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PILOT PLANT VERIFICATION OF OPERATING CONDITIONS FOR THE ROVER SECONDARY BURNER

March 1978

**Allied
Chemical**

IDAHO CHEMICAL PROGRAMS

IDAHO NATIONAL ENGINEERING LABORATORY

DEPARTMENT OF ENERGY

IDAHO OPERATIONS OFFICE UNDER CONTRACT EY-76-C-07-1540

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PILOT PLANT VERIFICATION OF OPERATING CONDITIONS
FOR THR ROVER SECONDARY BURNER

by

A. G. Westra

ALLIED CHEMICAL CORPORATION
IDAHO CHEMICAL PROGRAMS - OPERATIONS OFFICE

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ABSTRACT

Pilot plant verification of operating conditions for the fluidized bed combustion of graphite in the Rover Fuels Processing Facility Secondary Burner is described. Operating temperature, filter blowback frequency, fluidizing gas composition, ash elutriation time, and U_3O_8 to Al_2O_3 ratio in the burner bed were studied. A startup procedure and a graphite burn-out procedure for initial checkout of the Rover Secondary Burner are recommended.

SUMMARY

The Rover Fuels Processing Facility was built at the Idaho Chemical Processing Plant (ICPP) to recover uranium from the graphite matrix fuel used in the Rover nuclear rocket project. Rover fuel rods will be processed by burning the graphite in fluidized bed burners (primary and secondary) and then dissolving the residual ash in HNO_3 and HF . The uranium will be partitioned from the fission products by the solvent extraction system presently used at ICPP.

Experiments were performed in a 10.2 cm diameter fluidized bed pilot plant burner to further define operating parameters for the Rover Secondary Burner. The optimum combustion conditions in the pilot plant burner were found to occur at 1100 - 1110 K with a fluidizing gas mixture of 70 - 80% oxygen and 20 - 30% air (75 - 85% O_2 and 15 - 25% N_2). The optimum blowback frequency for the sintered metal filters, which were used to retain fines in the burner, was found to be one filter every 10 seconds during the combustion period. At these conditions, the temperature was stable and the burner performed well.

The maximum amount of U_3O_8 that could be present in the alumina bed before sintering occurred was found to be 44.5% of the weight of the alumina when the burner was operated at 1100-1110 K. This is substantially higher than will be present in the Rover Secondary Burner where the U_3O_8 is expected to be approximately 27% of the weight of the alumina in the fluidized bed.

Tests showed that 94% of the ash that can be elutriated from the bed will be elutriated in 4 hours using a fluidizing velocity at the distributor plate of 0.4 m/s. The residual U_3O_8 in the bed after elutriation averaged 14.9% of the bed by weight. Roughly 50% of this residual U_3O_8 either adhered to the alumina or consisted of ash particles in the same size range as the alumina particles. The average graphite combustion rate in the pilot plant burner runs was $72 \text{ kg/h}\cdot\text{m}^2$, based on the burner cross-sectional area.

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I. INTRODUCTION

The fuel elements from the Rover nuclear rocket project are composed of uranium dicarbide fuel particles coated with pyrolytic carbon dispersed in a graphite matrix. A protective coating of niobium carbide is present on the surfaces of the cooling channels of most of the fuel elements. The uranium will be recovered from these fuel elements at the Rover Fuels Processing Facility at ICPP.

The fuel elements, packed in cardboard containers, will be burned in a fluidized bed Primary Burner.^(1,2) The ash from the Primary Burner will contain some residual graphite along with U_3O_8 , Nb_3UO_{10} , and Nb_2O_5 formed in the burner. The residual ash from the Primary Burner will be burned batchwise in a Secondary Burner containing a bed of fluidized alumina. The ash from the Secondary Burner will be dissolved sequentially in nitric acid and hydrofluoric acid. The dissolver product will be complexed with aluminum nitrate to reduce corrosion. The complexed dissolver product will pass through a centrifuge and then a solvent extraction system to separate the uranium from the fission products.

Experiments done earlier with a 15.25 cm (6-inch) diameter fluidized bed burner showed that a single burner will not burn all of the graphite in the Rover fuel elements. Therefore, the ash from the Primary Burner will be burned batchwise in a Secondary Burner containing a bed of fluidized alumina. Early feasibility experiments on Rover secondary burning were performed in a 5.1 cm (2-inch) diameter burner.⁽³⁾ These tests showed that the graphite content could be reduced to an acceptable level in a Secondary Burner.

This report describes the larger scale tests done in the 10.2 cm (4-inch) diameter pilot plant burner to further define operating parameters. The burner operating parameters studied in these experiments were:

- (1) Fluidized bed temperature during the combustion period.
- (2) Blowback frequency for the sintered metal filters during the combustion period.

- (3) Oxygen-to-air ratio in the fluidizing gas during the combustion period.
- (4) Ash elutriation time and efficiency.
- (5) The maximum amount of U_3O_8 that can be present in the alumina bed before sintering occurs.
- (6) Combustion efficiency.
- (7) Burner pressure.

A safe and reliable startup procedure and a graphite burnout procedure for the Rover Secondary Burner were also established.

II. EQUIPMENT DESCRIPTION

The equipment used for the pilot plant secondary burner tests consisted of a burner vessel with an expanded filter chamber, a cyclone, a filter vessel, an ash collection pot, and associated instrumentation. Figure 1 is a diagram of the experimental setup.

The 10.2 cm pilot plant secondary burner was a scaled-down version of the Rover Facility Secondary Burner. The pilot plant burner was fabricated from Type 304L stainless steel and had a 10.2-cm diameter by 71-cm high fluidized section. Three sintered metal filters were located in a 25.4-cm diameter by 25.4-cm high filter chamber at the top of the burner. Each filter had an individual blowback line connected through a solenoid valve to a 690 kPa (100 psi) air line. The blowback system is shown in detail in Figure 2. The blowback solenoid valves could be actuated manually or with an automatic timer. A HEPA filter was located on the off-gas line downstream of the sintered metal filters to prevent radioactive material from escaping to the atmosphere. A 1.27-cm (1/2-inch) diameter elutriation line connected the top of the filter chamber to the cyclone. The off-gas line and the elutriation line both had ball valves on them so that the off-gas could be routed through the off-gas line during combustion and through the elutriation line when combustion was complete. Both the burner section and the filter chamber of the pilot plant burner had the same L/D as the Rover Secondary Burner built for the plant. The ratio of the filter chamber diameter to the burner diameter was essentially the same for the two burners.

Figure 1. Pilot Plant Secondary Burner

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Figure 2. Filter Blowback System Details

A 3.81-cm (1 1/2-inch) diameter charge tube with two ball valves spaced 20.3 cm apart extended down through the top of the filter chamber. The two ball valves were operated alternately during ash charging to fill the space between the two valves with ash and then drop the contents into the burner. This air-lock charge system allows charging of ash or bed material from a vessel at atmospheric pressure to a burner at a higher pressure.

Fluidizing air entered the burner vessel through a plenum and distributor plate at the bottom of the burner. The distributor plate had 0.635-mm diameter holes arranged in a 12.7-mm triangular pitch. The distributor plate in the Rover Secondary Burner will have 0.635-mm diameter holes arranged in a 13.2-mm triangular pitch. There were drain lines from both the distributor plate and the plenum. Nitrogen for quenching the burn could be added at the distributor plate or just below the filter chamber.

The burner was heated by twelve 1000-watt cartridge heaters attached vertically around the outside of the burner. The heaters were enclosed in a shroud through which air could be forced to cool the burner. The shroud was covered with a 5-cm thickness of magnesia insulation.

The 10.2-cm diameter section of the pilot plant burner had eight thermocouples spaced 5 cm apart from the distributor plate to the 43-cm level, and two thermocouples in the upper part of the burner at the 61-cm and 71-cm levels. Thermocouples were also located in the plenum, filter chamber, cyclone, filter vessel, and ash collection vessel. The thermocouples were the chromel-alumel type with 6.4-mm O.D. stainless steel sheaths. The thermocouple temperatures were printed out by a multipoint recorder. Pressure drops were measured across the distributor plate, the fluidized bed, the filters, the burner vessel, and the cyclone. The off-gas from the burner was continuously sampled just downstream from the filter vessel and analyzed for CO, CO₂, and O₂.

The cyclone and filter vessel were designed to separate the elutriated ash from the elutriation gas. The filter vessel contained three sintered metal filters with blowback solenoids connected to the same blowback switches used for the filters in the burner. After passing through the sintered metal filters, the off-gas from the filter vessel passed through a HEPA filter before it was released to the atmosphere. The elutriated ash dropped from the cyclone and filter vessel to a fines collection pot from which the ash could be drained at the end of the run.

III. EXPERIMENTAL PROCEDURE

The pilot plant secondary burner was operated batchwise under conditions closely duplicating those expected in the Rover Facility. First, fluidizing air was added to the burner to give a fluidizing velocity at the distributor plate of 0.03 m/s (0.1 ft/s). Then, a batch of ash was mixed with 60 mesh alumina, and the mixture was charged to the burner vessel. Since the burner was under a positive pressure from the fluidizing air, the two valves in the charge line were opened and closed alternately to fill the charge tube and then drop the contents into the burner vessel. In the Rover plant, the alumina bed will remain in the burner and a batch of ash will be charged to it approximately every 8 hours.

When all of the batch had been added to the burner, the fluidizing velocity was increased to 0.26 m/s (0.85 ft/s) and the current to the electrical resistance heaters was set at 5 A. As the temperature increased, the fluidizing air flow rate was adjusted to maintain a constant fluidizing velocity (0.26 m/s) at the distributor plate. When the burner bed temperature reached about 920 K, the CO and CO₂ concentrations in the off-gas started increasing and the O₂ concentration in the off-gas started decreasing. This was an indication that combustion had started. When the O₂ concentration in the off-gas fell below 15%, the fluidizing gas was slowly changed to a mixture of pure O₂ and air. The electrical resistance heaters were turned off when the burner temperature reached 1090 K. Shroud cooling air was added when necessary to prevent the burner from becoming too hot. The sintered metal filters were periodically blown back with 690 kPa air to lower the pressure drop across the filters and return fine material to the bed.

The O₂, CO, and CO₂ concentrations were watched closely during the combustion period. When the O₂ concentration in the off-gas increased rapidly and the CO and CO₂ concentration decreased rapidly, there was not enough graphite left in the bed to support combustion. At this point, the fluidizing gas was switched to 100% air, and the electrical heaters were turned on to maintain the bed temperature near 1100 K until the CO₂ concentration in the off-gas decreased to less than 2%. Then, the heaters were turned off and the bed temperature was cooled to 870 K.

The valve on the elutriation line was then opened, and the valve on the burner off-gas line was closed. The fluidizing velocity was increased to 0.4 m/s (1.3 ft/s) to elutriate the ash from the alumina bed. After elutriation was complete, the bed was drained from the burner and screened to determine how much ash was left in the alumina. Generally, the bottom of the burner was removed after a run to inspect the distributor plate and the inside of the burner. Samples of the ash and bed material were analyzed to determine combustion efficiency.

IV. RESULTS AND DISCUSSION

Five initial burner runs were made with a 100% alumina bed to check out the experimental equipment. Several electrical heaters failed during checkout, so the heater leads were replaced with high temperature wire. Once the heaters were working properly, three burns were made with a bed of 65% alumina and 35% graphite to familiarize personnel with the operation of the burner and to further check out the equipment.

Following equipment checkout, twenty-one burner runs were made to verify operability of the burner using a mixture of alumina and simulated primary burner ash as the bed. Not enough actual Rover primary burner ash was available so secondary burner ash was mixed with Rover graphite to simulate primary burner ash. The tests of the 10.2 cm secondary burner were concluded with six training runs for plant operators.

The simulated primary burner ash which was burned in the pilot plant secondary burner was generally a mixture of 75% U_3O_8 and 25% powdered Rover graphite. The graphite was obtained from the Los Alamos Scientific Laboratory where much of the Rover rocket development work was done. The ash from the Rover Primary Burner is expected to contain 10-25% graphite. No niobium was added to the simulated ash although it will be present in varying amounts in the ash from the Rover Primary Burner. The niobium fraction in the Primary Burner ash is expected to average 29% Nb_2O_5 and 10% $Nb_3UO_{10} \cdot (3)$. In the experiments performed in the 5.1 cm diameter secondary burner, Nb_2O_5 was included in the ash with no noticeable effect on performance, except for its buildup on bed particles. Particle size will be controlled in the Rover Secondary Burner by jet grinders.

A summary of the pilot plant burner operating conditions for the runs is presented in Table I. The same batch of U_3O_8 and alumina was used for runs 9 through 16. When the bed was drained after a run, the residual ash

TABLE I

PILOT PLANT SECONDARY BURNER OPERATING CONDITIONS

Run #	Burn Temp, K	Fluidizing Gas Mixture During Combustion		U_3O_8 to Al_2O_3 Ratio	Comments
		O_2	Air		
9	1150	100%	0%	1:2.0	Distributor plate became hotter than rest of bed; a clinker formed
10	1120	100%	0%	1:3.0	Distributor plate became hotter than rest of bed; a clinker formed
11	1120	80%	20%	1:3.4	Clinkers formed on distributor plate and at 43 cm level in the bed
12	--(a)	---	---	1:3.2	Terminated run before reaching combustion temperature
13	1070	80%	20%	1:3.2	Top of bed became hotter than rest, small clinkers formed
14	1070	80%	20%	1:3.2	Top of bed became hotter than rest, small clinkers formed
15	1090	70%	30%	1:3.6	Good temperature control, no clinkers
16	1120	70%	30%	1:3.6	Top of bed became hotter than rest, clinker at 43 cm level
17	1090	100%	0%	1:6.7	Difficult to control temperature, no clinkers
18	1120	60%	40%	1:8.1	Difficult to control temperature, no clinkers
19	1110	70%	30%	1:9.0	Above bed burning when O_2 in off-gas increased temporarily to 50%, no clinkers
20	1110	80%	20%	1:9.0	Above bed burning when O_2 in off-gas increased to 30%, no clinkers
21	1110	90%	10%	1:9.0	Difficult to control temperature, above bed burning, small clinkers formed
22	1110	70%	30%	1:3.6	Above bed burning when O_2 in off-gas went to 31%
23	1110	70%	30%	1:3.2	Poor fluidizing, scattering in bed temperatures
24	1100	70%	30%	1:3.2	Poor fluidizing, scattering in bed temperatures
25	1100	70%	30%	1:3.8	Poor fluidizing, scattering in bed temperatures
26	1100	100%	0%	1:3.2	Very smooth burn, no clinkers
27	1100	80%	20%	1:2.2	Smooth burn but small clinkers formed
28	1110	70%	30%	1:2.0	Temperature at 33 cm level became hotter than rest of bed; a clinker formed
29	1110	70%	30%	1:2.2	Small clinkers formed, otherwise a smooth run
30	1110	60%	40%	1:2.2	Distributor plate became hotter than rest of bed; 5 cm dia. clinker formed
31	1100	70%	30%	1:2.7	Very smooth burn, no problems
32	1100	60%	40%	1:2.1	Distributor plate became hotter than rest of bed
33	1100	60%	40%	1:2.1	Distributor plate became hotter than rest of bed; no clinkers formed
34	1100	60%	40%	1:3.0	Smooth burn
35	1100	70%	30%	1:3.0	Very smooth burn; no problems

(a) Heaters failed so this burn was not completed

which did not elutriate was separated from the alumina bed by screening through a 100 mesh screen. Since some of the ash adhered to the alumina particles or sintered into small agglomerates, the mass of ash recovered generally decreased in consecutive runs. There also was some holdup of ash and alumina in the equipment. If clinkers formed, they were crushed after the burn was completed and mixed with the recovered ash.

A large crumbly clinker formed on top of the distributor plate in run 9; therefore, the fluidizing velocity was increased from 0.26 m/s to 0.30 m/s for run 10 to determine if that would prevent sintering. A clinker again formed on the distributor plate in run 10, so the fluidizing gas mixture was changed from 100% O_2 to a 80% O_2 and 20% air mixture for runs 11, 13, and 14. A clinker again formed on the distributor plate in run 11 so the burner temperature was decreased from 1120 to 1070 K (the temperature actually ranged from 1070 to 1100 K). Small clinkers formed in runs 13 and 14, probably as a result of above-bed burning which occurred at the end of the combustion period. The O_2 concentration in the off-gas at the end of these runs exceeded 30% and contributed to the above-bed burning. Normally, the O_2 concentration in the off-gas remained below 12% during the combustion period.

Run 15 was excellent with no clinkers formed, no above-bed burning, and good temperature control between 1070 and 1110 K. In run 16, the temperature was maintained between 1110 and 1120 K with good temperature control, but a small clinker formed when above-bed burning occurred at the end of run (the O_2 concentration in the off-gas exceeded 30%).

In runs 17 through 21, the fluidizing gas mixture during combustion was varied from a 60% O_2 and 40% air mixture to 100% O_2 to determine the optimum O_2 to air ratio. Fluidizing gas mixtures of 70-80% O_2 and 20-30% air gave good temperature control, while higher and lower O_2 concentrations made temperature control more difficult.

In run 22 the amount of alumina in the bed was reduced to increase the U_3O_8 to Al_2O_3 ratio in the bed to determine the maximum amount of U_3O_8 that could be added before sintering occurred. This decreased the bed height from approximately 65 cm to approximately 40 cm and changed the L/D from 6.5 to 3.9. Run 22 was a very smooth run with good temperature control.

In runs 23, 24, and 25, the amount of alumina was further decreased to increase the U_3O_8 to alumina ratio. This decreased the bed height to approximately 33 cm and the L/D to 3.2. The smaller beds resulted in poor fluidization as indicated by a wide spread in bed temperatures. Apparently, the minimum L/D for good fluidization was between 3.2 and 3.9, with the distributor plate design and fluidizing velocity used.

The bed height was increased to 60 cm for run 26, and the automatic blow-back timer for the sintered metal filters was set on a 1-1/2 min cycle. This was a smooth run with good temperature control using 100% O_2 fluidizing during the burn period. The good temperature control with 100% O_2 fluidizing can be attributed to the 1-1/2 min filter blowback cycle which helped to maintain the bed temperature in a narrow range without adjusting the flowrate of cooling air through the shroud. No sintering occurred in this run with a U_3O_8 to Al_2O_3 ratio of 1:3.2, but when the ratio was increased to 1:2.2 for runs 27 through 30, with bed heights of 60 cm, small clinkers formed. Varying the O_2 to air ratio in the fluidizing gas in runs 27 through 30 did not eliminate the sintering problem with a U_3O_8 to alumina ratio of 1:2.2. Run 30 was the first of 6 operator training runs.

For run 31, the U_3O_8 to Al_2O_3 ratio was increased to 1:2.7, and there was no bed sintering. This was the run with the highest U_3O_8 to Al_2O_3 ratio in which no sintering occurred. Increasing the fluidizing velocity to 0.3 m/s in run 33 did not eliminate the sintering problem at a U_3O_8 to Al_2O_3 ratio of 1:2.1. The last two training runs were made at U_3O_8 to Al_2O_3 ratios of 1:3.0 with no sintering problems.

V. DEFINITION OF OPERATING CONDITIONS FOR THE ROVER FACILITY

1. Bed Temperature During Combustion

Combustion of the Rover graphite began when the bed temperature reached about 920 K, so ash can be charged to the Rover Secondary Burner at 870 K without combustion occurring during charging. The temperature of the fluidized bed during the combustion period was varied from 1070 to 1150 K during the different runs. The optimum bed temperature was found to be in the 1100 - 1110 K range. In this range, the burner operated smoothly, and sudden temperature increases could be controlled by decreasing the O_2 concentration in the fluidizing gas. The O_2 concentration

was decreased by switching to air fluidizing or by adding N_2 . The shroud cooling system worked well to control the overall temperature of the bed, but its response time was too slow to control sudden hot spots in the bed.

When the burner was operated at higher temperatures (1120-1150 K), controlling the bed temperature became more difficult and sintering was more likely to occur. The CO concentration in the off-gas reached 29% for brief periods when the burner was operated in the 1120-1150 K range, but remained below 16% when the burner was operated at 1100-1110 K. Bed temperature control also became a problem at temperatures below 1090 K. If the bed temperature fell below 1080 K during the burn, it was generally necessary to turn on the bed heaters to maintain combustion.

Filter blowback frequency also has a big effect on bed temperature control. This will be discussed in the next section.

A typical temperature profile of the secondary burner during the combustion period is presented in Figure 3 for a bed height of about 50 cm. As the temperature profile shows, the burner off-gas cools rapidly from 1110 K in the bed, to 770 K in the lower part of the filter chamber, to 620 K at the top of the filter chamber. During ash elutriation, the temperature in the cyclone never exceeded 420 K, although the elutriation line between the burner and the cyclone was only 60 cm long.

2. Filter Blowback Frequency During Combustion

Blowing back the fines from the sintered metal filters in the top of the burner had a quenching effect on combustion because the fines which dropped from the filters were 400-500 K cooler than the temperature of the fluidized bed. Decreasing the time interval between filter blowbacks decreased the effect the blowbacks had on the bed temperature. Ideally, the filters should be blown back in sequence as often as practical.

During the various pilot plant runs, the filter blowback system was operated either manually at 3-4 min intervals or with the automatic timer. When the automatic timer was used, the three filters were blown back in sequence 10 s apart after a set interval. During filter blowback, the burner pressure increased from around 11 kPa to about 30 kPa and the differential pressure across the filters increased from approximately 4.5 kPa to approximately 21 kPa.

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Figure 3. Typical Temperature Profile of the Pilot Plant Secondary Burner

With the 3-4 min intervals between manual filter blowbacks, the bed temperature would slowly increase until it was necessary to increase the flowrate of shroud cooling air. When the fines were blown back from the filters, they would cool the bed so much that the shroud cooling air often had to be shut off to prevent cooling the bed below 1080 K.

When the automatic timer was set on a 90 s interval, the bed varied 5-10 K between blowbacks. The bed temperature would drop immediately after the filter blowback sequence and, then, slowly increase until the next blowback.

The bed temperature did not fluctuate with filter blowbacks when the automatic timer was set on a 10 s interval. At this setting a filter was blown back every 10 s continuously throughout the burn. This shorter interval between blowbacks did not allow as many fines to accumulate on the filters, and the fines were returned to the bed more frequently which eliminated the temperature fluctuations.

The Rover Secondary Burner has ten sintered metal filters compared to three in the pilot plant burner. If the ten filters are blown back in sequence at evenly spaced intervals of approximately 10 s, it should be possible to operate the burner so the bed temperature does not vary more than ± 7 degrees K during combustion. The filter blowback frequency during the warmup and elutriation periods will be determined by other factors and was not investigated in these experiments.

3. Optimum Fluidizing Gas Mixture During Combustion

During the burner warmup period, the bed was always fluidized with 100% air. The point at which combustion started could be determined by watching the O_2 , CO, and CO_2 concentrations in the burner off-gas. As combustion started, the CO and CO_2 concentrations slowly increased, and the O_2 concentration slowly decreased. Once combustion was initiated, the fluidizing gas was slowly changed to a mixture of air and O_2 . Oxygen to air ratios varying from 60:40 to 100:0 were tested in different burner runs. This represents actual O_2 concentrations of from 68 to 100%.

The burner could be successfully operated with oxygen to air ratios in the fluidizing gas varying from 60:40 to 100:0; however, the smoothest burner operation was achieved with ratios of 70:30 and 80:20. Higher O_2 concentrations in the fluidizing gas made the burner temperature harder to control, although it resulted in slightly more efficient combustion. Lower O_2 concentrations in the fluidizing gas decreased combustion efficiency without improving burner temperature control.

The O_2 concentration in the fluidizing gas had to be increased cautiously at the beginning of the combustion period to prevent the O_2 concentration in the off-gas from exceeding 30%. If the O_2 concentration in the off-gas exceeds 30%, above-bed burning may occur with possible damage to the filters and burner vessel. The high temperatures which occur in above-bed burning also cause sintering of the bed material. At the end of the combustion period, the O_2 concentration in the off-gas had to be watched very closely since it increased very rapidly. When the O_2 concentration in the off-gas increased from < 10% to approximately 15% and the CO_2 concentration in the off-gas began decreasing rapidly, the fluidizing gas was switched to 100% air to prevent the O_2 concentration in the off-gas from exceeding 30%. If there was still some combustion occurring, a small amount of O_2 was added to the fluidizing air to shorten the time necessary to burn out the residual graphite in the bed. Once the CO_2 concentration in the off-gas decreased to less than 2%, 100% air was used as the fluidizing gas for the elutriation period.

4. Ash Elutriation

Following the combustion period, the ash was elutriated from the burner to the ash collection pot. After elutriation, the alumina bed was drained from the burner and screened through 24 and 100 mesh screens to determine how much ash remained in the bed. Material which would not pass through a 24 mesh screen was ash agglomerates which were crushed for use in later burns. Material which passed through the 24 mesh screen but remained on top of the 100 mesh screen was the alumina bed material (60 mesh) and any ash agglomerates in the same particle size range.

The ash screened from the bed varied from 4-14% of the bed weight with an average of 7.4%. Following runs 17, 21, 25, 26, and 35, samples of the screened alumina bed were analyzed for uranium. The residual ash in the bed could also be calculated using the following formula:

$$\text{Ash Percentage in bed} = \frac{\text{Wt of screened bed} - \text{Wt of alumina charged} \times 100}{\text{Wt of screened bed}}$$

Figure 4 shows the percentage of ash remaining in the bed after screening out the +24 and -100 mesh ash. The amount of U_3O_8 remaining in the alumina bed remained about the same as the bed was used in successive runs. The Rover Facility Secondary Burner has jet grinders to control buildup in the bed, but the pilot plant burner did not. The total U_3O_8 remaining in the bed after elutriation but before screening out the residual ash averaged 14.9%.

Experiments were also performed to determine the time necessary to elutriate a batch of ash from the burner. These consisted of both 4-hour and 8-hour elutriation tests. For the 4-hour elutriation tests, secondary burner ash was mixed with alumina bed and charged to the burner vessel. The ash was elutriated at ambient temperature using a fluidizing velocity of 0.4 m/s (1.3 ft/s), which is the same fluidizing velocity that will be used in the Rover Secondary Burner during elutriation. The velocity in the elutriation line was calculated to be 16.5 m/s. The ash was drained from the collection pot at intervals and weighed.

The weight of the ash elutriated during each period was divided by the total weight of the ash elutriated during all of the periods. The percentage of ash elutriated was plotted versus elutriation time and is presented in Figure 5 for the 4-hour elutriation tests. As expected, most of the ash elutriated in the first 2 hours.

Eight hour elutriation tests were performed immediately following runs 26, 27, 28, and 29. The data from these elutriation tests were plotted using the same method used for the 4-hour tests and are shown in Figure 6. Some scattering in this data can be attributed to the fact that the fluidizing air flowrate was adjusted as the burner cooled from 870 K to ambient temperature during these elutriation tests. The fluidizing velocities probably fluctuated somewhat and caused the ash to elutriate at slightly different rates in the four tests. The fluidizing velocity at the distributor plate was held as close to 0.4 m/s (1.3 ft/s) as possible. At the start of elutriation, the air cooled from 870 K in the bed to 550 K before it reached the elutriation line, resulting in a velocity in the elutriation line of 11 m/s. The velocity in the elutriation line increased to 16.5 m/s as the burner

Fig. 4 Residual Ash in Secondary Burner Bed

Figure 5. Four-Hour Ash Elutriation Test
After Secondary Burning

Figure 6. Eight-Hour Ash Elutriation Test
After Secondary Burning

cooled to ambient temperature. The data presented in Figure 6 show that elutriating longer than 4 hours does not remove much of the residual ash in the bed. After 4 hours, 94% of the ash that can be elutriated will have done so. In the Rover plant, the secondary burner ash should be elutriated until it is time to charge another batch of primary burner ash, or at least 4 hours.

5. Uranium Oxide to Alumina Ratio in the Bed

Bed sintering occurred just above the distributor plate when the U_3O_8 charged to the burner exceeded 37% of the weight of the alumina bed (U_3O_8 to Al_2O_3 ratio of 1:2.7) at a burn temperature of 1100 - 1110 K. The 37% does not include the residual U_3O_8 which remained in the alumina bed after previous burns and could not be screened out with a 100 mesh screen. If this residual ash which averaged 7.5% of the alumina bed is added, the total U_3O_8 in the burner was 44.5% of the weight of the alumina before sintering occurred. Increasing the fluidizing velocity to 0.3 m/s or varying the O_2 to air ratio in the fluidizing gas did not eliminate sintering if the U_3O_8 in the bed exceeded 44.5% of the alumina. When the burn temperature was 1120 - 1150 K, bed sintering occurred when the total U_3O_8 in the burner was 36.5% of the weight of the Al_2O_3 .

The Rover Secondary Burner will contain a bed of 90 kg of Al_2O_3 . If 10.6 kg of U_3O_8 are charged to the burner in each batch as called for in the Rover flowsheet, it will be 12% of the weight of the alumina. The residual U_3O_8 remaining in the bed from previous runs can therefore be 32.5% of the weight of the alumina before the addition of 10.6 kg of U_3O_8 will cause sintering. In the pilot plant burner, the total residual ash in the burner after elutriation averaged 15% with a maximum of 20%. In the Rover Secondary Burner, the total U_3O_8 in the bed is expected to be approximately 27% (12% + 15%) of the weight of the alumina. Therefore, sintering should not occur. The bed in the Rover Secondary Burner should be sampled frequently and analyzed for uranium to verify that uranium is not building up in the bed.

6. Combustion Efficiency

The graphite combustion rates for the pilot plant secondary burner runs averaged $72 \text{ kg/h} \cdot \text{m}^2$. The average combustion rates are presented in Table II along with the corresponding burn temperatures and fluidizing

TABLE II

GRAPHITE COMBUSTION RATES IN THE 10.2 cm PILOT PLANT BURNER

<u>Run #</u>	<u>Burn Rate (kg/h · m²)</u>	<u>Burn Temperature (K)</u>	<u>O₂ to Air Ratio In The Fluidizing Gas</u>
9	93	1150	100:0
10	78	1120	100:0
11	69	1120	80:20
12	(a)		
13	63	1070	80:20
14	37	1070	80:20
15	41	1090	70:30
16	75	1120	70:30
17	70	1090	100:0
18	54	1120	60:40
19	36	1110	70:30
20	66	1110	80:20
21	86	1110	90:10
22	114	1110	70:30
23	(b)		
24	(b)		
25	84	1100	70:30
26	88	1100	100:0
27	88	1100	80:20
28	75	1110	70:30
29	93	1110	70:30
30	88	1110	60:40
31	55	1100	70:30
32	61	1100	60:40
33	53	1100	60:40
34	79	1100	60:40
35	82	1100	70:30

(a) Heaters failed - no burn

(b) Incomplete burning because of poor fluidization

gas compositions. Other factors which affected the combustion rates were general smoothness of the burn and the point at which oxygen was added to the fluidizing gas. The combustion rates were calculated using the following formula:

$$\text{Combustion rate} = \frac{\text{Mass of graphite charged to burner}}{(\text{burn time}) \times (\text{cross-sectional area of burner})}$$

The burn time was taken as the period when the CO₂ concentration in the off-gas was greater than 3%.

Determining how much the graphite content in the ash could be lowered by secondary burning was not an objective of these experiments, however, after six of the runs, the alumina and ash from the burner were heated to 1170 K for 24 hours in a furnace to check for residual combustibles. The percentage of combustibles remaining in the ash and alumina are presented in Table III. The residual graphite in the ash could have been reduced further by holding the burner temperature at 1100 K until the CO₂ concentration dropped to zero.

TABLE III

PERCENT COMBUSTIBLE MATERIAL AFTER SECONDARY BURNING

<u>Run #</u>	<u>In Alumina</u>	<u>In Ash</u>
16	0.12	5.02
17	0.48	No data
25	0.03	No data
26	0.16	15.57
28	No data	14.81
29	0.19	9.97
31	No data	5.93
	Average = 0.2%	Average = 10.3%

During the combustion period the off-gas contained 7-29% CO, 6-12% O₂ with the remainder being N₂ and CO₂. The CO₂ levels reached in the off-gas varied from 45-50% using 60% O₂ for fluidizing to 65-70% using 100% O₂. Nitrogen concentration in the off-gas was not measured. CO levels of 16-29% were reached for short periods in the equipment checkout runs when the burner operating temperature was in the 1120 - 1150 K range. The formation of CO is thermodynamically favored over the formation of CO₂ at higher temperatures in a graphite system. If the gas is then cooled rapidly to below 875 K, some of the CO will not oxidize to CO₂. The result is a high CO concentration in the off-gas. At 1100 - 1110 K, the CO level in the off-gas was generally 7-10% but sometimes rose to 16%, usually at the beginning of combustion.

7. Burner Pressure

The pressure drops across four areas of the burner apparatus, as well as the total pressure in the burner vessel, were recorded. Table IV shows the range of pressures recorded during the burner runs and the surges caused by filter blowbacks. All the pressure drops remained in a narrow range throughout the burner runs except for the pressure drop across the distributor plate which decreased as the volume of fluidizing air to the burner was decreased during burner warmup. The pressure drop across the distributor plate decreased because the fluidizing air was not heated until after it passed through the distributor plate.

Blowing back the filters caused brief pressure surges in the burner above the bed level, but did not affect the pressure drop across the bed or the distributor plate. A pressure regulator on the burner vessel off-gas line was set to maintain the burner pressure at 17.2 kPa gauge (2.5 psig) but did not react fast enough to prevent the burner pressure from exceeding that value during the pressure surges from the filter blowbacks.

8. Recommended Startup Procedure for the Rover Facility Secondary Burner

The following is the recommended startup procedure for the Rover Facility Secondary Burner during the initial checkout based on runs in the 10.2 cm pilot plant burner. The procedure may have to be adjusted depending on the behavior of the full scale plant secondary burner during startup.

TABLE IV

PRESSURE DURING OPERATION OF THE PILOT PLANT SECONDARY BURNER

<u>Pressure Measured</u>	<u>Pressure Range During Burn (kPa)</u>	<u>Range of Pressure Peaks During Filter Blowback (kPa)</u>
Across distributor plate	3-10	3-10
Across bed	1.25-3.0	1.25-3.0
Across cyclone	5-5.5	10.2-11.2
Across filter	3.7-5.2	20-22.5
Burner pressure ^(a)	8.3-13.8	24-35

1 kPa = 0.145 psi = 4 inches H₂O

(a) Gauge pressure on the off-gas line just downstream from the filter vessel.

- (1) Fluidize the alumina bed in the Secondary Burner to 0.26 m/s at the distributor plate using 100% air as the fluidizing gas. Then, charge a batch of primary burner ash to the Secondary Burner.
- (2) Turn on the heaters to heat the fluidized bed to combustion temperature. As the burner bed temperature increases, decrease the fluidizing air flow rate to maintain a fluidizing velocity of 0.26 m/s (0.85 ft/s). The O_2 concentration in the off-gas should start decreasing, and the CO and CO_2 concentration should start increasing when the temperature reaches about 920 K.
- (3) When the O_2 concentration in the off-gas decreases to 15% or less, set the filter blowback system to blowback a filter every 10 seconds and adjust the fluidizing gas mixture to 10% O_2 and 90% air. Observe the O_2 concentration in the off-gas closely at this time because it will increase temporarily. The O_2 concentration in the off-gas must not be allowed to exceed 30% if above bed burning is to be averted. The bed temperature will start rising faster at this time.
- (4) When the O_2 concentration again decreases to 15% or less, adjust the fluidizing gas composition to 30% O_2 and 70% air.
- (5) Allow the O_2 concentration to decrease to 15% again and, then, adjust the fluidizing gas composition to 50% O_2 and 50% air.
- (6) The bed temperature will be increasing rapidly at this time. When it reaches 1080 K, shut off the electrical heaters and prepare to turn on the shroud cooling air.
- (7) When the bed temperature reaches 1090 K, turn on the shroud cooling air and adjust it to maintain the bed temperature as close to 1100 K as possible.
- (8) Adjust the fluidizing gas mixture to 75% O_2 and 25% air.
- (9) Adjust the shroud cooling air as necessary to maintain the bed temperature as close to 1100 K as possible for the duration of the burn.

9. Recommended Graphite Burnout Procedure for the Rover Facility Secondary Burner

During operation of the secondary burner, a point is reached when most of the graphite has been burned out of the ash and combustion will no longer sustain itself at 1100 K. Based on pilot plant experience, the following procedure is recommended for further reducing the graphite content in the ash before elutriation. The behavior of the Rover Secondary Burner during initial startup may dictate some modifications to this procedure.

- (1) When combustion will no longer sustain itself, the off-gas analyzers will show a rapid increase in O_2 concentration and a rapid decrease in CO_2 concentration. Decrease the O_2 concentration in the fluidizing-gas at this time so that the concentration above the bed will not exceed 30%.
- (2) Turn off the shroud cooling air and turn on the burner heaters to maintain the bed temperature near 1100 K.
- (3) Monitor the CO_2 concentration in the off-gas. When it decreases to 1.0%, switch to 100% air fluidizing and cool the bed to 870 K for the elutriation period.

VI. CONCLUSIONS AND RECOMMENDATIONS

The following conclusions were drawn from the Rover secondary burner tests in the 10.2 cm pilot plant burner.

- (1) Optimum operating temperature range was found to be 1100 - 1110 K.
- (2) Blowing back the fines from the sintered metal filters in the top of the burner has a quenching effect on the combustion in the burner. The quenching effect can be reduced by blowing back the filters as often as practical; one filter every 10 s is recommended.
- (3) Optimum fluidizing gas mixture during combustion was found to be 70-80% O_2 and 20-30% air.
- (4) If the O_2 concentration in the burner off-gas above the fluidized bed exceeds 30%, above-bed burning may occur.
- (5) The maximum amount of U_3O_8 that could be charged to the burner without causing sintering is 37% of the weight of the alumina bed. The alumina bed had been used in previous runs and contained 7.5% U_3O_8 before the ash was charged, so the total allowable U_3O_8 was 44.5% of the weight of the alumina.
- (6) At the end of a burn the bed temperature should be maintained at 1100 K until the CO_2 concentration in the off-gas decreases to 1.0%.
- (7) At a fluidizing velocity of 0.4 m/s (1.3 ft/s), 94% of the elutriable ash can be elutriated from the bed in 4 hours. The secondary burner ash should be elutriated until it is time to charge another batch of primary burner ash, or a minimum of 4 hours.
- (8) After elutriation, the bed in the secondary burner still contained an average of 14.9% U_3O_8 . Fifty percent of this residual U_3O_8 could be separated from the alumina bed using a 100 mesh screen. The other fifty percent was either adhering to the alumina particles or consisted of ash particles too large to pass through a 100 mesh screen. The amount of U_3O_8 remaining in the bed after elutriation did not show an increasing trend.
- (9) If the residual U_3O_8 in the alumina bed reaches 25% by weight, the bed should be replaced with fresh alumina.

VII. REFERENCES

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