3,4,5} As a matter of fact, combustion-digestion is presently used for this cold scrap salvage on a small plant scale basis at Y-12.⁶

The need for this study was established when ORNL assumed the responsibility for developing reprocessing methods for fuels fabricated for the Rover and Pluto programs. However, this responsibility may be broadened somewhat with the advent of reactors designed for project "TREAT" since it is known that these reactors will use fuel elements similar to those designed for Rover.^{7,8} This responsibility may also be extended to include high temperature power reactors designed to use U-graphite fuel elements.

Thus far, experimental effort has been confined to extraction of uranium from prototype elements designed and fabricated by Los Alamos Scientific Laboratory for the Rover reactor. Insofar as can be determined, the material being studied is physically similar to the elements which will be used in the first reactor assembly. These prototypes presumably differ from elements to be used in the reactor only in that they contain normal or depleted rather than enriched uranium. This report contains preliminary data obtained with the prototype fuel from both the grind-leach and combustion-digestion techniques.

The author wishes to acknowledge the groups of G. R. Wilson, W. R. Laing and R. L. Sherman of the ORNL Analytical Chemistry Division for the chemical and x-ray analyses. Appreciation is also extended to L. M. Ferris for his helpful comments.

2.0 GRIND AND LEACH TECHNIQUE

2.1 Results

The original U-graphite material was analyzed several times by different methods. The results appear in Table 2.1. It is seen that the various methods of analysis yield a value of 5.20 ± 0.20 wt % for the uranium content. The spread in uranium analyses corresponds to an analytical precision of $\pm 4\%$, which is probably within the practical limits of the methods employed. Heterogeneity of the U-graphite could conceivably account for some of the scatter in analyses.

Table 2.1 Analyses of U-graphite Prototype Fuel

| % U | % C | % Fe | % Insol. Residue | Method of Analysis |
|------|-------|------|------------------------|--|
| 4.99 | --- | --- | --- | Carbonate fusion. U by colorimetric analysis. |
| 5.2 | --- | --- | --- | Combustion of graphite. Pyrosulfate fusion of residue, U by colorimetric analysis. |
| 5.35 | 93.22 | 0.19 | 0.056 | Combustion of graphite. Digestion of residue in HNO_3 . U by colorimetric method. Fe by gravimetric method. Sample prepared for analysis by the author. |
| 5.40 | 93.83 | 0.23 | --- | Calculated using IASL loadings of 0.100 g U/cc; C density = 1.75. % Fe = avg. of 4 analyses. |

Found in Table 2.2 are the results of 9 grind-and-leach experiments. The average uranium recovery was 99.25% for a 1-hr leaching period. No measurable difference in uranium recovery was obtained when the particle size was varied from <0.15 mm to 1.2 mm. It should be noted that the uranium content of the original material, computed from the uranium analyses of the filtrate and the residue, is 5.31 ± 0.18 wt %, which is in good agreement with the value obtained by independent analyses (Table 2.1).

2.2 Discussion of Results

The results of the third run listed in Table 2.1 suggest that the combustion-digestion technique is a simpler method for preparing U-graphite material for standard uranium analysis. It is considered that the data pertinent to this method are insufficient to warrant a recommendation for changing

Table 2.2 Results of Leaching U-Graphite with 70% HNO₃ for 1 hr at 120°C

| Run | Particle Size, mm | Weight of Sample, g | Weight of Residue, g | Weight Loss, g | Uranium in Filtrate, g | Uranium in Residue, g | U in original Sample, % | % Uranium Recovered | Fe in Filtrate ^a , g | Fe in Residue ^b , g | Fe in original sample, % |
|------|-------------------|---------------------|----------------------|----------------|------------------------|-----------------------|-------------------------|---------------------|---------------------------------|--------------------------------|--------------------------|
| 1 | <0.149 | 10.0006 | 9.3848 | 0.6158 | 0.5100 | 0.014 | 5.24 | 97.33 | | | |
| 2 | <0.149 | 10.0007 | 9.3812 | 0.6195 | 0.5150 | 0.0007 | 5.16 | 99.86 | | | |
| 3 | <0.149 | 10.0002 | 9.3831 | 0.6171 | 0.5250 | 0.0003 | 5.25 | 99.94 | 0.0235 | 0.002 | 0.255 |
| 4 | <0.149 | 7.3000 | 6.8536 | 0.4464* | 0.3800* | 0.0003* | 5.21 | 99.92 | 0.0155* | 0.0095* | 0.342 |
| 5 | >0.149 <0.250 | 10.0001 | 9.3959 | 0.6042 | 0.5200 | 0.011 | 5.31 | 97.93 | | | |
| 6 | >0.149 <0.250 | 10.0002 | 9.3948 | 0.6054 | 0.5400 | 0.0013 | 5.41 | 99.76 | 0.0230 | 0.003 | 0.260 |
| 7 | >0.149 <0.250 | 10.0002 | 9.4059 | 0.5943 | 0.5125 | 0.0014 | 5.14 | 99.73 | 0.0220 | 0.006 | 0.280 |
| 8 | 0.59 to 6.35 | 10.0000 | 9.4017 | 0.5983 | 0.5450 | 0.0037 | 5.49 | 99.32 | | | |
| 9 | >0.59 <1.19 | 10.0002 | 9.3929 | 0.6073 | 0.5450 | 0.0020 | 5.47 | 99.63 | | | |
| Avg. | ---- | ---- | ---- | 0.6077 | 0.5265 | 0.0039 | 5.30 | 99.25 | 0.0228 | 0.0037 | 0.264 |

^aSpectrographic analysis.

^bRoutine laboratory analysis.

* These figures were omitted in computation of the final averages.

procedures at this time. However, work in this area is progressing, and if combustion-digestion proves most consistent, it will be recommended for use, at least in connection with this program.

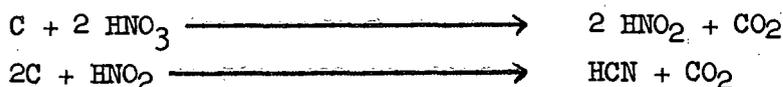
The comparatively low percent removal of uranium in runs 1 and 5 may be primarily attributed to the fact that these were run first to set up standard procedures for all other runs. In these runs, more attention was devoted to physical manipulation of the apparatus than to obtaining quantitative data. Errors incurred in this manner, when combined with errors inherent in analytical procedures, could easily explain these low results. However, these data were used in computing the average recovery without greatly detracting from the final average.

Run number 8 differed slightly from the others with respect to selection of particle size. Random sized particles were used, and though no attempt was made to determine exact proportions, it was estimated that the larger particles (6.35 mm) amounted to no more than 5% of the total sample. Smaller particles, approximately 0.60 mm to 1.0 mm, constituted the greater portion of this sample. This random size was chosen simply because it was available material which remained after grinding and sieving.

The percent weight loss exceeded the percentage of uranium removed by an average of 0.81%. All but a small portion of this excess can be attributed to the removal of iron along with the uranium. This statement can be supported by the following material balance:

| | | |
|--|---|-------|
| UC ₂ in original sample computed from average U in filtrate | = | 5.79% |
| Avg. Fe in filtrate = Fe in original sample | = | 0.23% |
| <hr/> | | |
| Avg. total UC ₂ + Fe removed | = | 6.02% |
| Avg. weight loss | = | 6.08% |
| Difference | = | 0.06% |

This small difference could be explained on the basis of the following reactions of graphite with HNO₃



or it can be attributed to experimental error. It does appear, however, that the iron present in the original material exists in the metallic state.

The prime conclusion from the data given in Table 2.2 is that uranium can be quantitatively leached with nitric acid from U-graphite particles ranging in size from below 0.15 mm to 1.2 mm.

It was also found that a considerable quantity of extremely fine, dust-like, particles will be produced while grinding the original U-graphite

plates. The fact that these fine particles are produced even by crude size reduction methods (striking, rubbing, etc.) implies that, on a large scale, a sophisticated facility will be required to reduce uranium losses due to dusting in addition to reducing radiation and spontaneous combustion hazards. Graphite, if finely divided, may be a spontaneous combustion hazard, especially when intrinsic heat from fission products is considered.

Despite the excellent uranium recovery, the bulk of the original material remains as an insoluble graphitic residue after nitric acid leaching. This becomes most significant when an attempt is made to visualize this process on an engineering scale. In this regard, the bulk residue perhaps should receive further treatment, such as controlled combustion, before disposal. Not only would this free the equipment for further use, but may also simplify the problem of solids handling. In any event treatment of the bulk residue is germane to handling the irradiated material on any scale.

The grind-leach method cannot be recommended as the sole pretreatment of U-graphite material unless an acceptable method for removing and disposing of the large residue is found. Several attempts to remove this residue from laboratory equipment are outlined as follows:

1. Mechanical removal
2. Fusion of residue with NaOH, NaCO₃, etc.
3. Chemical dissolution
4. Burning

All these methods were successful to a degree. Mechanical removal from laboratory scale equipment worked well except that the fine particles had to be removed from filters with chromic acid (cleaning solution), and only a small amount was removed over a considerable period of time. Fusion with NaCO₃ was abandoned after one attempt because it was not compatible with fritted glass filters. Fusion with NaOH is yet to be tried but may prove impractical on anything but a laboratory scale because of the large amounts of material which the equipment must accommodate. Chemical dissolution was attempted once. The method used was an adaption of an analytical technique employed by industry to determine the degree of graphitization of carbon in graphite products.¹⁰ This technique utilizes H₂SO₄, HNO₃, and KClO₄ to convert the carbon to soluble graphitic acid. Because of the extremely slow reaction rate and the hazardous, corrosive effects of this reaction, it is not recommended for use even though it was moderately successful. Burning of the leached residue, per se, has not been attempted, but should offer no severe problems.

3.0 COMBUSTION-DIGESTION TECHNIQUE

3.1 Results

Four attempts were made to oxidize the LASL material in a stream of pure oxygen. These experiments were conducted primarily to standardize a procedure for this operation rather than to obtain quantitative data; some data which are considered reliable were obtained and are presented here. The results of these combustions are listed in Table 3.1.

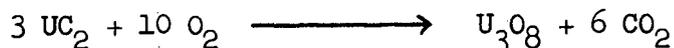
Table 3.1 Combustion of U-Graphite

| Run | Wt of Sample, g | Weight of Residue After Combustion, g | Wt Loss, g | Wt Loss, % | X-ray Analysis of Residue | Temp, °C | O ₂ Flow Rate, ml/min |
|-----|-----------------|---------------------------------------|------------|------------|--|----------|----------------------------------|
| 1 | 20.4819 | 1.3850 | 19.0969 | 93.24 | ---- | 700 | 400 |
| 2 | 20.3568 | 1.3596 | 18.9972 | 93.32 | ---- | 700 | 400 |
| 3 | 20.2928 | 1.3498 | 18.9430 | 93.35 | U ₃ O ₈ + Fe ₂ O ₃ | 700 | 400 to 1700 |
| 4 | 22.6261 | 1.5339 | 21.0922 | 93.22 | ---- | 700 | 1900 |

3.2 Discussion of Results

In every case, the reaction of the constituents with O₂ proceeded to completion without violence. Also, in every case the sample was surrounded by a blue corona during combustion. At low flow rates, 400 ml/min to 700 ml/min, the corona was slightly less distinct than at the higher rates. However, disappearance of the corona proved a reliable method for determining the end point of the reaction and was used for that purpose. In no case was dusting of the material observed during combustion.

The equation for the combustion appears to be:



The consistency of the percent weight loss indicates a high degree of homogeneity for this material. Similar results were obtained in an earlier study.¹ However, it must be pointed out that these samples represent only a small portion of one plate of the original material. It is felt that samples representative of the entire loading range would have to be run to determine the limits of homogeneity.

The low percent uranium obtained by digestion of the residue from run 1 (Table 3.2) was due to incomplete digestion of the residue. As a consequence, the procedure was modified, as described for run 4 (Section 4.2).

On the basis of run 4, the residue which remains after HNO_3 digestion amounts to only 0.3% of the total sample. However, this second residue represents the total solids which remain in the equipment after combustion-digestion has taken place. From the x-ray analysis of the combustion residue (Table 3.1), indications are that this second residue is composed almost entirely of Fe_2O_3 . No analysis of the second residue was made to determine whether or not uranium was present; rather, emphasis was placed upon determining how to remove the Fe_2O_3 residue from the equipment. The combustion residues from runs 2 and 3, as well as 4, were treated with regard to these latter two considerations.

From runs 2 and 3, it was observed that the Fe_2O_3 , in extremely fine particulate form, was dispersed throughout the boiling HNO_3 used to digest the U_3O_8 from the combustion residue. Due to passivity or insolubility, little of the Fe_2O_3 was dissolved with the U_3O_8 (Table 3.2).

When caught on a medium-porosity fritted glass filter, the fine particles of Fe_2O_3 became imbedded in the fritted portion, which precluded complete mechanical removal. As a result of various experiments with this second residue, the most successful method of removing the Fe_2O_3 was to boil the filter and residue in 30% HCl for about 45 min. Although this resulted in complete dissolution of the Fe_2O_3 , a third residue remained after filtering the solution which resulted from HCl digestion. This final residue was found to be insoluble in both dilute and concentrated HNO_3 , HCl , and aqua regia.

Due to the qualitative approach adopted for these four runs, the only attempt to weigh the third and final residue was in run number 4. In this instance, it was determined that the final insoluble residue constituted only 0.06% of the total sample. No attempt was made to remove this small quantity of material from the filter for x-ray or chemical analysis.

In summing up the combustion-digestion procedure, combustion of the U-graphite material in O_2 results in a 93.3% reduction of original bulk. This reduction is due entirely to the complete burning of the graphite during an extremely smooth reaction. Digestion of the combustion residue with 70% HNO_3 results in a second residue, but removes all but 0.3% of the original bulk. Digestion of the second residue with 30% HCl removes all but 0.06% of the original bulk but results in a third most insoluble residue.

Combustion-digestion appears quite feasible for use in the pre-treatment of U-graphite material for solvent extraction, mainly from the standpoint that it simplifies solids handling problems. However, this method imposes problems of selecting materials resistant to high-temperature and acid corrosion and radioactive off-gas treatment. In the event that the grind-leach pre-treatment

Table 3.2 Digestion of Combustion Residues

| | <u>Run 1</u> | <u>Run 4</u> |
|--|--------------|--------------|
| Weight of Sample ^a (g) | 1.3850 | 1.5339 |
| Weight of HNO ₃ -Leached residue (g) | 0.3028 | 0.0736 |
| Weight of U ₃ O ₈ + Fe Removed (g) | 1.0822 | 1.4603 |
| U in Filtrate (g) | 0.8878 | 1.2100 |
| Fe in Filtrate (g) | 0.0043 | 0.0047 |
| Fe in Residue (g) | 0.0386 | 0.0427 |
| Percent U in Original Sample | 4.33 | 5.35 |
| Percent Fe in Original Sample | 0.21 | 0.21 |

^aResidue left after combustion (see Table 3.1).

previously discussed is preferred, combustion-digestion would lend itself quite readily to the disposition of bulk residues, especially on a batch basis.

4.0 EXPERIMENTAL PROCEDURE

4.1 Grind and Leach

Uranium-graphite, in the form of plates approximately 8" x 11" x 3/8", was taken as received from IASL and cut with a saw into pieces which fit into a large mortar. These pieces were then ground into particles ranging from below 0.149 mm to 6.35 mm as determined by sifting through standard sieves.

Pyrophoricity of UC₂, as described by Katz and Gray,^{11,12} was not observed even though the material was treated rather severely in the grinding process. The low concentration of UC₂ in excess graphite may have reduced this hazard. However, this phenomenon should not be entirely eliminated from consideration when and if routine grinding becomes necessary.

Samples of ground material, weighing about 10 g, were refluxed for one hour in 75 ml of 70% (15.8 N) HNO₃ at 120°C. After refluxing, the residue and leachings were transferred to a weighed medium-porosity fritted glass filter by washing with about 75 ml of distilled H₂O. While on the filter, the residue was washed with four successive 25-ml portions of H₂O. Finally, the residue was vacuum dried on the filter for one hour or until a constant weight was obtained. After the final weighing the residue was analyzed

for uranium. In several cases, analyses for iron and other constituents were obtained.

The filtrate was transferred from the filter flask to either a 250-ml or a 500-ml volumetric flask. (The size of the flask depended mainly on the amount of H₂O required to wash the material from the reflux apparatus into the filter.) It was diluted to either 250 ml or 500 ml, sampled, and analyzed for uranium. In several instances, the filtrate was analyzed for iron and other constituents.

4.2 Combustion-Digestion

U-graphite, as received from LASL, was cut into pieces about 8 cm long by 2.5 cm wide. These samples, weighing approximately 20 grams each, were burned in either a porcelain or platinum boat inserted into a quartz tube. A quartz tube containing the boat was inserted into a resistance-wound tube furnace and the ambient temperature was raised to 700°C. Concurrent with the elevation of temperature from approximately 23°C (room temperature) to 700°C; a flow rate of about 400 ml/min of dry N₂ was maintained in the apparatus. All samples were allowed to soak at 700°C for about 15 min; then the N₂ was cut off and dry O₂ was introduced at flow rates no lower than 400 ml/min up to 1900 ml/min in order to check dusting characteristics. No energy, other than that required to provide the constant ambient temperature of 700°C, was added to the system while flow rates were varied.

The residue from run 1 (Table 3.2) was leached with 4 successive 25-ml portions of warm 70% HNO₃. Each portion remained in contact with the residue for 10 min before filtration. After leaching, the remaining solids were washed with 100 ml of water.

The residue from run 4 (Table 3.2) was digested for 45 min with 100 ml of boiling 70% HNO₃. About a 25-ml volume reduction occurred during digestion. After filtration, the remaining solids were washed with 100 ml of water.

5.0 FUTURE WORK

The experiments to determine the criteria of optimum particle size for leaching with HNO₃ will continue. Also, since burning of leached residues may become desirable, several experiments will be conducted to obtain data on this operation.

Combustion-digestion of U-graphite appears promising as a simple technique for preparing this material for standard laboratory analysis. Data to support this hypothesis may be derived from future runs. In addition, the problem of continuously removing the small amount of residue which remains after combustion-HNO₃ digestion emphasizes the need for corrosion resistant materials in the laboratory apparatus. The equipment will be modified in an attempt to supply the solution. Finally, an attempt will be made to adapt these methods to flowsheet conditions.

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