UNITED STATES DEPARTMENT OF THE INTERIOR

GEOLOGICAL SURVEY

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CHEMICAL ANALYSES OF DEEP-CORE COAL AND SHALE SAMPLES FROM THE ALMOND FORMATION, WASHAKIE BASIN, SWEETWATER COUNTY, WYOMING

by

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Open-File Report 79-1249
1979

Introduction

During gas and oil exploration efforts in the Washakie Basin in Sweetwater County, Wyoming, undertaken by Ladd Petroleum Co. (a subsidiary of Utah International Inc.) core from the Upper Cretaceous Almond Formation of the Mesaverde Group was collected from their I-22 Federal well. (See figure 1 for location.) From this core three coal and two carbonaceous shale samples, orginally at depths ranging from 3,091 to 3,116 m (see table 1 for descriptions), were selected and given to the U.S. Geological Survey for study. Results of chemical analyses on the samples and a comparison of these analyses with analyses of near-surface Almond Formation coal samples in Carbon County, Wyoming, are the topics of this report.

Explanation of Tables

Proximate and ultimate analyses, and heat-of-combustion, air-dried-loss, forms-of-sulfur, and ash-fusion-temperature determinations on the three coal samples (table 2) were provided by the U.S. Bureau of Mines, Pittsburgh, Pa. Analyses for ash content and contents of 31 major and minor oxides and trace elements in laboratory ash (table 3) and contents of seven trace elements in whole coal (table 4) were provided by the U.S. Geological Survey, Denver, Colo. The U.S. Geological Survey also provided analyses for contents of 37 major and minor oxides and trace elements; contents of sulfur; and total, mineral, and organic carbon analyses on two carbonaceous shale samples (table 5). Analytical procedures used by the U.S. Geological Survey are described in Swanson and Huffman (1976). Table 6 contains the data listed in table 3 converted to a whole-coal basis, and for completeness the whole-coal analyses listed in table 4, and the element compositions for shale from table 5. Twenty-seven additional elements not listed in tables 3 through 6 were looked for but not found in amounts greater than their lower limits of detection

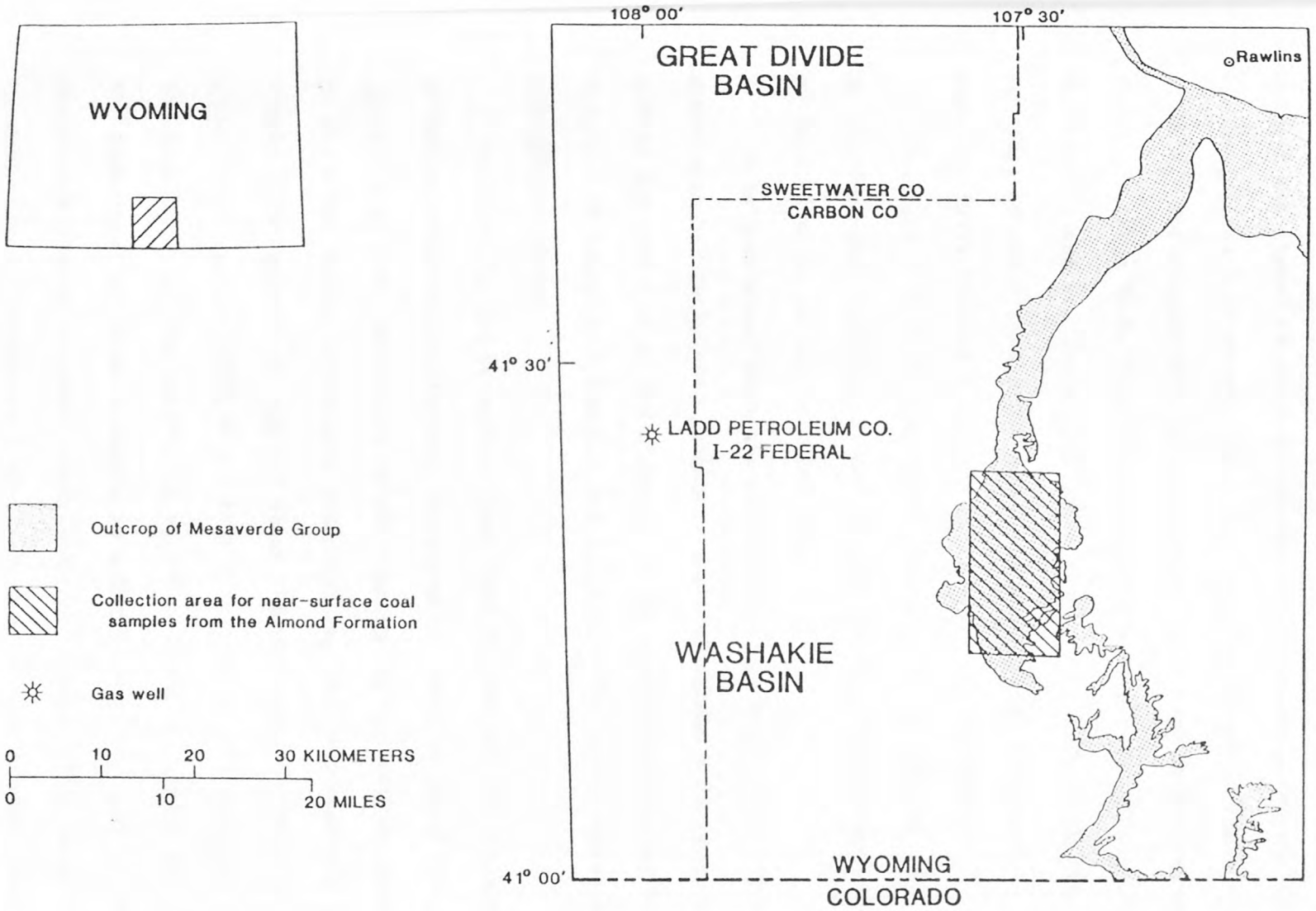

Figure 1.-- Index map of south-central Wyoming showing the location of the Ladd Petroleum Co. I-22 Federal Well, the collection area for the near-surface Almond Formation coal samples, the Little Snake River field, and the outcrop area for the Mesaverde Group. Map modified from Love, Weitz, and Hose (1955).

(table 7). Arithmetic means and standard deviations of the analytical data in table 2 are listed in table 8. Geometric means and geometric deviations of the analytical data on the three coal samples listed in table 6 are listed in table 9. For comparison, data summaries for Almond Formation coal samples from the Little Snake River field, Carbon County, Wyo. (fig. 1), are included in tables 8 and 9. The Little Snake River field samples are air-drilled cuttings of coals that occur above the present-day water table. The cuttings were carefully cleaned to remove extraneous rock fragments.

Statistics could not be calculated for the Co, La, Mo, Nb, and P contents on the deep-core samples, as one or more of the analyses were below the limits of detection (L) or not detected (N).

To be consistent with the precision of the semiquantitative emission spectrographic technique, geometric means of elements determined by this method are reported as the midpoint of the enclosing six-step brackets. (See subtitle of table 3 or Swanson and Huffman, 1976, p. 6, for an explanation of six-step brackets.)

In table 9, the geometric means (GM) is used as the estimate of the most probable concentration (mode); the geometric mean is calculated by taking the logarithm of each analytical value, summing the logarithms, dividing the sum by the total number of values, and obtaining the antilogarithm of the result. The measure of scatter about the mode used in table 9 is the geometric deviation (GD), which is the antilog of the standard deviation of the logarithms of the analytical values. These statistics are used because the quantities of trace elements in natural materials commonly exhibit positively skewed frequency distributions; such distributions are normalized by analyzing and summarizing trace-element data on a logarithmic basis.

If the frequency distributions are lognormal, the geometric mean is the

best estimate of the mode, and the estimated range of the central two-thirds of the observed distribution has a lower limit equal to GM/GD and an upper limit equal to GM·GD. The estimated range of the central 95 percent of the observed distribution has a lower limit equal to $GM/(GD)^2$ and an upper limit equal to $GM \cdot (GD)^2$ (Connor and others, 1976).

A common problem in statistical summaries of trace-element data arises when the element content of one or more of the samples is below the limit of analytical detection. This results in a "censored" distribution. Procedures developed by Cohen (1959) were used to compute unbiased estimates of the geometric mean and geometric deviation when the data were censored.

Discussion

The apparent ranks of all Almond Formation coal samples from both the deep core and Little Snake River field were calculated using the formulas in ASTM designation D-388-77 (American Society for Testing and Materials, 1978). The apparent rank for the deep-core samples is medium-volatile bituminous coal; the apparent rank for the relatively near surface samples of the Little Snake River field ranges from subbituminous B to subbituminous A coal.

A statistical comparison (student's t test, 95 percent confidence level) of the U.S. Bureau of Mines data for the Almond Formation samples from the deep core with data for the coal samples from the Little Snake River field, summarized in table 8, shows that the deep-core samples have significantly higher contents of fixed carbon and carbon, a significantly higher heat of combustion, and significantly lower contents of moisture, hydrogen, nitrogen, and oxygen. When compared at the 99 percent confidence level, the hydrogen and nitrogen contents of the two sets are not significantly different. The significantly higher carbon and fixed-carbon contents, increased heat of

combustion, and significantly lower moisture, hydrogen, and oxygen contents of the deep-core samples appears due to the deep burial (3,100 m) and the higher temperatures that would be encountered at that depth.

A statistical comparison of the geometric mean contents of 32 elements in the deep-core coal samples with mean contents in the Little Snake River field coal samples shows that the deep core samples have significantly higher contents of Li and Na and significantly lower contents of Ca, Mg, B, Mn, and Zn. When compared at the 99 percent level, only the Ca, Mg, B, and Mn contents in the two sample sets are different.

The decrease in the contents of Ca, Mg, B, and Mn in coal with an increase in rank has been noted by several previous researchers. Summary analytical data on 799 coal samples from most coal-producing areas in the United States (Swanson and others, 1976, table 4c) shows that Ca, Mg, B, and Mn contents in coal all decrease with the increasing coal rank. Data summarized by Hildebrand and Hatch (1977) shows that for 754 samples of lignite, subbituminous coal, and bituminous coal from the Rocky Mountain and Northern Great Plains provinces the concentrations of calcium and magnesium decrease rapidly with increasing coal rank. The explanation of Hildebrand and Hatch (1977) involves elimination of cation-exchange positions on the organic materials as coal rank increases. In a related study by Ong and Swanson (1966) on copper adsorption from aqueous solution by peat, lignite, and bituminous coal, it was concluded that many cations are fixed (at least in part) on the organic material by ionic bonds (cation-exchange positions), that the amounts of cations fixed are directly dependent on the surface area, and that the surface area of naturally occurring organic material decreases with increasing rank (degree of metamorphism).

Acknowledgments

Fundamental to this paper is the contribution of the team of chemical laboratory personnel in the U.S. Geological Survey under the direction of Claude Huffman, Jr.: James W. Baker, Ardith J. Bartel, Leon A. Bradley, George T. Burrow, Edward J. Fennelly, Johnnie M. Gardner, Patricia G. Guest, John C. Hamilton, Raymond G. Havens, Jay P. Hemming, Jessie O. Johnson, Roy J. Knight, Robert E. McGregor, Violet M. Merritt, Hugh T. Millard, Jr., Jeffry O'Kelley, Gaylord D. Shipley, James A. Thomas, Richard E. VanLoenen, James S. Wahlberg, Ralph J. White, Thomas L. Yager. In connection with the acknowledgment to the above staff of chemical analysts, the invaluable contribution of the chemists in the Coal Analysis Section (Forrest E. Walker, Chemist in Charge), U.S. Bureau of Mines, Pittsburgh, Pa., is also gratefully acknowledged. We also appreciate Betty L. Arnone's drafting of the illustration.

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Table 1.--U.S. Geological Survey sample number, description, sample thickness, and depth interval represented for five coal and carbonaceous shale samples from the Almond Formation, Sweetwater County, Wyo.

[All samples are of Late Cretaceous age and were collected from Ladd Petroleum Corporation's I-22 Federal well located in SW 1/4 SE 1/4 sec. 22, T. 17 N., R. 94 W., 1 ft = 0.3048 m]

USGS sample number	Description	Sample thickness, in centimeters	Depth interval, represented in meters
D177050	Coal----------	15	3,091.3 - 3,091.6
D177053	Shale, carbonaceous	15	3,091.3 - 3,091.6
D177051	Coal, shaly--------	15	3,102.9 - 3,103.2
D177054	Shale, carbonaceous	15	3,102.9 - 3,103.2
D177052	Coal----------	38	3,115.4 - 3,115.8

Table 2.--Proximate and ultimate analyses, and heat-of-combustion, forms-of-sulfur, free-swelling-index, and ash-fusion-temperature determinations for three deep-core coal samples from the Almond Formation, Sweetwater County, Wyo.

[All analyses except heat of combustion, free-swelling index, and ash-fusion temperatures in percent. For each sample number, the analyses are reported three ways: first, as received; second, moisture free; and third, moisture and ash free. Kcal/kg = 0.556 (Btu/lb); $^{o}F = (^{o}C \times 1.8) + 32$]

Sample number	Proximate analysis				Ultimate analysis					Heat of combustion	
	Moisture	Volatile matter	Fixed carbon	Ash	Hydrogen	Carbon	Nitrogen	Oxygen	Sulfur	Kcal/kg	Btu/lb
D177050	1.2	24.8	56.6	17.4	4.5	71.0	1.7	4.6	0.8	7,020	12,630
	---	25.1	57.3	17.6	4.4	71.9	1.7	3.6	.8	7,100	12,780
	---	30.4	69.6	---	5.4	87.3	2.1	4.2	1.0	8,620	15,520
D177051	1.4	17.8	33.4	47.4	3.2	41.7	.9	6.5	.3	3,990	7,190
	---	18.0	34.0	48.0	3.0	42.3	.9	5.5	.3	4,050	7,290
	---	34.7	65.3	---	5.9	81.4	1.7	10.4	.6	7,794	14,030
D177052	1.0	25.5	69.4	4.1	5.1	84.3	1.8	4.0	.7	8,270	14,880
	---	25.7	70.2	4.1	5.0	85.1	1.8	3.3	.7	8,350	15,030
	---	26.8	73.2	---	5.2	88.7	1.9	3.5	.7	8,710	15,680

Sample number	Air-dried loss	Forms of sulfur			Free-swelling index	Ash-fusion temperature, ^{o}C		
		Sulfate	Pyritic	Organic		Initial deformation	Softening	Fluid
D177050	0.34	0.02	0.01	0.76	9	1,470	1,500	1,560
	---	.02	.01	.77				
	---	.02	.01	.93				
D177051	.28	.01	.01	.30	3.5	1,600+	1,600+	1,600+
	---	.01	.01	.30				
	---	.02	.02	.58				
D177052	.21	.02	.03	.64	9	1,232	1,280	1,350
	---	.02	.03	.65				
	---	.02	.03	.67				

Table 3.--Major- and minor-oxide and trace-element composition of the laboratory ash of three deep-core coal samples from the Almond Formation, Sweetwater County, Wyo.

[Coal ashed at 525°C. L means less than the value shown; N, not detected. S after element title indicates determinations by semiquantitative emission spectrography. The spectrographic results are to be identified with geometric brackets whose boundaries are part of the ascending series 0.12, 0.18, 0.26, 0.38, 0.56, 0.83, 1.2, etc., but reported as midpoints of the brackets, 0.1, 0.15, 0.2, 0.3, 0.5, 0.7, 1.0, etc.; precision of the spectrographic data is plus-or-minus one bracket at 68 percent or plus-or-minus two brackets at 95 percent confidence level]

Sample number	Ash (percent)	SiO_2 (percent)	Al_2O_3 (percent)	CaO (percent)	MgO (percent)	Na_2O (percent)	K_2O (percent)	Fe_2O_3 (percent)	TiO_2 (percent)	P_2O_5 (percent)	SO_3 (percent)	Sample number
D177050	18.9	71	19	0.45	0.93	0.54	2.7	2.5	0.82	1.0L	0.47	D177050
D177051	50.1	61	31	.13	.31	.84	5.8	1.1	1.5	1.0L	.24	D177051
D177052	4.1	37	27	7.9	.52	1.31	.24	6.1	1.9	7.9	2.2	D177052

Sample number	B-S (ppm)	Ba-S (ppm)	Be-S (ppm)	Co-S (ppm)	Cr-S (ppm)	Cu (ppm)	Ga-S (ppm)	La-S (ppm)	Li (ppm)	Mn (ppm)	Mo-S (ppm)	Sample number
D177050	100	1,500	7	10L	70	47	30	100L	166	130	N	D177050
D177051	70	700	N	N	30	30	50	N	150	30	N	D177051
D177052	200	15,000	20	30	70	100	50	100	145	100	7	D177052

Sample number	Nb-S (ppm)	Ni-S (ppm)	Pb (ppm)	Sc-S (ppm)	Sr-S (ppm)	V-S (ppm)	Y-S (ppm)	Yb-S (ppm)	Zn (ppm)	Zr-S (ppm)	Sample number
D177050	20L	15	35	15	500	150	30	3	64	150	D177050
D177051	20L	10L	65	15	70	70	20	2	34	150	D177051
D177052	50	70	70	30	7,000	150	70	7	78	150	D177052

Table 4.--Content of seven trace elements in three deep-core samples from the Almond Formation, Sweetwater County, Wyo.

[Analyses in parts per million on air-dried (32°C) coal. L, less than the value shown]

Sample number	As	F	Hg	Sb	Se	Th	U
D177050	1L	265	0.05	0.3	0.9	6.7	1.8
D177051	1	130	.05	1.7	3.1	26	4.3
D177052	2	300	.08	.5	.8	2.0L	.4

Table 5.--Major- and minor-oxide, sulfur, carbon, mineral carbon, organic carbon, and trace-element composition of two deep-core shale samples from the Almond Formation, Sweetwater County, Wyo.

[L means less than the value shown. -S after element title indicates determinations by semiquantitative emission spectrography. The spectrographic results are to be identified with geometric brackets whose boundaries are part of the ascending series 0.12, 0.18, 0.26, 0.38, 0.56, 0.83, 1.2, etc., but reported as midpoints of the brackets, 0.1, 0.15, 0.2, 0.3, 0.5, 0.7, 1.0, etc.; precision of the spectrographic data is plus-or-minus one bracket at 68 percent or plus-or-minus two brackets at 95 percent confidence level]

Sample number	SiO_2 (percent)	Al_2O_3 (percent)	CaO (percent)	MgO (percent)	Na_2O (percent)	K_2O (percent)	Fe_2O_3 (percent)	TiO_2 (percent)	P_2O_5 (percent)	S (percent)	Sample number
D177053	59	13	0.14	0.63	0.56	2.6	1.5	0.59	1.0L	0.32	D177053
D177054	50	14	.60	1.18	.81	3.2	3.1	.63	1.0L	.98	D177054

Sample number	C (percent)	Mineral C (percent)	Organic C (percent)	As (ppm)	B-S (ppm)	Ba-S (ppm)	Be-S (ppm)	Co-S (ppm)	Cr-S (ppm)	Cu (ppm)	Sample number
D177053	13.7	0.06	14	5	100	700	3	7	100	28	D177053
D177054	4.9	.17	4.8	40	150	700	3	15	70	34	D177054

Sample number	F (ppm)	Ga-S (ppm)	Hg (ppm)	La-S (ppm)	Li (ppm)	Mn (ppm)	Mo-S (ppm)	Nb-S (ppm)	Ni-S (ppm)	Pb (ppm)	Sample number
D177053	100	30	0.09	70	72	25	3L	20	15	25L	D177053
D177054	1,020	50	.26	50	52	240	3	20	30	25L	D177054

Sample number	Sb (ppm)	Sc-S (ppm)	Se (ppm)	Sr-S (ppm)	Th (ppm)	U (ppm)	V-S (ppm)	Y-S (ppm)	Yb-S (ppm)	Zn (ppm)	Sample number
D177053	0.7	10	1.3	150	21	7.1	150	30	3	77	D177053
D177054	1.6	15	2.4	150	22	4.8	150	30	3	180	D177054

Sample number	Zr (ppm)
D177053	150
D177054	150

Table 6.--Major-, minor-, and trace-element composition of three coal and two shale deep-core samples from the Almond Formation, Sweetwater County, Wyo.

[All coal analyses except As, F, Hg, Sb, Se, Th, and U were calculated from analyses of coal ash. S after an element title means analysis by emission spectrography; L, less than the value shown; N, not detected. Leaders (---) indicate no data.

Sample number	Si (percent)	Al (percent)	Ca (percent)	Mg (percent)	Na (percent)	K (percent)	Fe (percent)	Ti (percent)	P (percent)	S (percent)	Sample number
D177050	6.3	1.9	0.06	0.11	0.076	0.43	0.33	0.093	0.10L	---	D177050
D177053	28	6.9	.10	.38	.39	2.2	.84	.35	.44L	.32	D177053
D177051	14	8.2	.05	.094	.31	.24	.39	.45	.22L	---	D177051
D177054	26	9.0	.43	.71	.60	2.7	2.2	.37	.44L	.98	D177054
D177052	.71	.59	.23	.013	.040	.008	.17	.047	.14	---	D177052

Sample number	C (percent)	Mineral C (percent)	Organic C (percent)	As (ppm)	B-S (ppm)	Ba-S (ppm)	Be-S (ppm)	Co-S (ppm)	Cr-S (ppm)	Cu (ppm)	Sample number
D177050	---	---	---	1L	20	300	1.5	2L	15	8.9	D177050
D177053	13.7	0.06	13.6	5	100	700	3	7	100	28	D177053
D177051	---	---	---	1	30	300	N	N	15	15	D177051
D177054	4.9	.17	4.8	40	150	700	3	15	70	34	D177054
D177052	---	---	---	2	7	700	.7	1.5	3	4.1	D177052

Sample number	F (ppm)	Ga-S (ppm)	Hg (ppm)	La-S (ppm)	Li (ppm)	Mn (ppm)	Mo-S (ppm)	Nb-S (ppm)	Ni-S (ppm)	Pb (ppm)	Sample number
D177050	265	7	0.05	20L	31	25	N	3L	3	6.6	D177050
D177053	100	30	.09	70	72	25	3L	20	15	25L	D177053
D177051	130	20	.05	N	75	15	N	10L	5L	33	D177051
D177054	1,020	50	.26	50	50	240	3	20	30	25L	D177054
D177052	300	2	.08	5	5.9	4.1	.3	2	3	2.9	D177052

Sample number	Sb (ppm)	Sc-S (ppm)	Se (ppm)	Sr-S (ppm)	Th (ppm)	U (ppm)	V-S (ppm)	Y-S (ppm)	Yb-S (ppm)	Zn (ppm)	Sample number
D177050	0.3	3	0.9	100	6.7	1.8	30	7	0.7	12	D177050
D177053	.7	10	1.3	150	21	7.1	150	30	3	77	D177053
D177051	1.7	7	3.1	30	26	4.3	30	10	1	17	D177051
D177054	1.6	15	2.4	150	22	4.8	150	30	3	180	D177054
D177052	.5	1.5	.8	300	2.0L	.4	7	3	.3	3.2	D177052

Sample number	Zr-S (ppm)
D177050	30
D177053	150
D177051	70
D177054	150
D177052	7

Table 7.--Elements looked for but not detected in deep-core coal and shale samples from the Almond Formation Sweetwater County, Wyo.

[Approximate lower detection limits for all elements except Cd in coal or shale ash, as determined by the six-step spectrographic method of the U.S. Geological Survey, are included. Cd lower detection limit is by atomic absorption method]

Element Name	Symbol	Lower limit of detection (ppm) in ash
Silver	Ag	1
Gold	Au	50
Bismuth	Bi	20
Cadmium	Cd	1
Cerium	Ce	500
Dysprosium	Dy	100
Erbium	Er	100
Europium	Eu	200
Gadolinium	Gd	100
Germanium	Ge	20
Hafnium	Hf	200
Holmium	Ho	50
Indium	In	20
Lutetium	Lu	70
Neodymium	Nd	150
Palladium	Pd	5
Praseodymium	Pr	200
Platinum	Pt	100
Rhenium	Re	100
Samarium	Sm	200
Tin	Sn	20
Tantalum	Ta	1,000
Terbium	Tb	700
Tellurium	Te	5,000
Thallium	Tl	100
Thulium	Tm	50
Tungsten	W	200

Table 8.--Arithmetic means and standard deviations of the proximate and ultimate analyses, and heat of combustion for three deep-core Almond Formation coal samples, Washakie Basin, Sweetwater County, Wyo., and for 21 Almond Formation coal samples from the Little Snake River coal field, Carbon County, Wyo.

[All values are in percent except Kcal/kg and Btu/lb]

	Deep-core samples		Little Snake River field samples	
	Arithmetic mean	Standard deviation	Arithmetic mean	Standard deviation
Proximate and ultimate analyses (as received)				
Moisture	1.2	.2	15.4	2.8
Volatile matter	22.7	3.5	28.6	3.7
Fixed carbon	53.1	14.9	37.6	6.4
Ash	23.0	18.1	18.7	8.5
Hydrogen	5.5	.3	5.1	.4
Carbon	85.8	3.2	49.4	7.4
Nitrogen	1.9	.16	1.1	.2
Oxygen	6.0	.31	25.1	2.2
Sulfur	.8	.2	.6	.2
Heat of combustion (as received)				
Kcal/kg	6,425	1,795	4,730	780
Btu/lb	11,560	3,230	8,510	1,400
Heat of combustion (moist, mineral-matter free)				
Kcal/kg	8,570	150	5,940	340
Btu/lb	15,410	270	10,680	610

Table 9.--Geometric means and geometric deviations of the contents of 37 elements in three deep-core Almond Formation coal samples Washakie Basin, Sweetwater County, Wyo., and in 30 Almond Formation coal samples, Little Snake River field, Carbon County, Wyo.

[All analyses except geometric deviation are in percent or parts per million and are reported on a whole-coal basis. As, F, Hg, Sb, Se, Th, and U values used to calculate the statistics were determined directly on whole coal. All other values used were calculated from determinations made on coal ash. Leaders (——) indicate statistics could not be calculated owing to an insufficient number of analyses above the lower detection limit]

Element	Deep-core samples		Little Snake River field samples	
	Geometric mean	Geometric deviation	Geometric mean	Geometric deviation
	Percent			
Si	4.0	3.5	5.7	2.0
Al	2.1	2.9	1.5	1.8
Ca	.088	2.0	.72	2.2
Mg	.051	2.6	.15	1.5
Na	.098	2.4	.025	2.3
K	.094	5.8	.18	2.1
Fe	.28	1.4	.41	1.9
Ti	.13	2.6	.084	1.7
	Parts per million			
As	1.0	1.5	1.0	1.6
B	15	1.9	100	1.7
Ba	500	1.5	200	1.9
Be	1	1.4	1	2.1
Co	——	——	2	2.2
Cr	10	2.1	10	1.9
Cu	8.2	1.7	2.3	1.6
F	220	1.9	118	1.7
Ga	7	2.6	3	2.2
Hg	.06	1.2	.04	1.7
La	——	——	10	1.7
Li	24	2.9	8.7	2.0
Mn	12	2.1	77	2.5
Mo	——	——	1.5	1.7
Nb	——	——	5	1.9
Ni	3	1.0	5	2.5
P	——	——	270	2.7
Pb	8.6	2.7	4.8	2.2
Sb	.6	2.1	.4	1.9
Sc	3	1.9	2	2.0
Se	1.3	1.8	.8	1.2
Sr	100	2.6	70	2.3
Th	7.1	2.9	2.1	2.6
U	1.5	2.7	1.7	2.3
V	20	2.0	15	1.9
Y	7	1.7	10	1.9
Yb	.7	1.7	1	1.9
Zn	8.7	2.1	51	3.6
Zr	20	2.6	70	2.0