SUMMARY

The Western Coal Study is on schedule for completion in October, 1976.

The highlights of major accomplishments during the first five weeks of this study include:

1. Successful adaption of analytical methods.
2. Preparation of detailed operational sheets.
3. Selection of mines to be sampled, through U.S.G.S. and various state geologists.
4. Successful completion of coal mine sampling.

Major accomplishments expected during the next reporting period ending September 10, 1976, include:

1. Collection and evaluation of coal sample results.
2. Selection of power plant for study.

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Mine Selection

A list of mines for sampling was carefully selected from discussions with U.S. Geological Survey people in Denver, Colorado. Discussions were held with Mr. Vern Swanson and Paul Averett of U.S.G.S./Denver on June 28, 1976. As per these discussions, a tentative list of mines for sampling was generated. Criteria for selection of this list was based on: (1) Western location, (2) a large producer (> ~0.5 million tons per year), (3) that the mine was a mine mouth (if possible), and (4) that mines producing both lignite and bituminous coal were selected.

This list was further refined (at the suggestion of U.S.G.S./Denver) by calling the state geology people (supplied by V. Swanson) in each state to be visited (see Table I). These calls were to assure that there were no large mines which had recently started to mine, and also to not exclude any mines which may not be located on Federal land which is leased for coal mining.

Table I.
State Personnel Contacts

<table>
<thead>
<tr>
<th>Contact</th>
<th>City and State</th>
<th>Telephone Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mr. Gary B. Glass</td>
<td>Laramie, Wyoming</td>
<td>FTS 328-1110</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Off-Net 307-766-2286</td>
</tr>
<tr>
<td>Mr. Ed Englerth</td>
<td>Bismark, N. Dakota</td>
<td>FTS 783-4011</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Off-Net 701-224-2400</td>
</tr>
<tr>
<td>Mr. Frank Kblanski</td>
<td>Santa Fe, New Mexico</td>
<td>FTS 474-5511</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Off-Net 505-835-5420</td>
</tr>
<tr>
<td>Mr. Bob Matson</td>
<td>Butte, Montana</td>
<td>FTS 585-2611</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Off-Net 406-792-8321</td>
</tr>
<tr>
<td>Mr. Dick Moore</td>
<td>Tucson, Arizona</td>
<td>FTS 762-6011</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Off-Net 602-884-2733</td>
</tr>
<tr>
<td>Mr. Keith Murray</td>
<td>Denver, Colorado</td>
<td>FTS 327-0111</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Off-Net 303-892-2611</td>
</tr>
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</table>
Mine Sampling

Two employes (a supervisor and an analytical technician) departed from Mound Laboratory by automobile on Saturday, July 31, 1976. The employes arrived in Denver on Sunday, August 1, 1976. Sample collection started with the Seneca mine at Hayden, Colorado, on Monday, August 1, 1976. A total of twenty (20) samples from 18 mines were sampled in six states as shown in Table II. These locations are also shown in Figure 1. A total of 500 grams were collected at each mine. The samples were generally collected by ASTM methods, using an automatic sampler which grabs a sample at a fixed interval of time. Exceptions to this include: (1) the Dave Johnston mine at Glen Rock, Wyoming, in which the sample was pulled by hand, and (2) the Hanna #5 and Vanguard #2 (both at Hanna, Wyoming), and the Jim Bridger mine at Rock Springs, Wyoming, in which core borings were taken to represent a seam. It should be noted that core borings are considered more representative than grab samples from a conveyor even if collected by ASTM methods. The samples collected at each mine usually represent several thousand tons - generally the total mined in a 24-hour period. As the early samples were collected, they were shipped by air express so that the laboratory analysis could begin quickly. A total of 6,200 miles were driven in 18 days used for the collection of the samples.

Adaption of Analytical Procedures

Procedures already developed and routinely used for soil, air, water, etc. have been successfully modified for coal. Approximately 50 analyses have been performed with various modifications to improve the chemical recovery.

A known amount of U-232 tracer will be added to each sample analyzed for uranium so that the final result can be corrected for chemical recovery. A known amount of Po-208, 209 tracer will be used in the samples analyzed for polonium.

As per the experimental plan, four sets of quadruplicates at different levels of activity are being analyzed from the lowest to the highest level to establish an error curve for the range of values expected during this study. Known amounts of NBS standard for uranium and polonium are being added to the quadruplicate samples. Additionally, blanks have been run to establish a laboratory base line or background so that a good MDA value can be assigned to these analyses.

Results of quality control samples as well as the actual samples will be reported in progress report number 2.
<table>
<thead>
<tr>
<th>Mine</th>
<th>Date Collected</th>
<th>Production (in millions of tons for 1974)</th>
<th>Location (City and State)</th>
<th>Tons Represented by Sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kayenta</td>
<td>6/76</td>
<td>2.50</td>
<td>Kayenta, Arizona</td>
<td>20,000</td>
</tr>
<tr>
<td>Black Mesa #1</td>
<td>8/4/76</td>
<td>3.93</td>
<td>Kayenta, Arizona</td>
<td>3,076</td>
</tr>
<tr>
<td>Seneca</td>
<td>7/25/76</td>
<td>0.46</td>
<td>Hayden, Colorado</td>
<td>2,500</td>
</tr>
<tr>
<td>Navajo</td>
<td>7/31/76</td>
<td>6.96</td>
<td>Fruitland, N. Mexico</td>
<td>21,560</td>
</tr>
<tr>
<td>Navajo</td>
<td>8/2/76</td>
<td>6.96</td>
<td>Fruitland, N. Mexico</td>
<td>11,479</td>
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<tr>
<td>Sorenson</td>
<td>8/4/76</td>
<td>2.44</td>
<td>Kemmerer, Wyoming</td>
<td>3,700</td>
</tr>
<tr>
<td>Elkol</td>
<td>8/4/76</td>
<td>0.92</td>
<td>Frontier, Wyoming</td>
<td>3,700</td>
</tr>
<tr>
<td>Hannah #5</td>
<td>7/76</td>
<td>0.63</td>
<td>Hanna, Wyoming</td>
<td></td>
</tr>
<tr>
<td>Vanguard #2</td>
<td>7/76</td>
<td>1.01</td>
<td>Hanna, Wyoming</td>
<td></td>
</tr>
<tr>
<td>Jim Bridger</td>
<td>7/31/76</td>
<td>0.74</td>
<td>Rock Springs, Wyoming</td>
<td>Seam Boring (upper)</td>
</tr>
<tr>
<td>Jim Bridger</td>
<td>7/16/76</td>
<td>0.74</td>
<td>Rock Springs, Wyoming</td>
<td>Seam Boring (lower)</td>
</tr>
<tr>
<td>Dave Johnston</td>
<td>8/6/76</td>
<td>2.09</td>
<td>Glen Rock, Wyoming</td>
<td>4,500</td>
</tr>
<tr>
<td>Belle Ayr</td>
<td>8/7/76</td>
<td>3.31</td>
<td>Gillette, Wyoming</td>
<td>10,000</td>
</tr>
<tr>
<td>Big Horn</td>
<td>8/9/76</td>
<td>0.99</td>
<td>Sheridan, Wyoming</td>
<td>4,745</td>
</tr>
<tr>
<td>Decker #1</td>
<td>8/8/76</td>
<td>6.79</td>
<td>Decker, Montana</td>
<td>10,443</td>
</tr>
<tr>
<td>Absoloka</td>
<td>8/6/76 McKay Seam</td>
<td>4.00</td>
<td>Harden, Montana</td>
<td>3,785</td>
</tr>
<tr>
<td>Absoloka</td>
<td>8/9/76 Robinson Seam</td>
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<td></td>
<td>5,750</td>
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<tr>
<td>Savage²</td>
<td>8/10/76</td>
<td>0.30</td>
<td>Savage, Montana</td>
<td>480</td>
</tr>
<tr>
<td>Gascoyne²</td>
<td>8/11/76</td>
<td>0.20³</td>
<td>Gascoyne, N. Dakota</td>
<td>4,500</td>
</tr>
<tr>
<td>Beulah²</td>
<td>8/11/76</td>
<td>1.70</td>
<td>Beulah, N. Dakota</td>
<td>1,000</td>
</tr>
</tbody>
</table>

¹Date represents the date the sample was pulled, not the date the mine was visited.
²Lignite Mines
³Production now up to three million tons per year.
⁴Production now up to 10-12 million tons per year.
Operational Sheets

Complete operational sheets have been prepared which include steps taken from collection of the sample at the mine through counting of that sample for radioactivity. The purpose of these operational sheets is to bring consistency to all the data and traceability of a sample result back through the operational sheets to the mine and day it was sampled. See Appendix A for examples of operational sheets 1, 2 and 3.

In summary, the program is on schedule according to the attached milestone chart. Results of the analysis of mine samples and selection of the power plant to be studied will be reported in progress report number 2 for the period ending September 10, 1976.
PRELIMINARY WESTERN COAL STUDY

MILESTONES

Adapt Analytical Procedures
Select Coal Mines
Contact Officials
Select Best Method of Sampling
Obtain Coal Samples
Analyze QC Samples
Analyze Coal Samples
Select Three Possible Power Plants
Select Power Plant
Collect Power Plant Samples
Analyze Power Plant Samples
Prepare Draft Report
Prepare Final Report

7/12/76
8/13/76

0 1 2 3 4 5 6 7 8 9 10 11 12 13 (Weeks)

PROGRESS TO DATE
Sample Collection: Coal, Soil, or Air

1. Sample No. _______ of _______

2. Collection (Air Only)
   Date ___________ Flow Rate ___________
   Time ___________ Volume ___________

3. Location
   Site Name ____________________________
   County ______________________________
   State ________________________________

4. Members of Sample Crew
   Name __________________ Initials _______

5. Shipping
   Date ___________ Time ___________

6. Transfer to Laboratory
   Date __________________________

7. Accepted By ________________________

8. Remarks

Issue 1 © 7-30-76
COAL OR SOIL

1. Air dry sample at 35-40°C.
2. Weigh the sample to the nearest gram.
3. Place weighed sample into an aluminum pan.
4. Break up sample in increments to load into grinding mill.
5. Grind sample (~15 minutes) in a rotary mill.
6. Pass entire sample through a 60-mesh sieve, screening 95-100% of ground sample.
7. Record the following weights:
   - Sample + container ________g
   - Container ____________g
   - Sample ____________g
8. Riffle with a Soil Riffle (Soil Test, Inc.) to obtain an aliquot of ~10 g.
9. Weigh the aliquot to the nearest 0.1 g.
   - Aliquot + container ________g
   - Container ____________g
   - Aliquot ____________g
10. Return remainder of sample to sample library.

AIR

1. Divide the filter used in gathering the sample into two equal portions.
2. Place half of the filter into a casserole.
3. Place remaining half of the filter into an envelope and return to sample library.
1. FIRING

1.1. Transfer the aliquot from Operation 2 to a #4 casserole.

1.2. Add U-232 tracer solution to the sample in the casserole. Record the activity (in dis/min/ml) of the tracer on the Composite Data Sheet.

1.3. Heat sample in the casserole at 500°C for four hours.

1.4. Remove the casserole from the furnace and cool to room temperature.

2. WET CHEMISTRY

2.1. Transfer the fired sample to a 250-ml beaker.

2.2. Rinse the casserole with 10 ml of conc HNO₃ and transfer the rinse to the same 250-ml beaker.

CAUTION

Perform the following two steps slowly to avoid excessive foaming. Allowing the reaction to proceed too quickly will cause loss of material from the beaker.

2.3. Using a 50-ml graduated cylinder, continuously add 30 ml of conc HNO₃ (\~15.4N) to the fired sample. Allow the solution to cool to room temperature.

2.4. Using a 50-ml graduated cylinder, add 10 ml of conc HCl (\~11.7N). Allow reaction to subside and the solution to cool to room temperature.

2.5. Transfer the beaker to a magnetic hot plate and stir (with a 1-1/2 inch long, teflon-coated stirring bar) for two hours at a temperature just under the boiling point of the solution. During this two-hour leaching period, add conc HNO₃ as needed to retain a constant volume.
2.6. Cool the solution to room temperature and quantitatively transfer it, with distilled H₂O rinses, to a 250-ml centrifuge bottle. Centrifuge the contents for 15 minutes at 3500 rpm.

2.7. Gently pour off the supernatant into a clean 250-ml beaker and evaporate on a hot plate set to maintain the temperature of the solution at just under boiling.

   NOTE: To avoid possible loss of material, cover the beaker with a 100-mm diameter watch glass and curtail any "bumping" at near boiling temperatures.

2.8. Quantitatively remove the residue from the centrifuge bottle and place it in a second 250-ml beaker. Repeat the leaching operation on the residue (steps 2.5 through 2.6) one more time.

2.9. Add the supernatant from the second leach to that of the first leach and slowly evaporate to dryness. (Cover with a 100-mm watch glass.)

2.10. Quantitatively transfer the solid residue from the centrifuge bottle to a glass bottle; seal, label and retain for possible later analysis.

2.11. Add 10 ml of conc HCl (≈11.7N) to the dried residue of step 2.9; cover with a 100-mm watch glass and slowly evaporate to dryness.

2.12. Repeat step 2.11 one time.

2.13. Add 20 ml of 8M HCl to the residue in the 250-ml beaker using a 100-ml graduated cylinder. Slowly dissolve residue, using hot plate set at low heat to aid dissolution.

2.14. Filter the solution through a filter funnel containing Whatman #1 filter paper. Rinse the filter paper twice with 10 ml of 8M HCl. Collect filtrate in a clean 250-ml beaker.

2.15. Add five drops of 30% H₂O₂ to the solution.

2.16. Deliver the solution to a 250-ml separatory funnel using two 5-ml rinses of 8N HCl to complete the transfer.
2.17. Add 60 ml of isopropyl ether to the solution and shake for two minutes. Allow the phases to separate and then transfer the aqueous phase (lower phase) to a second separatory funnel.

2.18. Add 60 ml of isopropyl ether to the second separatory funnel and shake for two minutes. When the phases have separated, transfer the aqueous phase to a clean 100-ml beaker.

2.19. Add 5 ml of 8N HCl to the first separatory funnel and shake for 30 seconds. When the phases have separated, add the aqueous phase to the residual ether in the second separatory funnel.

2.20. Shake the second separatory funnel for about 30 seconds and transfer the aqueous phase to the same 100-ml beaker.

2.21. Repeat steps 2.19 and 2.20 after which the organic phase may be discarded.

2.22. Place the beaker containing the aqueous phase on a hot plate and evaporate any residue ether at a low heat.

3. ANION EXCHANGE SEPARATION

3.1. Assemble the ion exchange columns, as described in Appendix A, for anion exchange separation (chloride resin form).

3.2. Condition the anion exchange resin by rinsing the column with 60 ml of 8N HCl at the rate of 1 ml/min. Discard rinse solution.

3.3. Transfer the solution from step 2.22 to the conditioned anion exchange resin column. Rinse the beaker twice, each time with 5 ml of 8N HCl from a 10-ml graduated cylinder; add the rinse to the column.

3.4. Rinse the column with 40 ml of 8N HCl.

3.5. Discard all rinse solutions which were passed through the column.

3.6. Place a 100-ml beaker under the column and elute the uranium from the column resin into the beaker with 60 ml of 1N HCl.
3.7. Rinse the column with 15 ml of 1N HCl (use 50-ml graduated cylinder). Collect the rinse in the same 100-ml beaker.

3.8. On a hot plate, slowly evaporate to dryness the 100-ml beaker containing the column elution and rinse. Use a 90-mm watch glass as cover. Twice during the evaporation, add 5 ml of conc HNO₃ (~15.4N) using a 10-ml graduated cylinder.

3.9. Add 20 ml of conc HNO₃ (~15.4N) to the dried residue and slowly evaporate to near dryness. Cover with watch glass during evaporation.

4. ELECTRODEPOSITION

4.1. Prepare the electrodeposition cell as described in Appendix B.

4.2. Add 10-12 drops of conc H₂SO₄ (~35.6N) to the beaker containing residue obtained in step 3.9.

4.3. Using beaker tongs, carefully heat the beaker over the flame of a Meeker burner in a fume hood until dense, white fumes of SO₂ are evolved.

4.4. Allow solution to cool to room temperature.

4.5. Using a burette, pipette, or 10-ml graduated cylinder, carefully add 2 ml of distilled water to the solution and gently swirl the contents until the solution is thoroughly mixed.

4.6. Quantitatively transfer the solution to the prepared electrodeposition cell for plating.

4.7. Rinse the beaker with 3 ml of distilled water from a pipette, burette, or 10-ml graduated cylinder, and transfer this rinse to the plating cell.

4.8. Add 2-3 drops of methyl red indicator from a medicine dropper pipette to the solution in the plating cell.

4.9. Clamp the plating cell into place with a clamp attached to a vertical steel rod, set the Variac of the electric stirring motor on low speed, turn on the electric motor, and stir continuously while slowly adding conc NH₄OH (~14.8N) dropwise until the solution changes from a red to a yellow color.
4.10. Immediately add 3N H₂SO₄ dropwise (about three drops) until the color of the solution changes back to red; then add four additional drops of 3N H₂SO₄.

4.11. Assemble the plating cell onto the electrodeposition unit, connect the platinum wire anode in place, and adjust the current on the ammeter to read 0.3 amps.

4.12. Continue the plating operation for a three-hour period. When three hours have elapsed, add 1-2 ml of conc NH₄OH (~14.8N) to the solution in the cell using a pipette or graduated cylinder. Continue plating for one additional minute with the current remaining at 0.3 amps.

4.13. When step 4.12 is completed, turn the current off, disconnect the plating cell from the electrodeposition unit, discard the cell solution, and rinse the cell 2-3 times with distilled water.

4.14. Grasping the disc near its outer edge, carefully remove the disc from the plating cell using a tweezer forceps. Wash the disc thoroughly with distilled water and place on a clean, dry paper towel to air dry.

4.15. Transfer the disc, using tweezer forceps, to a cardboard slide box and transport to A-Building, Room 191 for pulse height analysis.