

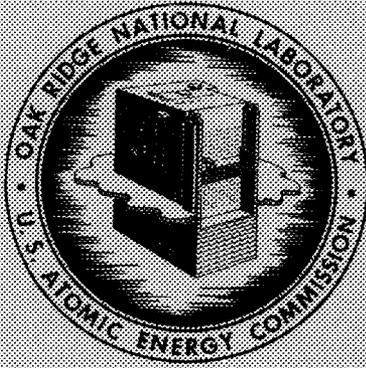
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ROVER FUEL PROCESSING: SUMMARY TO FEBRUARY 28, 1962

F. L. CULLER, JR.

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CHEMICAL TECHNOLOGY DIVISION
ROVER FUEL PROCESSING: SUMMARY TO FEBRUARY 28, 1962

F. L. Culler, Jr.

DATE ISSUED

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ABSTRACT

Progress in chemical and engineering studies of Rover fuel processing is reported. In the grind-leach process, there was no difference between feeding intact rods or sheared pieces to a jaw crusher (at least 50% yield of 4-12 mesh material in both cases). Greater than 99% uranium recovery was effected by leaching for six hours -8 mesh prototype fuel with 4-10 M HNO_3 containing 0.3-0.5 M HF. In similar reagents containing ~ 0.7 M Al^{3+} uranium losses exceeded six percent since niobium was not dissolved. After dissolution, niobium separation from uranium by precipitation was complicated by the formation of a slimy hydrous oxide. Plastic equipment of Teflon or Fluorothene will probably be required for present aqueous leaching flowsheets. In the combustion-dissolution process, boiling 1-10 M HNO_3 containing 1-5 M HF is required for Nb_2O_5 dissolution. In chloride volatility studies, uranium recovery was improved by treatment of ground fuel with chlorine at 800°C compared with 700°C . At 800°C , niobium carbide was severely attacked.

1.0 INTRODUCTION

The objectives of the Rover fuel processing program are (1) to develop chemical and engineering information on several processing methods and (2) to select the best process for preliminary plant design and cost estimation at a scale of ~20 kg of enriched uranium per day. The earlier work was reported in ORNL-3196 and 3283*.

2.0 GRIND-LEACH PROCESS

In the current grind-leach technique being investigated the fuel is ground and then digested with nitric acid to leach most of the uranium or with nitric acid containing another reagent to dissolve both the niobium carbide liner and the uranium. Uranium is recovered from the solution by solvent extraction.

2.1 Mechanical Size Reduction

Size reduction of several 3/4-in.-dia Rover prototypes** in a laboratory jaw crusher demonstrated that >50% yield of 4-12 mesh material can be produced, without excessive fines, by using a relatively large jaw opening and recycling the oversize material. Feeding an intact rod or sheared pieces to the crusher produced identical crushed material, 50% of which was 4-12 mesh (1680-4760 μ) (Fig. 1).

2.2 Leaching

2.2.1 Nitric-Hydrofluoric Acid Leachant

Aluminum nitrate, when added to niobium-uranium solutions containing fluoride, precipitates the niobium and complexes the fluoride. The precipitated niobic oxide is filtered off.

* L. M. Ferris and L. A. Kambach, "Uranium Recovery from KIWI Fuel Elements: Laboratory Development," ORNL-3196 (Dec. 4, 1961); also L. M. Ferris, T. A. Gens, and A. H. Kibbey, ORNL-3283 (in preparation) (preliminary record of same work in "Processing of Rover Fuel Elements: Laboratory Development for Period Sept. 1, 1961 - Jan. 15, 1962, internal memo CF-62-2-22).

** Same configuration as actual fuel but containing depleted U. These elements were ~27 in. long, graphitized, NbC lined (seven holes, no end coating) and unfluted.

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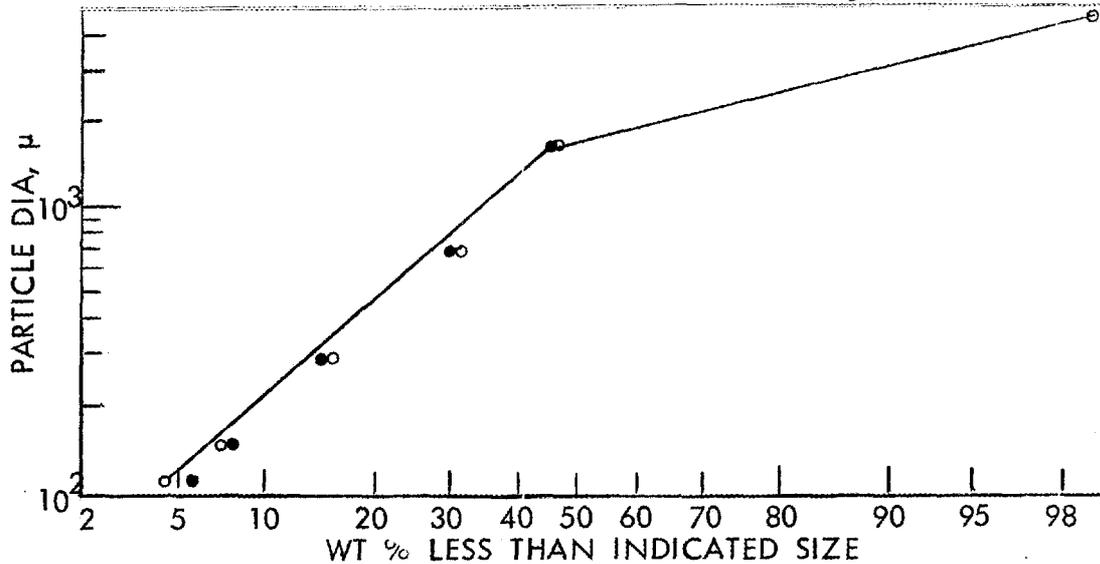


Fig. 1. Particle size distribution of Kiwi B-1B fuel crushed to -4 mesh in a laboratory jaw crusher. Distribution by sieving. One 300-g rod fed to crusher (○) intact and (●) as 1-in. sheared lengths.

Effect of Reagent Concentrations on Uranium Recovery. In laboratory studies, at least 99% of the uranium was recovered from -8 mesh prototype fuel with either 4 or 10 M HNO₃ containing 0.3-0.5 M HF when the F/total Nb mole ratio was 3.5-5 (Table 1). The fuel was digested 6 hr, the mixture was filtered, and 1.5 M Al(NO₃)₃ was added to the filtrate to give an Al/F mole ratio of 1. At least 35% of the niobium remained with the graphite residue, and the slimy niobic oxide precipitate formed on addition of the aluminum nitrate retained <0.25% of the uranium. A serious disadvantage was the difficulty of filtering the hydrous niobic oxide and the instability of solutions in which the F/Nb mole ratio is <10.

In nine laboratory-scale batch leachings with 4-23 M HNO₃ containing 0.5 M HF, uranium losses to the residue were <0.5% after 3 hr with a large excess of leachant (Table 2). With 0.2 M HF, losses were 2.5-3%, indicating that this reagent might be effective but a longer leaching time would be required. In control run with 0.66 M Al-0.44 M HF-13 M HNO₃, for comparison with results in the next section, the uranium loss was 26.3%. The fuel leached was 2- to 2.5-g portions of the Nb-rich 25-50 mesh fraction obtained by screening crushed prototype fuel.

Table 1. Leaching of -8 Mesh KIWI-B Fuel with Boiling Nitric Acid-Hydrofluoric Acid

Leaching time: 6 hr

Run No., GL-	Fuel Comp., %		Leachant		F/Nb Mole Ratio ^a	Recoveries, %					
	U	Nb	HNO ₃ , M	HF, M		Solution ^b		Ppt.		Residue	
						U	Nb	U	Nb	U	Nb
XIII	16.7	15.9	4	0.3	2.3	98.3	6.2	0.02	----	1.67	93.8
XII	20.0	13.5	4	0.3	5	99.3	50.8	----	----	0.74	49.2
XI	18.9	10.2	10	0.3	3	99.3	14.5	0.004	----	0.73	85.5
X	20.4	10.3	10	0.3	5	99.5	65.3	0.005	----	0.47	34.7
XXI	20.7	16.5	4	0.5	2.2	97.6	6.3	0.20	15.1	2.23	78.6
XX	22.4	16.9	4	0.5	3.6	98.6	9.4	0.24	19.5	1.16	71.1
XIX	22.5	16.5	10	0.5	2.2	99.1	3.4	0.045	1.7	0.89	94.9
XVIII	20.2	12.5	10	0.5	5	99.4	20.7	0.002	0.9	0.63	78.4

^aDefined as fluoride to total niobium in sample.

^bVirtually all aluminum remained in solution. In most cases, 1 mole of fluoride remained in the graphite residue for each mole of niobium present there, presumably as NbO₂F.

Table 2. Batch Leaching of 25-50 mesh Rover Fuel
(Nb-rich fraction) with Refluxing Nitric-Hydrofluoric Acid

Leaching time: 3 hr

Leachant vol: 300 ml

Run No.	Leachant		Sample			Product			Residue		
	HNO ₃ , M	HF, M	Wt, g	U, %	Nb, %	U, g	Nb, g	Vol, ^a ml	Wt, g	Nb, g	U Loss, %
8	23	0.5	2.043	14.1	55.1	0.287	1.125	330	0.962	0.0004	0.326
4	21	0.5	1.962	12.5	25.4	0.245	0.498	361	0.835	0.0007	0.188
6	15	0.5	2.357	12.25	31.2	0.288	0.734	335	0.874	-	0.114
1	10	0.5	2.663	26.0	31.9	0.691	0.850	298	1.135	0.0002	0.107
3	7	0.5	2.574	27.4	47.5	0.703	1.200	348	0.691	0.022	0.129
7	4	0.5	2.234	34.6	>15	0.771	0.335	352	1.1022	-	0.172
5	21	0.2	2.354	22.2	>14	0.510	0.326	367	1.6132	-	2.55
2	10	0.2	2.401	14.7	49.0	0.272	0.397	320	1.933	0.779	2.80
9 ^b	13	0.44	3.350	9.27	58.4	0.229	<0.0004	382	3.1423	1.958	26.3

^aIncludes wash water.

^bLeachant contained 0.66 M aluminum and was 340 ml volume.

In nine other laboratory-scale runs of 4-6 hr, uranium losses were <1% when the HF was 0.5 M, but increased above 1% as the HF concentration was increased above 1 M. This effect was very pronounced with 24 M HNO₃ (~100%) where the uranium loss was almost 10%. The lack of water in the dissolvent may have been a factor in the 24 M HNO₃, but there is a direct comparison of 1.0 and 0.5 M HF at 23 M HNO₃ (~95%) with losses of 3.36 and 0.60%, respectively. The fuel samples used in runs 304, 305, and 307 were ~1-in. lengths of prototype fuel crushed to -8 mesh; about three times as much of the same material was used in run 309. A similar 1-in.-long piece of intact fuel (3/4 in. dia with seven ~0.15-in. lined holes) was used in each of the other runs. The initial uranium and niobium concentrations for each fuel specimen were calculated from the amounts found in the liquid and residue. The uranium percentages (nominally ~18%) are reasonable, but it is obvious that the niobium values are subject to question. (See Table 3.)

There was no correlation between the uranium loss and the niobium content of the residue. Unfortunately, in the residue analyses there is no distinction between niobium or uranium present as a carbide and niobium as an oxyfluoride precipitate or uranium not leached from the graphite matrix. However, this lack of correlation indicates that the uranium is not present as an unleachable uranium-niobium carbide. Also, there was no correlation between niobium in the residue and initial fluoride although there was a trend with the initial HNO₃. Residual niobium was high in runs 304 and 302, with 15.5 and 21 M HNO₃; niobium was much lower with HNO₃ concentrations both above and below this range. In run 304 the slightly higher uranium and niobium losses to the residue cannot be compared directly with the other results since three times as much fuel was leached in the same volume of dissolvent. The lower nitric acid concentration, higher fluoride or higher dissolvent loading could have contributed to this effect; the initial F/Nb ratio was 7.3, with a ratio of 5 necessary to supply the fluoride actually combining with all the niobium in forming the pentafluoride.

Table 3. Reflux Leaching of Representative Power Fuel Material

Run No.	Dissolvent			Fuel Specimen			Leaching Time, hr	Wt Loss, %	Liquid Product			Residue			Corrosion Rates mils/yr					
	HNO ₃ , M	HF, M	Vol, ml	Form	Wt, g	U, %			Nb, %	Vol, ml	U, g/l	Nb, g/l	Wt, g	Nb, g	U Loss, %	304 SS	304L SS	Worthite	347 SS	Al(2S)
304	15.5	0.5	300	-8 mesh	10.87	17.0	8.7	6	21.1	310	5.93	1.23	8.578	0.562	0.38					
305	10	0.5	300	-8 mesh	10.53	16.6	8.5	6	32.5	348	4.99	2.47	7.103	0.032	0.40					
307	5	0.5	300	-8 mesh	10.61	16.8	8.7	6	32.9	337	5.26	2.74	7.118	0.001	0.64		690			
309	4	0.85	300	-8 mesh	31.39	15.5	10.4	6	32.2	500	9.63	6.22	21.26	0.147	1.27					
302	21	0.5	250	1" cyl	11.80	22.4	5.7	4	18.3	290	8.09	0.76	9.646	0.453	0.86	3.4 4.8 ^a		130		
301	23	0.5	250	1" cyl	11.00	23.4	3.3	4	29.1	445	5.76	0.46	7.794	0.156	0.23	2.1			90	
303	23	0.5	250	1" cyl	12.48	16.3	1.8	4	30.3	304	6.63	0.30	8.703	0.035	0.60	3.4 3.6 ^a 4.8 ^a				
306	23	1.0	250	1" cyl	11.08	17.1	11.8	4	30.7	375	4.85	3.33	7.683	0.058	3.86	2.9 2.8 ^a	3.8			
308	24	1.0	250	1" cyl	11.22	16.8	6.5	4	28.8	305	5.59	2.30	7.996	0.031	9.35	0.39	0.37			0.35

^aDuplicates

^bIncludes wash water

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Addition of Aluminum Nitrate before Filtration. In laboratory scale experiments in which digestion in $\text{HNO}_3\text{-HF}$ was followed by addition of aluminum nitrate prior to filtration, at least 95% of the niobium remained in the graphite residue. Uranium recovery was >99% with a 6-hr $\text{HNO}_3\text{-HF}$ leach when the F/Nb mole ratio was >3.5 (Table 4). Increasing the digestion time to 20 hr in two experiments did not increase uranium recovery, and, on the basis of earlier work, a second leach with fresh reagent would probably be more beneficial than extended leaching. The experiments were conducted by digesting the sample in $\text{HNO}_3\text{-HF}$ for 6 or 20 hr. Then 1.5 M $\text{Al}(\text{NO}_3)_3$ was added to an Al/F mole ratio of 1 and digestion was continued for an additional hour. The product solution was then removed by vacuum filtration, and the residue was washed with 3 vol of cold water.

In a single semicontinuous engineering-scale run in which -4 mesh prototype fuel (Kiwi B-1B) was leached 6 hr at 100°C with 4.5 M $\text{HNO}_3\text{-}$ 0.75 M $\text{Al}(\text{NO}_3)_3\text{-}$ 0.50 M HF, 6% of the uranium was lost to the washed residue. The high loss was apparently due to the presence of aluminum, which prevented dissolution of NbC liner material.

In this run a 16-cm-deep bed of crushed prototype fuel was leached by a continuous downflow of reagent (Fig. 2). Reagent was metered to the top of the column at 5 ml/min, and a constant liquid level was maintained over the bed by regulating the receiver vacuum as required. Effluent was collected in the receiver in 100-ml increments. Samples of fuel residue were taken frequently from the base of the column by closing the large ball valve momentarily and "coring" the solids isolated in the bore of the valve.

After the first hour of contact (Table 5) the rate of uranium removal dropped rapidly. Water washing of the bed proceeded very slowly, although the wash flow rate was approximately double the acid flow rate. Samples of leached fuel (~4 mesh lumps) contacted overnight with water produced a yellow wash solution despite a prior water wash of 2-3 hr. Analysis of cored samples of fuel taken from the bottom of the bed at intervals during the acid leach showed a slow decrease in uranium concentration with time and higher uranium losses to the residue with increasing particle size (Table 6).

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Table 4. Leaching of -8 Mesh KIWI-B Fuel with Boiling Nitric-Hydrofluoric Acid Solutions

Aluminum nitrate added prior to filtration

Run No., GL-	Fuel Comp., %		Leachant		F/Nb Mole Ratio ^c	Leaching Time ^a , hr	Amt in Residue, %	
	U	Nb	HNO ₃ , M	HF, M			U	Nb
XVII	17.5	16.3	10	0.3	2.2	6	1.31	99.4
XVI	17.1	16.2	10	0.3	3.8	6	0.95	99.0
II	18.6	11.0	4	0.5	3	6	1.48	98.1
120	17.0	20.5	4	0.5	3	20	1.80	99.2
I	19.0	13.1	4	0.5	5	6	0.75	97.0
V	18.8	11.6	10	0.5	3	6	1.12	98.1
IV20	16.6	16.3	10	0.5	3.7	20	0.76	98.0
IV	19.7	10.1	10	0.5	5	6	0.89	95.4
XXII	19.3	13.0	10	0.5	5	6 ^b	0.48	98.9

^aLeaching time in HNO₃-HF only; sample leached an additional hour after Al(NO₃)₃ addition.

^bDigestion was continued for an additional 16 hr after addition of aluminum nitrate.

^cDefined as fluoride to total niobium in sample.

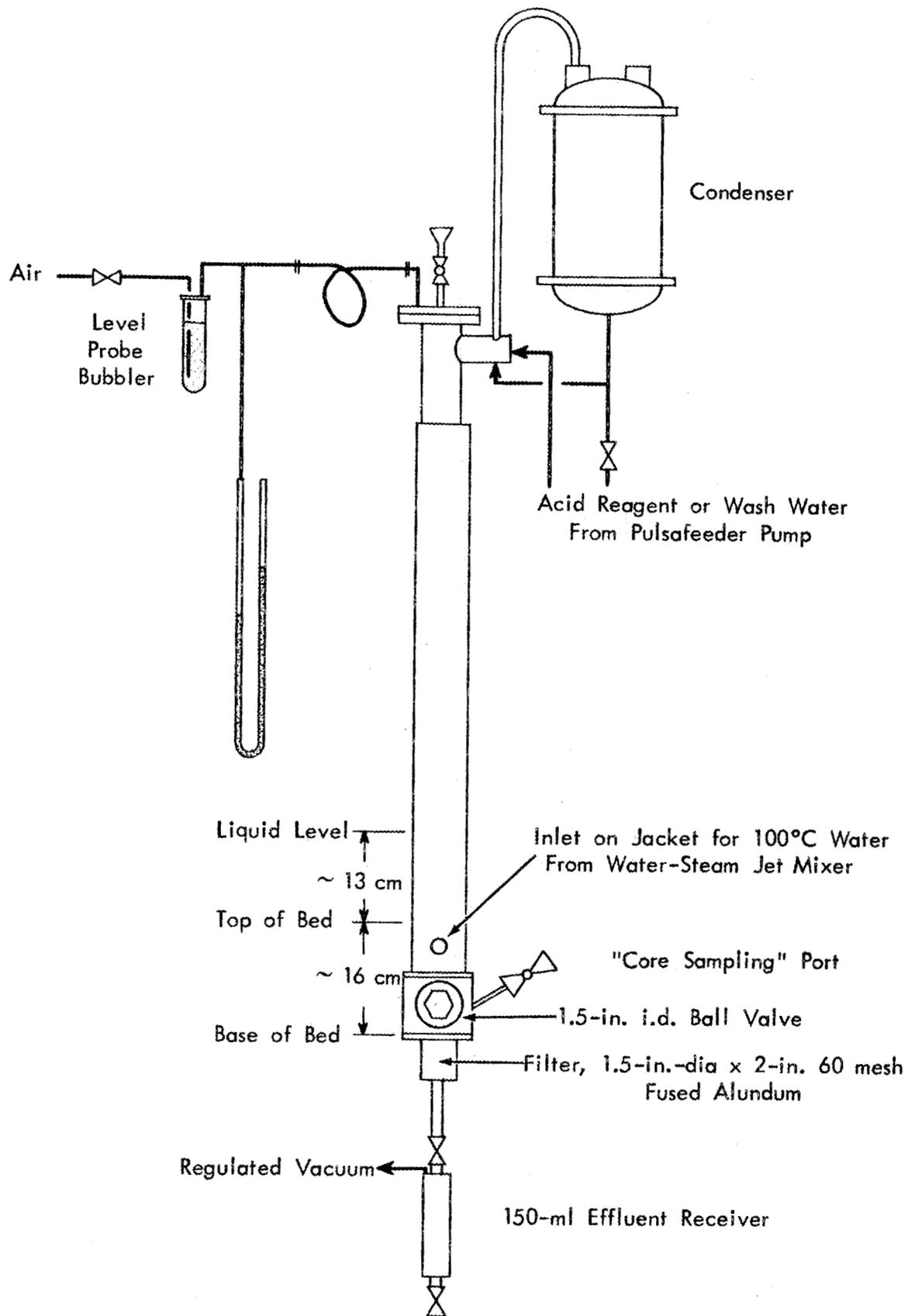


Fig. 2. Schematic diagram of jacketed 1.5-in.-i.d. x 36-in.-high downflow dissolver with provisions for periodic residue sampling.

Table 5. Fractional U Recovery vs Acid Contact Time and Effluent Volume

Bed void volume: ~50 ml

Time, hr	U Recovered, %	Cumulative Effluent Vol, ml
1	53	~200
2	73	500
3	81	800
4	85	1100
5	88	1400
6	91	1700
4 hr water wash	94	3700

Table 6. Undissolved Uranium Concentration in Leached Fuel vs Acid Contact Time and Particle Diameter

Time, hr	U Conc, %	Equiv. Dia, μ*
1.8	8.64	4010
2.1	8.15	4590
2.5	3.13	2660
2.8	8.11	4570
3.2	4.83	3870
3.5	2.37	2630
3.9	4.76	3930
4.2	5.68	4200
4.6	4.59	3510
4.9	5.17	4320
5.3	1.11	2270
5.6	3.54	4350
5.9	2.1	2920
6.1	4.86	5040

* $D = 955 M^{1/3}$ (μ), where M = weight in mg (calculated assuming $\rho = 2.2$ g/cm³).

2.1.2 Nitric Acid-Hydrogen Peroxide

Five 40-min runs with intermittent addition of 30% H₂O₂ were made to determine the effect of nitric acid concentration on the rate of dissolution of NbC. The average rate decreased with increasing HNO₃ concentration:

<u>Run No.</u>	<u>HNO₃ Conc., M</u>	<u>Avg. Rate, %/hr</u>
NC-5	3	1.97
NC-4	4	0.78
NC-3	8	0.69
NC-6	10	0.93
NC-2	16	Wt. gain (0.35)

The proportionately higher rates in runs 5 and 6 may be the result of using fresh NbC specimens. The buttons used in the other experiments had previously been contacted with HNO₃-HF solutions, and their appearance suggested the presence of a surface film.

2.2 Design

A brief literature survey indicated a fully resistant material, e.g. Teflon or Fluorothene, is probably necessary as the material of construction for the leachers. In the design of the proposed new leacher (Fig. 3), Teflon or Fluorothene is used for all parts exposed to reagent solution at reflux temperature, with polyethylene or PVC substituted where convenient in lower-temperature components. The 2.5-in. i.d. was chosen to permit charging a cluster of seven rods if desired, and as a compromise between degree of scaleup and cost of fuel for experimental work. Fabrication of the solids sampling valve and the filter section was begun, and an order was placed for the 2.5-in.-i.d. Teflon liner.

The literature survey also indicated that the 18-8 stainless steels might be adequate materials of construction for dissolvent concentrations of ~95% HNO₃--1% HF. Short-term liquid phase corrosion tests showed corrosion rates <5 mils/mo for 304 stainless steels with 21-24 M HNO₃. Corrosion rates were lower at higher HNO₃ concentrations, and at higher HF up to 1 M (higher concentrations were not investigated). Aluminum appeared to be an excellent material of construction with 24 M HNO₃--1 M HF. Type 347 stainless steel did not perform well in 23 M HNO₃--0.5 M HF. No common or special alloys are expected to perform well with reflux in the range 4-16 M HNO₃, 0.5 M HF.

Conceptional designs of mechanical head ends for the Grind-Leach flowsheet were started by the Engineering Department. The design concepts are to cover plans for handling the fuel from the reactor test site through the necessary steps in mechanical head end treatment. The

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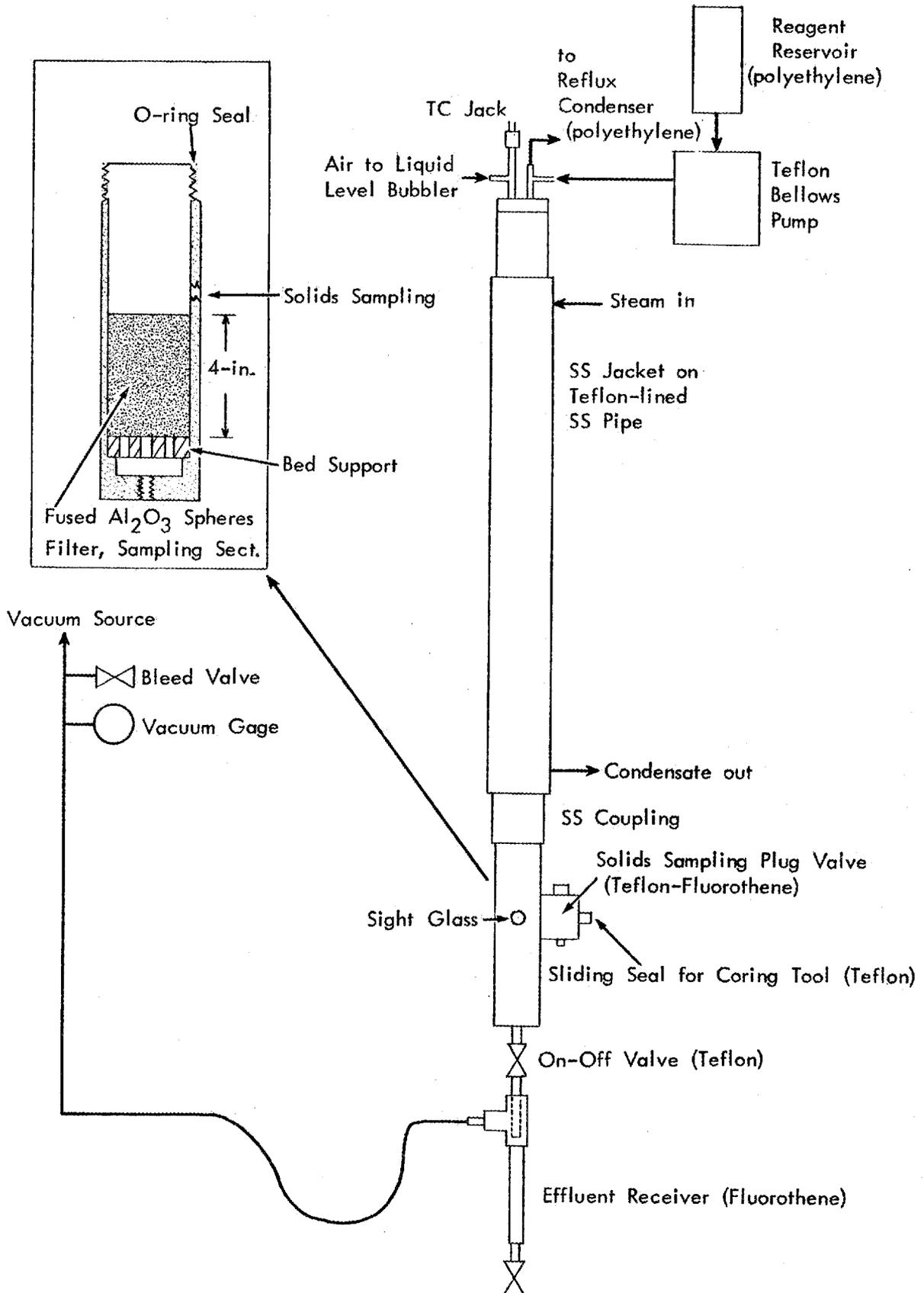


Fig. 3. Jacketed 2.5-in.-i.d. by 45-in.-high downflow leacher.

study will include canning or packaging methods for storage and/or shipment, carriers, unloading and storage facility requirements at the processing site, crushing and/or grinding equipment, and other necessary auxiliaries. Several concepts of crushing and grinding equipment have been proposed, and inexpensive test devices are being built to demonstrate the design concepts.

3.0 COMBUSTION-DISSOLUTION PROCESS

For dissolution of the ash produced by burning KIWI-B fuel specimens, the most promising reagent is $\text{HNO}_3\text{-HF}$. In 5-hr experiments, uranium recoveries were >99.5% in boiling 1-10 M HNO_3 containing 1-5 M HF when the F/Nb mole ratio was >20 (Table 7). Inspection of these and earlier data reported shows that uranium recovery is proportional to the amount of Nb_2O_5 dissolved. Boiling $\text{HNO}_3\text{-H}_2\text{SO}_4$ and $\text{HNO}_3\text{-HCl}$ recovered only about 95% of the uranium from the ash.

A piece of KIWI-B fuel element held 6.5 hr at 800°C in a stream of CO_2 lost only 3.1% of its weight. The NbC liner apparently was converted to Nb_2O_5 , but the sample otherwise appeared intact.

Another piece of KIWI-B fuel was contacted with steam for 7.5 hr at 175°C . There was some gross fracturing of the specimen but no chemical attack of the liner.

4.0 CHLORIDE VOLATILITY STUDIES

Uranium removal was improved somewhat in laboratory studies by first treating ground Rover fuel (12-25 mesh) with pure chlorine for 2 hr at 800°C . Although with massive pieces of NbC the rate of attack by chlorine was very slow at 700°C , the thin liner material apparently is severely attacked during 2 hr at 800°C . The sample was further treated for 1 hr with 50% $\text{COCl}_2\text{-O}_2$ to remove residual niobium, for 2 hr with 50% $\text{COCl}_2\text{-Cl}_2$ to chlorinate uranium oxide, and finally for 2 hr with chlorine to volatilize all uranium. The treatment with 50% $\text{COCl}_2\text{-O}_2$ resulted in much burning of graphite, apparently because the NbC liner material had all been destroyed in the previous treatment with chlorine. Thus the treatment with 50% $\text{COCl}_2\text{-O}_2$ is probably unnecessary. Solution analyses are

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Table 7. Acid Leaching of Ash Produced by Burning KIWI-B Fuel Specimens in Oxygen

Leaching time: 5 hr

Run No.	Ash Comp., %		Reagent	F/Nb Mole Ratio ^a	Amount in Acid-Insoluble Residue, %	
	U	Nb			U	Nb
7	39.1	2.7	1 M HNO ₃ -5 M HF	250	0.13	29.0
4	54.9	19.7	3 M HNO ₃ -1 M HF	43	0.2	21.0
2	58.8	23.5	3 M HNO ₃ -1 M HF	47.2	0.25	14.8
3	52.0	34.6	10 M HNO ₃ -1 M HF	24.5	0.25	10.0
10	43.0	19.5	3 M HNO ₃ -3 M HF	35	0.032	3.0
8	31.6	7.7	1 M HNO ₃ -5 M HF	131	0.008	2.8
9	45.4	25.5	3 M HNO ₃ -3 M HF	40	0.005	1.8
1	52.4	27.4	10 M HNO ₃ -1 M HF	40.5	0.01	1.1
11	42.4	33.0	3 M HNO ₃ -3 M HCl	---	4.9	99.9
12	42.8	32.4	3 M HNO ₃ -3 M HCl	---	4.6	99.8
13	41.9	32.7	10 M HNO ₃ -2 M H ₂ SO ₄	---	4.8	99.7
14	42.2	32.9	5 M HNO ₃ -5 M H ₂ SO ₄	---	5.7	99.5

^aDefined as total moles of fluoride to total niobium in sample.

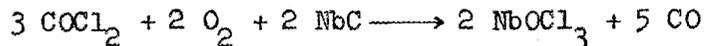
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not yet available, but analysis of the graphite residue indicated <0.1% of the original uranium was present. Surprisingly, when larger particles, 4-12 mesh, of Rover fuel were treated as above, the graphite residue contained nearly 4% of the original uranium.

Chlorination studies indicated the feasibility of complete conversion to chlorides of the ash obtained by burning Rover fuel. A 1-g sample of ash, in a small porcelain boat (2.5 cm² bed surface), volatilized completely in 1.5 hr at 500°C in a stream of chlorine (300 cc/min) saturated at room temperature with carbon tetrachloride vapor. The volatilization rate with 50% COCl₂-50% Cl₂ was 50% higher. Corrosion studies have shown that materials such as Nichrome V resist both oxygen at high temperatures (<0.04 mil/mo corrosion at 725°C) and chlorinating reagents at temperatures as high as 500°C (2 mils/mo corrosion).

Immersion of ground (16 mesh) 2.5-g samples of 3.5% uranium-graphite 1 to 4 hr in refluxing (208°C) perchloropropene removed only 40-50% of the uranium. Approximately half the uranium removed was soluble in the perchloropropene; the remainder was recovered by a water wash of the graphite product.

In engineering-scale studies niobium carbide burned rapidly in phosgene-oxygen mixtures at 700°C when the mixture contained at least 40% oxygen. In 1 hr in pure phosgene a 0.29-g sample lost 0.02 g, and in 1 hr in 75% COCl₂-25% O₂, a 0.36-g sample lost 0.06 g. In 15 min in 60% COCl₂-40% O₂, a 0.31-g sample reacted completely, forming volatile niobium oxychloride and 0.02 g of nonvolatile niobium oxide. It apparently is necessary to use at least the stoichiometric amount of oxygen according to the equation



In the runs with pure phosgene and 75% COCl₂-25% O₂, the usually light metallic color of the NbC changed to shiny black, probably because of depletion of niobium at the surface, leaving a carbon-rich film.

Phosgene-oxygen mixtures appeared to attack NbC more rapidly than chlorine-oxygen mixtures and produce less nonvolatile niobium oxide. The niobium oxide residue is not expected to form with NbC in contact with graphite.

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