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## **Methane Contents of Oil Shale From the Piceance Basin, CO**

By S. J. Schatzel, D. M. Hyman, A. Sainato,  
and J. C. LaScola



**UNITED STATES DEPARTMENT OF THE INTERIOR**

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

bbl	barrel	h	hour
°C	degree Celsius	hp	horsepower
cm <sup>3</sup>	cubic centimeter	in	inch
cm <sup>3</sup> /g	cubic centimeter per gram	K	kelvin
cm <sup>3</sup> /(g·h)	cubic centimeter per gram per hour	lb/ft <sup>3</sup>	pound per cubic foot
cP	centipoise	lbf·(s/ft <sup>2</sup> )	pound (force) seconds per square foot
D	darcy	mD	millidarcy
ft	foot	mi <sup>2</sup>	square mile
ft <sup>3</sup> /d	cubic foot per day	min	minute
ft/mi	foot per mile	mm Hg	millimeter of mercury (pressure)
ft/min	foot per minute	pct	percent
ft <sup>3</sup> /st	cubic foot per short ton	psi	pound per square inch
g	gram	psig	pound per square inch, gauge
gal/min	gallon per minute	wt pct	weight percent
gal/st	gallon per short ton	yr	year

# METHANE CONTENTS OF OIL SHALE FROM THE PICEANCE BASIN, CO

By S. J. Schatzel,<sup>1</sup> D. M. Hyman,<sup>1</sup> A. Sainato,<sup>2</sup> and J. C. LaScola<sup>3</sup>

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## ABSTRACT

The Bureau of Mines determined the gas contents of 135 oil shale samples obtained from approximately 630 ft of core drilling. Drilling was done within a projected mining zone of the Cathedral Bluffs Mine, located in the Piceance Basin of western Colorado. Methane contents were determined by the modified direct method, which can measure the volumes of several gases released from or reacted with mine rock without destructive sample treatment. Over the duration of the test, nitrogen was largely unreactive, CO<sub>2</sub> increased slightly, oxygen decreased markedly, and methane increased more than the total increase of all contained gases.

The gas volumes were normalized per unit sample mass and are given in cubic centimeters per gram. Common time indices for test duration of 3, 40, and 125 days were used to compare methane desorption among samples. The means of the sample populations for these indices were 0.0316, 0.114, and 0.195 cm<sup>3</sup>/g, respectively. These quantities are lower than those reported in a Bureau emissions study at the Horse Draw Mine in the Piceance Basin. Oil shale samples that were gas enriched beyond the mean methane contents frequently contained bitumens and pyrite.

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## INTRODUCTION

Oil shale in the Mahogany Zone of the Green River Formation is a significant potential fuel source. In the Piceance Basin, the Mahogany Zone organic content averages about 16 wt pct (1).<sup>4</sup> The remaining material in oil shale is mostly mineral matter. The composition of the mineral matter varies, but major constituents include dolomite, calcite, quartz, illite, sodium and potassium feldspars, pyrite, and analcite (1). Approximately 80 billion bbl of shale oil was thought to be recoverable using technology available in 1976, and the total reserves of the Green River Formation were estimated as 4 trillion bbl.

The release of methane gas during the mining of fossil fuels is a concern of the mining industry and Federal regulatory bodies. New Federal regulations for metal and nonmetal mines are presently under review, and oil shale mines would be subject to these regulations if approved. The portion of the proposed regulations that directly affects oil shale mining was established in accordance with available documentation of methane emissions from oil shale mine production. Some of this information is the result of work done at the Horse Draw Mine site (fig. 1).

The Horse Draw Mine was developed by the Bureau of Mines in 1978 as an experimental mine. Between the middle of 1979 and the end of 1981, the Horse Draw Mine was operated for the Bureau by the Multi Minerals Corp. Four mining levels were driven at the Horse Draw Mine, between 1,840 and 2,230 ft below the surface (fig. 2). All of these depths are below the dissolution surface, which is the horizon where leaching of soluble minerals terminates and ground water sinks no deeper into the Green River Formation. The minerals halite, dawsonite, and nahcolite, in addition to oil shale, were preserved and were planned mining coproducts.

<sup>4</sup>Underlined numbers in parentheses refer to items in the list of references preceding the appendix at the end of this report.

The Bureau collected gas emissions data during development of mine workings at the Horse Draw Mine, which indicated an average of 42 ft<sup>3</sup>/st (1.3 cm<sup>3</sup>/g) CH<sub>4</sub>. The emissions data were obtained by relating the amount of rock mined in a blast to the volume of methane released. The volume of gas emitted was quantified by measuring concentrations using chromatography. Tube bundles extended from the gas chromatography instruments on the surface to underground sampling locations. Ventilation measurement probes located in mine level exhaust ducts and the shaft exhaust provided volumetric data for the calculation of gas emissions (4).

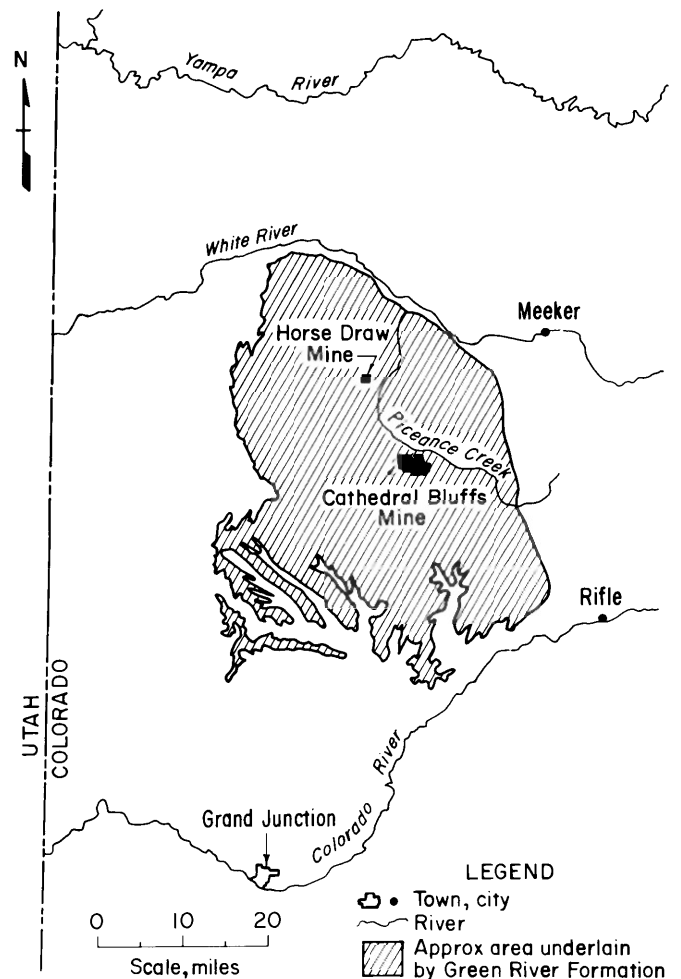


FIGURE 1.—Outline of Green River Formation within margin of Piceance Basin, CO. (Adapted from Russell (2).)

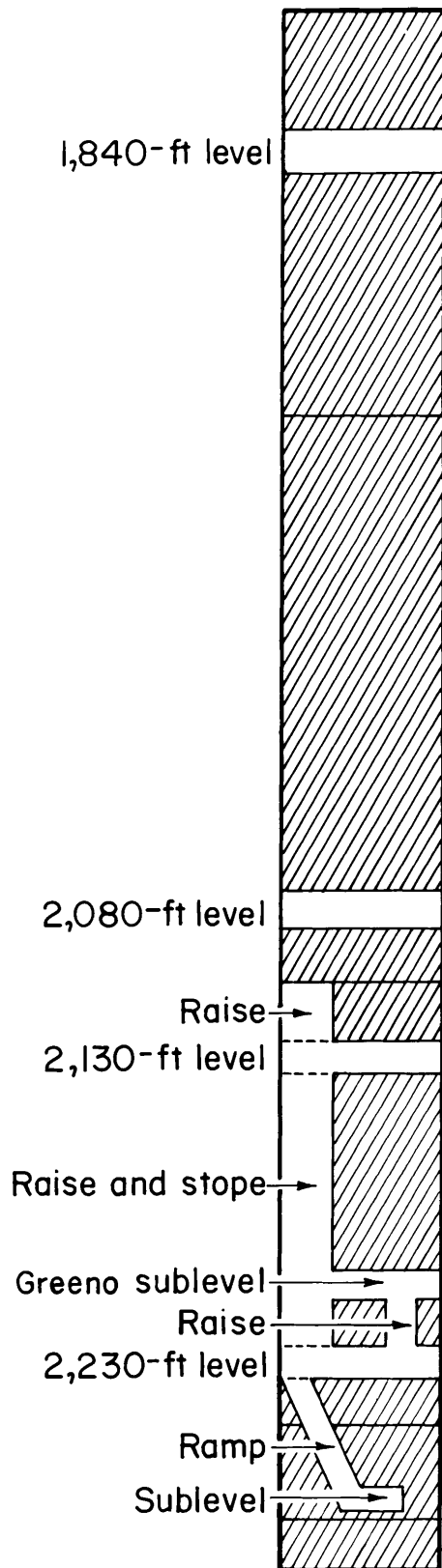


FIGURE 2.—Cross section of Horse Draw Mine. (Adapted from Cole (3).)

The relationship between methane and oil shale is supportable on a theoretical as well as an empirical basis. It is generally accepted that the preservation, degradation, and thermal alteration of organic matter in sediments can lead to the formation of methane in fossil fuels. Methane is known to be an inherent by-product of the coalification process. Methane associated with petroleum is usually considered the result of thermal cracking, the breaking down of complex hydrocarbon chains under intense heat and pressure conditions into lighter and simpler compounds. Genetically, oil shale is related to certain unusual coals containing abundant spore and/or algal material. These coals have historical significance, but little current economic importance.

Petrologic studies can be very useful in identifying the specific progenitors of solid organically derived fuels. The petrographic identification of organic matter in Green River oil shale is usually difficult owing to interactions with the mineral matrix, the fine-grained nature of the organic and mineral matter, and the disseminated state of the organic material. However, there is general agreement that the organic matter in the Green River oil shale is chiefly formed from algae. Partially because of the relationship between oil shales and algal coals, some of the same controls on gas content in coals (depth of burial, temperature, age, geologic structure, etc.) have been suggested for oil shale. The decomposition of algae may also play an important role in petroleum genesis from oil source rocks (5).

The Bureau recently undertook field experiments at the Cathedral Bluffs Mine (fig. 1) to determine the amount of gas contained in oil shale and the behavior of the gas within the oil shale and in the mine atmosphere. Two holes were core-drilled horizontally from the Upper Void Level, each hole being about 200 ft deep. These holes were drilled into the face of an exploration drift. They were oriented parallel to each other and separated by about 25 ft at the hole collars. In addition, six vertical holes were core-drilled from the Upper Void



Level to between 30 and 40 ft below the mine floor.

The drilling of each hole was integrated with experimental tasks. The main tasks were (1) to quantify the amount and composition of gas emitted from both the core and the hole, (2) to count and locate fractures intersecting the core, and (3) to record detailed core lithology. Following removal of an interval of rock sample, the sample length was measured, depths and orientation indicators were marked on the core, core loss intervals were assigned if necessary, and core fractures were logged. Fracture orientation data were meaningful only relative to other fractures within a single reconstructed core interval because the core did not have an absolute orientation when drilled. Lithologic data were recorded next. All core logging was done in accordance with Hartley and Beard (6), standard procedure for the Cathedral Bluffs Mine. Once the descriptive lithologic logging was complete, the cored intervals were inserted into polyvinyl chloride (PVC) or steel containers. The containers were then sealed so that gases desorbing from the core would remain within the container.

At the time of the planning of this study, there was no acceptable test to determine quantities of gas desorbing from oil shale. The direct method test, designed to quantify methane in coal samples, has been used by the Bureau and other investigators on coal (7). However, adaptations of direct method testing to potential methane-emitting rock samples other than coal can produce less than satisfactory results. One problem is that preliminary data indicate that an oil shale sample may emit only one-tenth

the amount of gas as that emitted by a coal sample of equal size. Smaller quantities of gas can lead to erroneous results when the amounts of gas released approach the sensitivity of measurement. Because oil shale can generally be expected to contain less methane than coal, the sensitivity of direct method testing would pose a greater problem. A second problem results from the chemical composition of desorbing gas. Bureau experience with U.S. coalbed gas has identified its composition as generally at least 90 pct CH<sub>4</sub> and as high as 99 pct CH<sub>4</sub>. The direct method test incorporates no gas analysis in the technique and assumes all gas to be methane. Without substantiation of similar oil shale gas composition or the incorporation of gas composition analytical techniques, oil shale gas determinations by the direct method would be inadequate.

A modified direct method test has been designed to measure an amount of gas which is similar to that emitted from mined rock into the mine environment. This gas is normally diluted by ventilation air flow. The modified direct method can produce methane content data under field or laboratory conditions with an accuracy superior to that of the direct method. Reactions between gases emitted from the ore or within the mine atmosphere can be observed by compositionally analyzing and quantifying changing volumes of gas compounds. Quantifying standard temperature and pressure (STP) gas volumes at each point in time produces information on rates of gas released and reactions, which is considered essential in understanding methane emissions in oil shale mines.

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## REGIONAL GEOLOGY

## GEOLOGIC AND DEPOSITIONAL HISTORY

The Green River Formation, located in the States of Wyoming, Colorado, and Utah, contains one of the world's largest known deposits of oil shales. Deposition of these units took place in response to orogenic activity and subsequent downwarping on the western slope of what are now the Rocky Mountains. Two large intermontane lakes were formed. Lake Uinta occupied the region known as the Piceance Basin and parts of Utah, and persisted for 5 to 8 million yr (1). The combined maximum areal extent of the two lakes was about 20,000 mi<sup>2</sup> (1). Evidence may exist for the initiation of mountain building in the Precambrian, although control of sediment distribution did not begin until the development of the structural units in the Pennsylvanian, as described by Murray and Haun (8). The Green River Formation was formed during the Eocene epoch. The depocenter for the major oil shale units is located about 25 miles southwest of Meeker, CO. Several tuffaceous layers found in Green River oil shales are frequently used as marker beds and were produced during volcanic episodes concurrent with orogenic activity.

The deposition of Green River oil shales resulted from a combination of conditions that led to preservation of the organic material. The intermontane lakes were quite saline, and conditions of surface waters were such that a food supply was available for micro-organisms to flourish (9). The lakes were sufficiently saline near the bottom so that the water was stratified, preventing mixing of the dense saline water with the more oxygenated surface water. Organic material flowing to the lake bottom was preserved under reducing conditions. The Mahogany beds make up a persistently rich layer of oil shale ranging from 50 to 200 ft thick in the Piceance Basin. During the deposition of the Mahogany beds, the formation of Piceance Basin saline minerals was most abundant near the basin's center. Throughout the basin, saline

minerals are preserved below the dissolution surface and are leached out above the surface. Leaching of salines leaves vuggy porosity, which is well developed above the dissolution surface.

Evaporite minerals are associated with oil shales in many areas of Colorado and Wyoming. The most abundant of these minerals are nahcolite (NaHCO<sub>3</sub>), halite (NaCl), dawsonite [NaAl(OH)<sub>2</sub>CO<sub>3</sub>], and trona (Na<sub>2</sub>CO<sub>3</sub>·NaHCO<sub>3</sub>·2H<sub>2</sub>O). Nahcolite beds are common in the oil shales of Colorado's Piceance Basin as interbedded deposits showing growth structures that deformed surrounding oil shale bedding. Evaporation and recession of the lake shoreline may have been important in deposition of the evaporites. Another mechanism for formation of these minerals is possible because of the production of CO<sub>2</sub> during the decay of organic matter (10).

## STRATIGRAPHY

The oldest unit of the Eocene Green River Formation is the Douglas Creek Member which is made up of light-colored sandstone and shales, as well as some limestone. In the northern portion of the Piceance Basin, the Douglas Creek grades into the overlying Garden Gulch Member, which consists of gray marlstone, gray and brown shale, and some thin beds of oil shale. In the vicinity of the Cathedral Bluffs tract, the two units have a combined thickness of approximately 500 ft. The Parachute Creek Member lies above the Garden Gulch. The Parachute Creek Member is a gray, brown, and black marlstone with the persistent dark, rich oil shale beds such as those in the Mahogany Zone. Near the mine location, the total thickness of the Parachute Creek Member is about 1,500 ft. The Uinta Formation rests on the Parachute Creek Member and is composed of gray or brown sandstone interbedded with marlstone and some thin oil shale beds (1).

The Parachute Creek Member of the Green River Formation contains the principal

oil shale units. Stratigraphic units in the Parachute Creek are frequently differentiated in terms of oil yield due to the vertical lithologic consistency of the oil shales. The richness or leanness of the oil shale unit is signified by R or L, respectively, as in figure 3. (R7 is commonly known as the Mahogany Zone.) Figure 3 shows a generalized stratigraphic column for the Horse Draw Mine and Cathedral Bluffs Mine sites. The locations of the mine workings are superimposed on the rock units. Although the elevations of the two mine sites are very different, the columns are matched at the top of the A-Groove unit, showing the differing mining horizons.

### STRUCTURE

Within the Piceance Basin Eocene units, structural trends are quite gentle. In Rio Blanco County, there is a series of synclines and anticlines with axes generally ranging from east to west and from northeast to southwest (8). Dips are limited to no more than a couple of degrees. On the Cathedral Bluffs tract, beds strike east-west and dip northward about 150 ft/mi (approximately 1.5°) (11).

Underground mapping at Cathedral Bluffs revealed a few small-scale folds. The largest fold in the Lower Void Level was about 10 ft in amplitude and disrupted a vertical section about 50 to 75 ft in height. The fold axis had a strike of N 40° E, and the axial plane of the fold dipped 55° NW. Folds of this magnitude can be considered local features.

Joint patterns on the mine tract have been noted by previous investigations. Stellavato recorded joint orientations at the surface and at all five subsurface levels in the mine (11). One set of joints was oriented at N 75° W with dips increasing from 66° on the surface to 90° at the Upper Void Level. Joint dips were found to increase with depth. In particular, pronounced joint steepening was measured just below the A-Groove. Also, joints were found to be persistent in lean oil shale units and to end abruptly

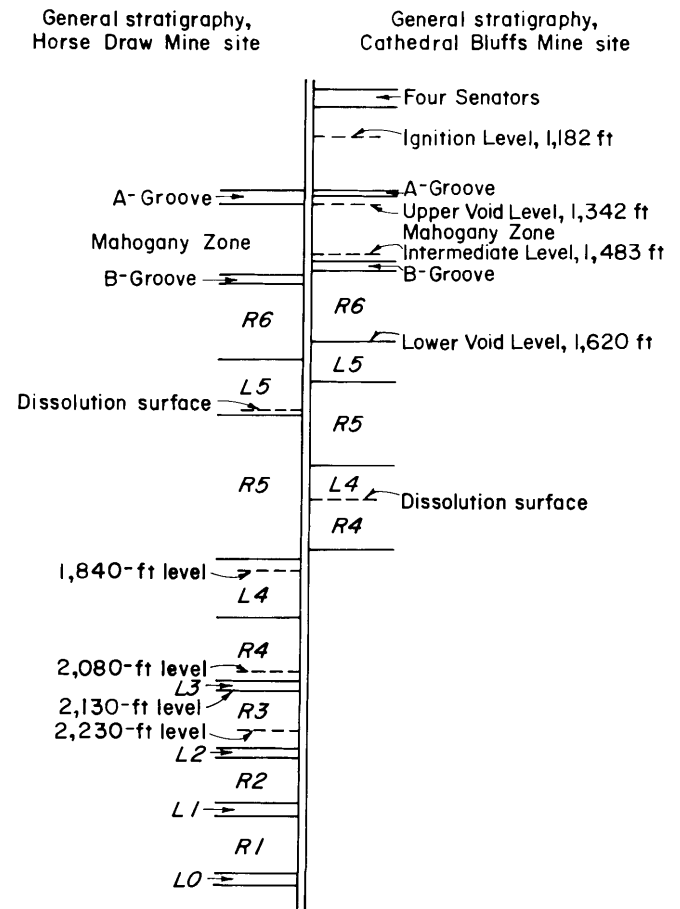


FIGURE 3.—Green River stratigraphy and location of mine workings at Horse Draw Mine and Cathedral Bluffs Mine sites.

at richer units. At the service and production shafts in the Upper Void Level, other joints had strikes of N 76° W, N 57° E, N 60° E, N 78° E, and east-west.

Tuff dikes are frequently encountered in the Cathedral Bluffs Mine workings. They generally follow established joint patterns, especially the northeast-trending joints. Numerous tuff dikes were encountered on the Upper Void Level, ranging from 3 to about 12 in thick (11). The contact between the dike and the oil shale beds ranges from sharp and abrupt to very irregular. The source of the dikes may be a curly bedded tuff, which shows characteristic thinning and thickening and achieves a maximum thickness of 18 in (11). This tuff is located just above the B-Groove (fig. 3).

## DRILL EQUIPMENT AND DRILL-HOLE CONFIGURATIONS

An electrohydraulic drill and powerpack unit was used for drilling all coreholes for the Bureau study at the Cathedral Bluffs shale oil mine site. The drill unit had a 30-in feed and independently variable thrust and rotational controls. All of the holes were drilled with NQ (2.75-in OD) drill rod and a wire-line core-retrieval system. Horizontal coring was done with 10-ft rods and a 10-ft core barrel. Because of height limitations at the drill sites, the six vertical holes were drilled with a 5-ft core barrel and 5-ft drill rods. Roof heights had a wide range: less than 13 ft for holes UV3 and UV4 and over 30 ft for holes UV5 and UV6. The width of the Upper Void Level workings, as measured near the horizontal hole collars, was slightly over 30 ft.

An assortment of NQ core bits of differing configurations were used. The best core-drilling results were achieved by using a surface-set diamond bit with a semiround crown and internal discharge. In bit designs with conventional waterways, drill water pressure below about 180 psig was not sufficient to keep drill cuttings from becoming lodged between the inner barrel bit and the inside annulus of the drill bit.

A platform was constructed on which to mount the drill for drilling the vertical holes. It was constructed of 1/8-in steel plate and H-beams, and it situated the drill about 50 in above the drill floor. Scaffolding was assembled to reach the required locations for drill string assembly, disassembly, and maintenance.

The face drilled for the collars of horizontal holes UH1 and UH2 was stepped in shape (fig. 4). The added thickness of oil shale at the UH2 hole collar was highly fractured, probably as a result of blasting. Core drilling through this zone was highly inefficient since the drill tools had to be pulled from the hole many times, as the result of loss of drill fluid through unusually large fractures. The highly fractured rock also

had a tendency to become lodged in the inner core barrel. A split aluminum barrel that fit inside the inner barrel was used on the second horizontal hole drilled (UH1). This provided a much improved method of removing the core from the barrel. Fractures frequently almost paralleled the axes of the holes, which created a tendency for pieces of the core to lodge in the core barrel. The split barrel reduced the time required to remove the core from the barrel. A split inner barrel is also recommended if any detailed and/or quantitative analysis of the core is planned. The fractured portion of UH2 was penetrated by rotary drilling with a 3-in steel drag bit and reamed (the core drill string was larger in diameter than a 3-in hole) with a 6-in pilot and reamer drag bit.

The spacing of the six vertical holes ranged from approximately 55 to 165 ft (fig. 4). The terminal depth of each of these holes was planned to be 40 ft below the Upper Void Level mine floor. The

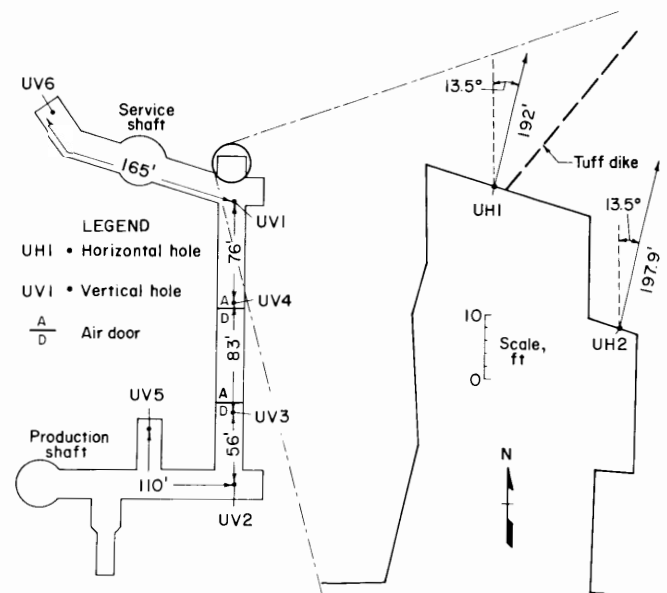


FIGURE 4.—Location of holes drilled in Cathedral Bluffs Upper Void Level, with an enlarged view of the horizontal holes site.

depth at which core drilling began in each hole varied because of drill site conditions. Ideally, core drilling would begin at the mine floor so that the entire drilled interval could be recovered. However, only holes UV2 and UV5 were cored entirely, starting from the Upper Void Level mine floor. At all other vertical hole locations, the presence of muck accumulations of varying thicknesses deterred core drilling the mine floor. Core drilling from the mine floor was

attempted on hole UV1, but the influx of rubble necessitated reaming the hole with a drag bit. Holes UV4 and UV6 were rotary-drilled to depths of 4.1 and 1.7 ft, respectively, where core drilling was begun. A hole was dug through the muck to establish the collar of hole UV3. A short section of 8-in steel pipe was cemented in place at a depth of 4.2 ft, and coring was initiated through the pipe. Bedrock was encountered 4.5 ft deep in the hole.

#### SITE GEOLOGY AND CORE LITHOLOGY

##### SITE GEOLOGY

In projects of this nature, drilling activities frequently consume so much time that little is left for detailed geologic investigations. The most time available for observation in this study was at the site of the horizontal holes. Continuous laminations in the oil shale were easily traced across a rib, the face, and the adjacent rib, although color changes and sometimes thickness variations occurred within the confines of this site. Fracture or joint orientations were measured on a few locations and were found to be striking very near true north, roughly east-west, and also about  $N 45^{\circ} E$ . All were steeply dipping. This is in general agreement with far more detailed data reported by Stellavato at the same locations (11).

A bed of nahcolite persisted about 9 ft above the mine floor, which showed boudinage-like growth structures throughout. The bed ranged in thickness from a couple of inches to about 2 ft. A tuff dike striking  $N 39^{\circ} E$  was intercepted by UH2 and was readily visible in the rib adjacent to UH1. Holes UV3 and UV4 were situated near several identifiable dikes, corresponding to the area where Stellavato had mapped four tuff dikes on the Upper Void Level (fig. 5). Most of the roof on this mining level is shotcreted to deflect water drippage around the openings, making geologic observations impossible in these localities.

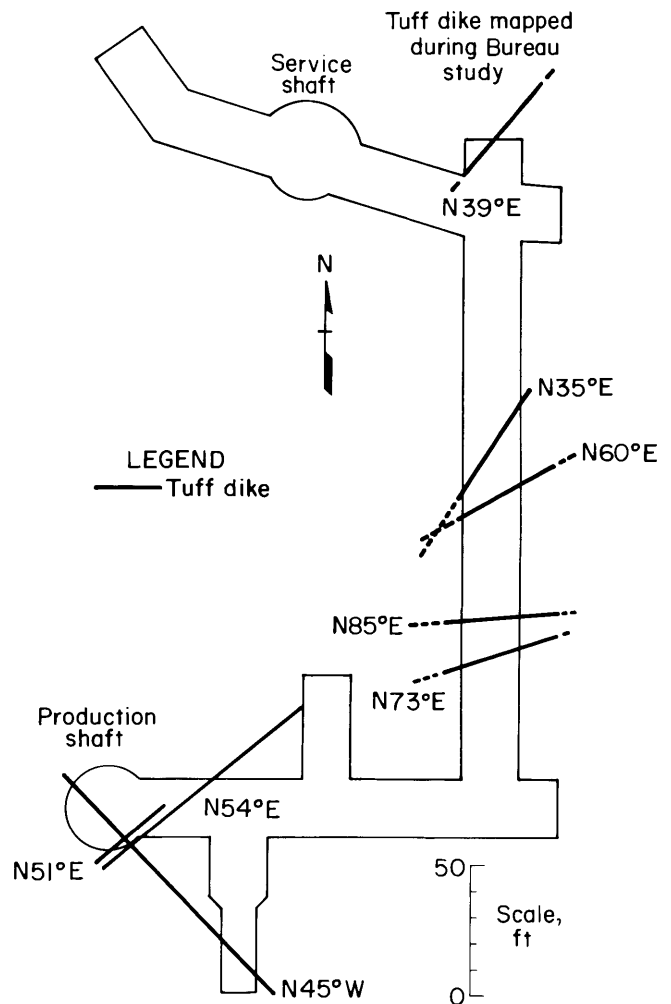


FIGURE 5.—Map view of Cathedral Bluffs Upper Void Level showing locations of tuff dikes. (Adapted from Stellavato (11).)

## VERTICAL HOLE CORE LITHOLOGY

Detailed lithologic descriptions of the cores were made before the cores were inserted into desorption cannisters. Following completion of desorption testing, the cores were removed from the airtight containers, and each of the six vertical holes were reconstructed and situated next to the nearest adjacent core in accordance with the drill hole sequence (fig. 4). No reliable marker bed was observed within the vertically drilled sequence to facilitate a lithologic correlation between holes. Apparent rich and lean oil shale zones could not be effectively used for correlation probably because of laminae thickening and thinning and lateral changes in laminae color. The top of each hole, the Upper Void Level minefloor, was used for stratigraphic correlation.

Core lithologies described below can be observed in figure 6 (in pocket). Fractured zones and vugs partially filled with clay were encountered between about 32 and 40 ft in four of the six vertical holes. These were zones of variable thickness. The holes with the greatest lateral distance to adjacent holes were the two holes at the end of the vertical sequence, hole UV5 (about 110 ft) and hole UV6 (about 165 ft). Holes UV5 and UV6 are characterized by harder, denser oil shale with fewer fractures than the inside four holes have. The core from UV5 and UV6 was removed from the core barrel in fewer pieces. Occasionally, core from these holes was removed from the 5-ft inner barrel completely unbroken.

### UV1

Zones of core loss in UV1 occurred between footages 5.9 and 8.4, 17.6 and 20.2, 36.2 and 36.8, and 44.7 and 45. Solution fractured and brecciated zones were located between 34.2 and 37.6 ft, 43 and 43.7 ft, and 44.5 and 44.7 ft. Gas flowed from the hole upon penetration

below 36.2 ft, a core loss interval, and just below 41 ft in an area of oblique and longitudinal fractures. Methane-rich gas also emanated from the bottom two fractured-brecciated zones. The first and third of these gas-producing horizons also ejected water from UV1. Tuffaceous layers were located at 8.6 to 8.7 ft and at 25.8 to 26.4 ft, pyritic layers from 20.2 to 21.8 ft, and 26.5 to 30.6 ft. A marl-rich interval was intercepted between 4 and 5.9 ft, and calcite and kaolinite mineralization frequently accompanied fractured and vuggy horizons. Bitumen appeared only sporadically in UV1.

### UV2

Two zones of core loss were located in UV2; from 4.4 to 5 ft and from 31.3 to 32.3 ft. Other vuggy and/or highly fractured zones were located at depths of 34.7 to 36.8 ft and 39.2 to 40 ft. Marl-lime-rich horizons were found near the top of the hole from 1.7 to 3.5 ft and from 5.2 to 8.4 ft. Bitumen is abundant in the upper two thirds of the hole, occurring with kaolinite and pyrite between 0 and 3 ft. Another bitumen-rich layer occurs between 15.5 and 17.5 ft, with associated marly streaks. Below this zone, pyrite, kaolinite, and calcite, are found in zones of fractured vuggy oil shale.

### UV3

Hole UV3 penetrated densely fractured and vug-prone zones from 27.4 to 28 ft, from 33.2 to 35.5 ft, and from 36 to 39.2 ft. Core loss intervals occurred just below the first and last of these zones, and the methane-producing horizons in UV3 were located just above the uppermost zone and in the middle zone. Bitumen and pyrite were abundant, with the greatest combined concentrations found between 13.5 and 28 ft and between 31 and 34.5 ft. Calcite, kaolinite, and some pyrite were associated with the upper two fractured zones.

## UV4

Three core loss zones were penetrated in hole UV4--between 13.4 and 14.8 ft, between 20.9 and 21.9 ft, and between 37.7 and 39.2 ft. Water and methane-rich gas were discharged from the middle zone and also from a thin kaolinitic and bitumen-rich zone from 12.3 to 13.7 ft. Additional zones of dense fracturing and dissolution were found from 15.3 to 17.5 ft, 33.1 to 36.3 ft, and 36.9 to 37.6 ft. The lithologies below 29.5 ft were highly tuffaceous; a tuff dike occupied about a third of the cross section of the core from 29.5 to 32 ft, and the lower two vuggy zones were very tuff rich. The top of a 0.6-ft tuff layer was located at 26.3 ft. Kaolinite and pyrite were found in fractured and water-producing zones; bitumen and pyrite were found from 4.1 to about 7 ft.

## UV5

Core from hole UV5 was characterized by "poker chips" (closely spaced, regularly occurring partings) from 1.6 to 7.2 ft and 18 to 22 ft. Outside these portions,

the cored intervals were mostly unbroken and without core loss zones. Kaolinite mineralization was generally associated with fractures between 1.7 and 3 ft, 17 and 20 ft, and 21 and 29 ft. Two zones of solution fractures were encountered, from 23.5 to 27.5 ft and from 35.5 to 37.5 ft. Limy zones with associated tuff stringers were intercepted between 9.5 and 12 ft and 30 and 31 ft. Bitumen was found between 17 and 20 ft. Finely disseminated pyrite crystals were located between 34.5 and 37 ft. Two zones of small vugs (usually less than 0.5 in across their longest axis) were logged from 21 to 30 ft and 31.5 to 37 ft.

## UV6

The most frequent material identified in the UV6 core was bitumen. The bitumen-rich intervals were located between 9 and 10.6 ft, 15.6 and 18.5 ft, and 25.5 and 29 ft. Infrequent calcite and kaolinite mineralizations were found on fracture planes. Methane gas was encountered in hole UV6 at 16.5 ft; gas flow was greatly reduced after 5 min.

## RESULTS OF MODIFIED DIRECT METHOD TESTING

The modified direct method test (MDM) was conducted on essentially all cored oil shale samples drilled during the course of this study. A total of 631.1 ft of oil shale was core-drilled, although all core was not recovered. The recovered shale was divided into 76 horizontally cored desorption samples and 59 vertically cored desorption samples. Sample lengths were usually determined by the length of core recovered in a single core barrel run.

The 135 oil shale core samples sealed in airtight containers were gas-sampled repeatedly during the study. Each sampling included recording the date and time of sampling, measuring the differential pressure between the container atmosphere and the mine atmosphere, retrieving a gas sample of the container atmosphere, bleeding the container gas pressure to mine atmospheric pressure, and recording the temperature and atmospheric pressure at the underground

work station. The duration of gas desorption monitoring was nonuniform because measurements began as soon as the core sample was removed from the hole and placed in the airtight container. As a result, the position of the sample in the hole and in the drill hole sequence determined the duration of desorption monitoring.

The concentration of gases at each sampling was multiplied by the STP total contained gas volume. After the completion of gas sampling, the gas volumes can be totaled to find the amount of gas liberated (or consumed) over a known period of time. Some deviations to this procedure did occur, and the MDM test is discussed in greater detail in the appendix.

To compare gas desorption and/or reactions between samples, it was necessary to eliminate inconsistencies in test duration and sample size. Time indices were chosen to facilitate sample comparison. The longest common duration of MDM

testing for a group of core samples, vertical or horizontal, was chosen for the index: a 40-day (960-h) index for the vertically cored samples and a 125-day (3,000-h) index for the horizontally cored samples. The horizontal core sample gases were also indexed at 40 days so they could be compared with the vertical samples. In addition, a 3-day (72-h) desorption time index was determined, to simulate that period of time gas would be released from a muckpile before being removed from the mine environment. All gas totals were divided by the sample mass, providing weight-normalized gas quantities for interpretation. Although there is precedence for metal and nonmetal gas normalization on a basis of cubic centimeters per 100 g, gas normalization on a basis of cubic centimeters per gram was chosen to be more easily related to the convention of existing coal desorption data. Multiplying these numbers by 32 converts the data to cubic feet per short ton.

Previous investigations on coal and oil shale gas desorption have shown a linear relationship for cumulative methane when graphed in terms of the square root of time (7). Figure 7 shows a graph of square root of time in hours against cumulative gas volume per unit weight. The sample shown is labeled UV208, indicating oil shale removed from the eighth coring run on the second vertical hole drilled from the Upper Void Level during this study. This sample is an example of typical behavior of the gases as shown by

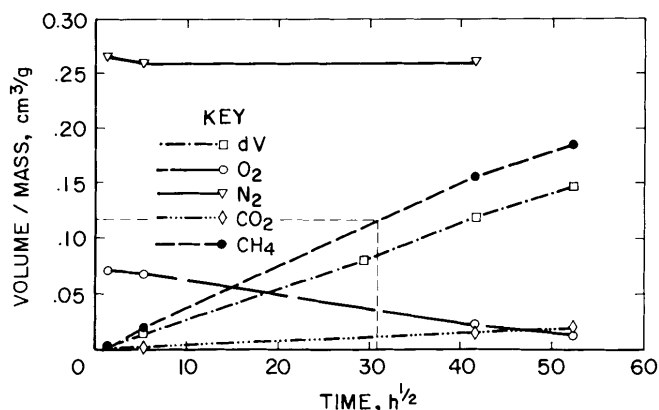


FIGURE 7.—Gases measured within container for sample UV208. The dashed line shows the time index value for methane content at 40 days.

the MDM test in the latter portion of this study. Error analysis of the MDM technique showed an additive effect of inaccuracies in volume measurements. Relatively infrequent gas sampling was important to maintain optimum accuracy in gas content determination. Cored intervals removed from drill holes early in the study were gas-sampled as many as 13 times. Oil shale core produced later in the study was gas-sampled three to six times, and the duration of MDM testing was much shorter.

For sample UV208 (fig. 7), the volume of nitrogen was quite high at the beginning since the mine atmosphere was enclosed in the sealed container at the start of the test. The volume of nitrogen decreased slightly. Oxygen decreased steadily in volume during the test interval. The slope of the oxygen curve is much steeper than the slopes of other atmosphere-contained gases, such as nitrogen, which were not emitted in measurable volumes from the oil shale sample. Presently, it is not clear whether oxygen is lost because of sorption onto surface sites on the oil shale or whether oxygen is consumed by low-level oxidation reactions. The decrease in oxygen was not accompanied by a significant increase in CO or CO<sub>2</sub>, as was noted in some trona and coal mines where self-heating reactions were found to occur (12). CO<sub>2</sub> increased in volume with time. However, the total volumetric increase was small compared with that of the more abundant and more reactive gases. The volume of methane increased markedly over the course of this test. The 40-day indexed value for methane content equals about 31 on the square root of time axis and equals 0.116 cm<sup>3</sup>/g. Oxygen, nitrogen, and CO<sub>2</sub> were also measured by indexing. An indexed methane content value is estimated by interpolating a straight line between the closest data points before and after the chosen time index. Figure 7 shows a substantial increase in overall gas volume (dV) over time. The total increase in gas volume is smaller than the increase in methane alone, largely because of the volumetric decrease in oxygen. Although sample comparisons favor normalizing gas volumes on



a weight or mass basis, figure 8 shows the changing contained gas volumes in cubic centimeters for sample UV310, to show the scale of the measured data from a 5-ft core sample of oil shale.

A linear increase in methane from oil shale over time as measured in the square root of hours was found not to be exact in many cases. Similar data plots of a number of samples show a tendency towards curvature. The curves are generally concave down. The curved methane plots have been fitted to second-degree polynomials very successfully, with the coefficient of determination ( $r^2$ ) values generally in excess of 0.98. Another graph was made to better discern whether the second-degree polynomial was a better description of the oil shale desorption data. A sample was chosen that was gas-sampled frequently and that showed a relatively linear increase of cumulative methane volume when graphed against the square root of time (fig. 9). The difference in gas volume between successive points for sample UV102 was found and divided by the amount of time, in hours, that passed between the two points. These gas volumes were graphed against time in hours to show how much methane was released per unit time from the core and to illustrate how these volumes changed over time (fig. 10). The curve shows the largest amounts of gas released per unit time were at the beginning of the MDM test. The curve declines quickly and approaches the X-axis asymptotically.

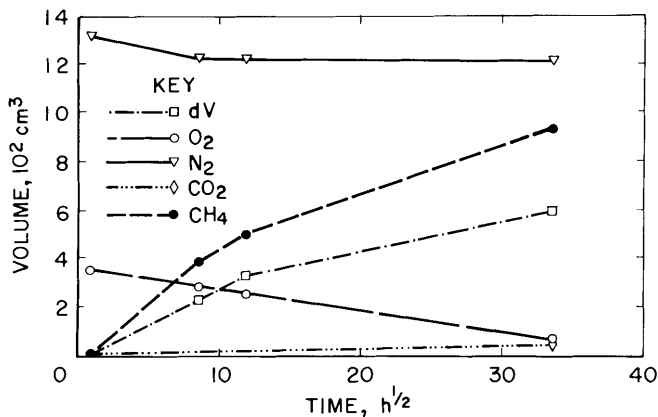


FIGURE 8.—Volumes of gases measured within container for sample UV310.

The aforementioned plot of incremental gas volumes per unit time against time essentially represents the derivation of the graph of cumulative gas versus square root of time. Linear plots on the latter graph have the form  $y = aT^{1/2} + b$ . The first derivative with respect to time would be  $dy/dT = a(1/T^{1/2})$ . Second-degree polynomial curves on the cumulative-gas-versus-time graph have the form  $y = a(T^{1/2})^2 + bT^{1/2} + C$ , where the first derivative with respect to time is  $dy/dT = a + b(1/T^{1/2})$ . A graph of  $y = (1/X^{1/2})$  is very similar to the shape of the curve shown in figure 10. The derivative of the second-degree polynomial is very similar to the derivative of the linear, except that a constant is added. This would increase the Y-intercept of the plot. Since the gas desorption rate is high at the beginning of MDM testing but declines so rapidly, infrequent gas

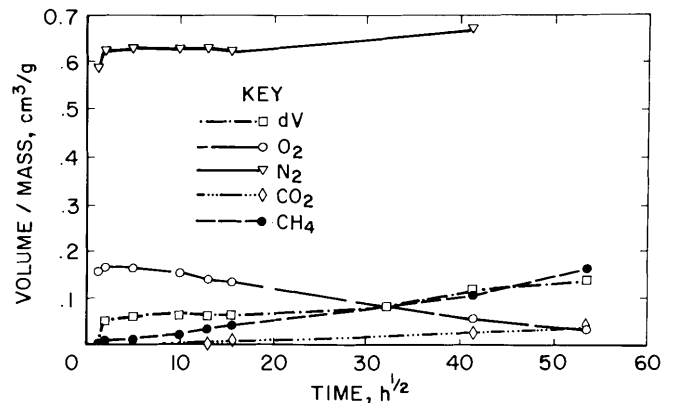


FIGURE 9.—Gases measured within container for sample UV102.

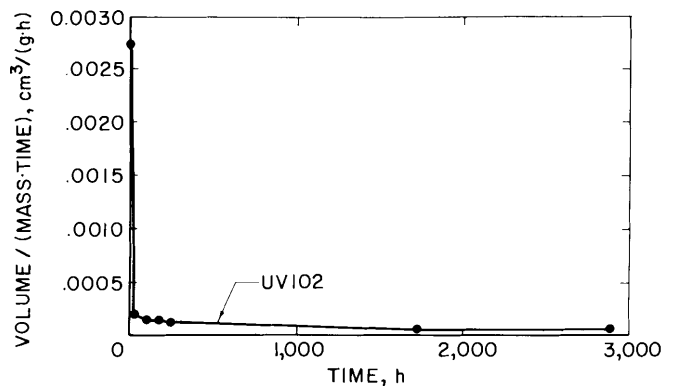


FIGURE 10.—Amount of methane desorbed from sample UV102 between each gas sampling, divided by the amount of time elapsed between each gas sampling.

sampling at the beginning of the test averages high rates of desorption with lower ones that occur shortly thereafter. More frequent sampling at the beginning of gas monitoring reduces the averaging of high and low desorption rates and increases the Y-intercept of the graph. This is consistent with the form of the first derivative of the second-degree polynomial. It appears that sampling frequency and duration of testing can affect the shape of the graph of cumulative gas versus square root of time and that second-degree polynomial curve fits to some graphs are better approximations of the observed desorption phenomena.

An example of a successful linear curve fit is given in figure 11. Sample UV303 is shown with gas volumes normalized per unit mass in figure 11A. The methane content of this sample is lower than that of the majority of Cathedral Bluffs samples. At the 40-day time index ( $31 \text{ h}^{1/2}$ ), UV303 desorbed  $0.0750 \text{ cm}^3/\text{g}$   $\text{CH}_4$ . The curve fit in figure 11B shows methane data only. The time between the Y-axis and the first data point represents the estimated lost-gas period in square root of hours before the sealing of the core

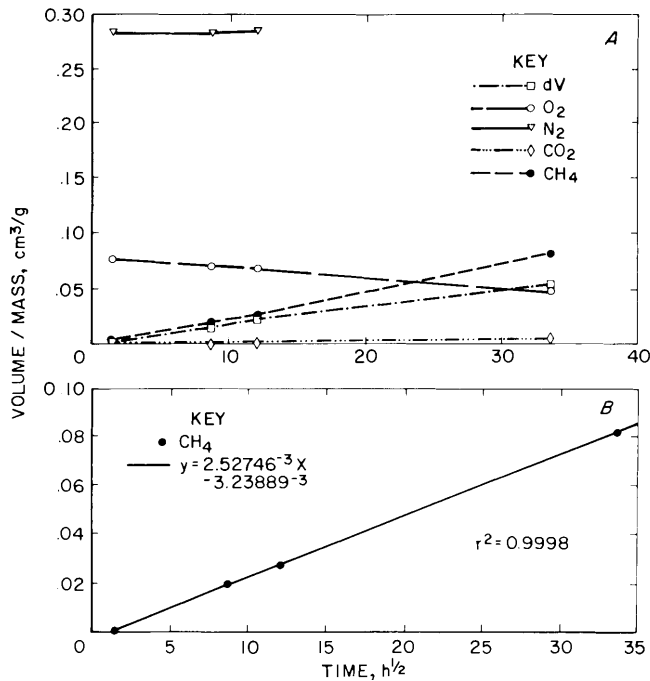


FIGURE 11.—Sample UV303: A, gases measured within container; B, linear regression fit to desorbed methane data.

sample in an airtight container. The nitrogen curve in 11A has three points instead of four possibly because either nitrogen analysis was not done on the gas sample or a gas-sampling or gas-analysis malfunction occurred.

Figure 12A presents a graph of gas volume per unit mass plotted against square root of time for sample UH109. Sample UH109 produced a larger quantity of methane than did most other Cathedral Bluffs samples undergoing MDM testing. The time-volumetric methane indices for this sample are  $0.0804$  and  $0.253 \text{ cm}^3/\text{g}$  at 3 and 40 days, respectively. Also, the duration of testing was far longer for this sample, 132 days, than for those samples presented thus far. The plot shown has been interpreted as an example of a concave-down plot of methane per unit mass versus square root of time. It possesses all three of the qualities recognized as conducive to producing concave-down plots:

1. The duration of testing was long, so that high early desorption rates were combined on the same graph with lower ones occurring later in the test.

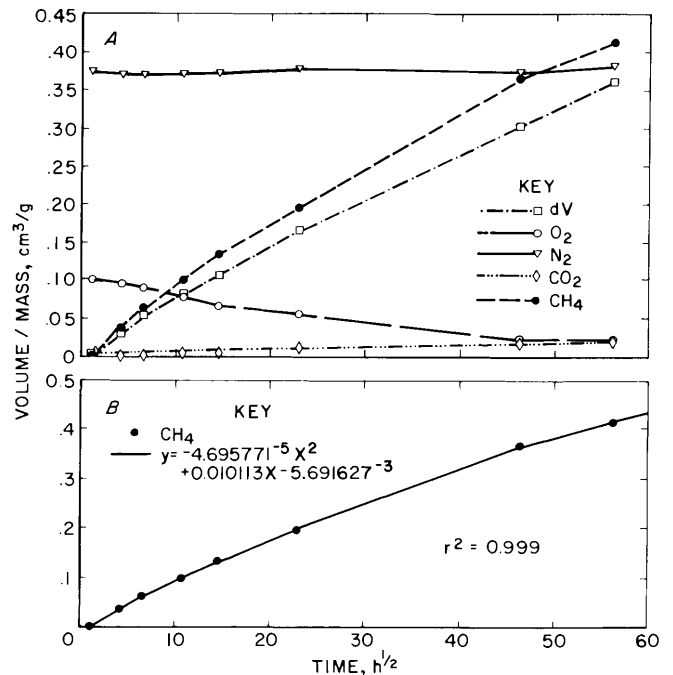


FIGURE 12.—Sample UH109: A, gases measured within container; B, second-degree polynomial fit to desorbed methane data.

2. Frequent gas samples were taken at the beginning of the test to minimize averaging high desorption rates with lower ones that followed.

3. Compared with other samples in the study, this sample is relatively gas enriched.

Figure 12B shows only methane data plotted on a graph of normalized cumulative gas volume versus square root of time. A second-degree polynomial was fitted to this data. For  $X = T^{1/2}$ ,  $y = -4.696 \times 10^{-5}X^2 + 0.0101X - 5.692 \times 10^{-3}$ . This equation fits the data with an  $r^2$  coefficient of determination equal to 0.999. Methane-rich gas flowed from the hole during core drilling at a depth of 45 to 47.5 ft. The UH109 desorption sample interval contains core from 41.8 to 51.3 ft of hole UH1. This core sample is gas enriched beyond the majority of oil shale samples presented here and included a gas-flow-producing zone. An association between methane flows from a drill hole and high methane contents from the same footage interval is tentatively supported.

Hole UH2 also contained examples of gas-producing zones. High-concentration methane gas averaging 95.1 pct, measured by gas chromatography on gas samples, was released from hole UH2 at a depth of about 59.2 ft. Average gas concentrations of  $O_2 + Ar$ ,  $N_2$ ,  $CO_2$ , and  $C_2H_6$  measured 0.45 pct, 2.8 pct, 1.65 pct, and 0.01 pct, respectively. The first signs of a free-flow gas zone were observed at about 57 ft. When the hole was sealed at about 59.2 ft, no pressure buildup was observed; however, the equipment used was not sensitive enough to register a pressure buildup of less than about 10 psi. Gas flow measurements of up to 20,000  $ft^3/d$  were measured following coring to 63.5 ft. Manometer readings of gas flows through an orifice plate were oscillating greatly as water and gas blew out of the hole. The interval from 57 to 63.5 ft was divided between two desorption samples for MDM testing, UH210 and UH211. Unfortunately, gas sampling of sample

UH211 was not successful in obtaining representative contained-gas concentrations early in the duration of MDM testing, and no gas data are available after that time.

Figure 13 shows a plot of the square root of time versus cumulative gas volume per mass for sample UH210. Some deviations from smooth predictable trends of gas behavior can be seen at about the 5- and 29- $h^{1/2}$  times. These have been attributed to atmospheric contamination of the contained gases. This phenomenon is usually indicated by increases in the volume of gases that are abundant in atmosphere, such as nitrogen and oxygen, and a decline in those gases that are not contained in the atmosphere, such as methane (fig. 13A). In terms of the overall sample gases, the erroneous gas data is not extreme, and this graph is still useful in showing trends of gas data. The methane data is fit to a second-degree polynomial in figure 13B. The equation is given, and the  $r^2$

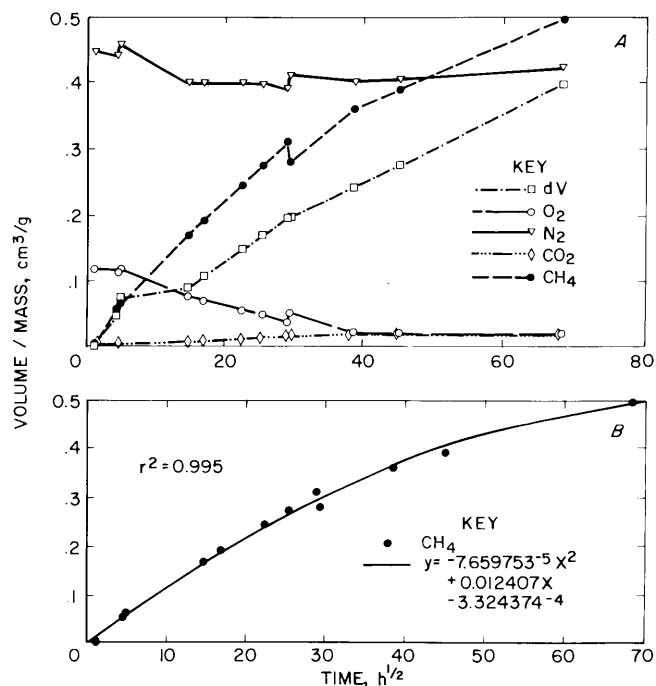


FIGURE 13.—Sample UH210: A, gases measured within container; B, second-degree polynomial fit to desorbed methane data.

coefficient of determination is 0.995. At the 40-day methane time index, sample UH210 has desorbed  $0.295 \text{ cm}^3/\text{g}$ , one of the highest gas quantities in this study. In this case, methane enrichment in the desorption sample corresponds well with gas emissions encountered while drilling the interval.

Methane gas flows were observed again in UH2 at a depth of about 115 ft and at a terminal depth of 197.9 ft. Chromatography on gas samples taken at the hole collar once terminal depth had been reached averaged 80.47 pct  $\text{CH}_4$ , 15.57 pct  $\text{N}_2$ , 3.84 pct  $\text{O}_2 + \text{Ar}$ , 0.44 pct  $\text{CO}_2$ , and about 0.02 pct  $\text{C}_2\text{H}_6$ . The last two desorption samples from UH2 were interrupted during MDM testing by experimental irregularities. The last two desorption samples for hole UH2 that yielded reliable experimental data measured from 172.1 ft to 180.9 and from 180.9 to 182.4 ft and resulted in 40-day indices of methane contents of 0.101 and 0.147  $\text{cm}^3/\text{g}$ , respectively. The deeper of these two samples shows a significant degree of methane enrichment.

Desorption sample UH223 contains the gas-producing horizon at a depth of 115 ft. Figure 14 shows the graph of the square root of time versus cumulative volume divided by mass for this sample. Although an atmosphere leak appears to have occurred during MDM testing, it did not significantly affect the overall

#### IN SITU PERMEABILITY

The in situ hydraulic conductivity determinations were performed using a discontinuum approach described by Bennett and Anderson (13). In this approach, the significant permeability is attributed to the fractures intercepted by the test interval (a 5.5-ft section isolated by inflatable packers); the unfractured rock matrix does not make a significant contribution. The average equivalent aperture of the fissure was calculated, and the average fracture permeability calculation was made assuming a parallel-plate model. Two test methods were used.

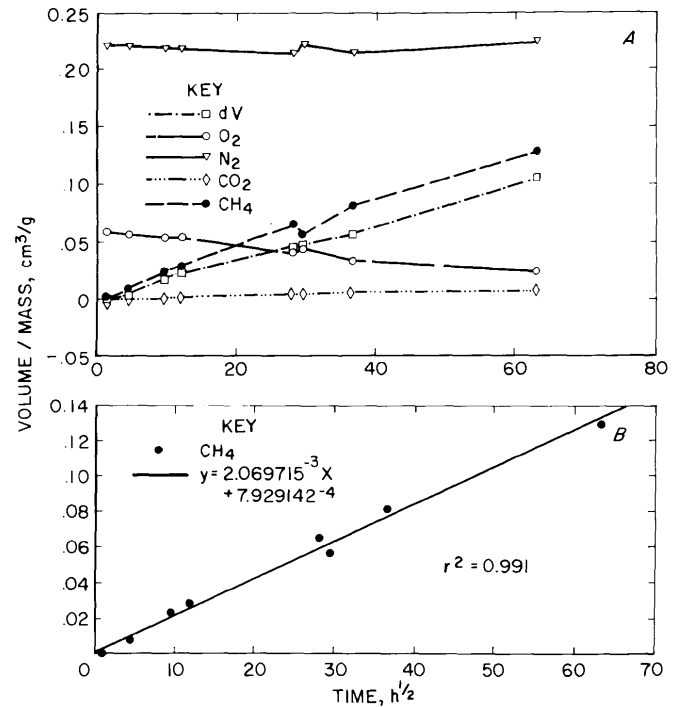


FIGURE 14.—Sample UH223: A, gases measured within container; B, linear regression fit to desorbed methane data.

desorption curve in figure 14B. The 40-day methane index for this sample measures  $0.0619 \text{ cm}^3/\text{g}$ , which is below the 40-day indexed value of the majority of Cathedral Bluffs samples. No quantitative gas measurements were taken from the drill hole at a depth of 115 ft, but gas could be seen bubbling from the returning drilling water.

One measures the steady-state fluid flow from the test interval into the fissure at a constant head or pressure. The other, the falling-head method, is performed by injecting water into a test interval and measuring the pressure drop over a period of time. The average equivalent aperture ( $e$ , in inches) for a constant-head test is calculated from

$$e = \left\{ (1/n) \left[ Q / (2\pi H_0) \right] \left( 12\mu_w / \gamma_w \right) \left[ \ln (R/r_0) \right] \right\}^{1/3}, \quad (1)$$

where  $n$  = number of intercepted fractures;

$Q$  = measured steady-state waterflow rate, gal/min;

$H_o$  = applied waterhead, ft;<sup>5</sup>

$\mu_w$  = dynamic viscosity of water, cP = 1/47,880 lbf·(s/ft<sup>2</sup>);

$\gamma_w$  = unit weight of water, 62.4 lb/ft<sup>3</sup>;

$R$  = radius of influence, which is assumed to be half the test interval length, ft;

and  $r_o$  = radius of borehole, ft.

When the units used in this report are inserted,

$$e = \left\{ \left[ \frac{(Q \text{ gal/min})}{(n2\pi (P - P_o) (1\text{bf/in}^2))} \right] \right. \\ \left. \left[ \frac{12 (1/47,880) \text{ lbf}\cdot(\text{s/ft}^2)}{1} \right] \left[ \ln (R/r_o) \right] \right. \\ \left. \left[ \frac{(231 \text{ in}^3/\text{gal})}{(60 \text{ s/min}) (12 \text{ in/ft})^5} \right]^{1/3} \right\},$$

where  $P - P_o$  = pressure differential.

Simplifying the above equation yields

$$e(\text{in}) = 8.514 \times 10^{-4} \text{ ft} \left\{ \left[ \frac{Q}{(n(P - P_o))} \right] \ln (R/r_o) \right\}^{1/3}. \quad (2)$$

$R$  and  $r_o$  should be measured in the same units so that  $R/r_o$  becomes a dimensionless quantity.

The permeability ( $K$ ) of the average fissure from above is

$$K = e^2 \gamma_w / 12 \mu_w.$$

The intrinsic permeability ( $k$ ) is given by

$$k = K(\mu_w/\gamma_w) = e^2/12.$$

Substituting for  $e$  and simplifying,

$$k = 6.041 \times 10^{-8} \left\{ \left[ \frac{Q}{(n(P - P_o))} \right] \ln (R/r_o) \right\}^{2/3}. \quad (3)$$

Converting the above equation from square feet to millidarcies and simplifying gives

$$k = 5.686 \times 10^6 \text{ mD} \left\{ \left[ \frac{Q}{(n(P - P_o))} \right] \ln (R/r_o) \right\}^{2/3}. \quad (4)$$

The equivalent aperture concept is used because the surface roughness of the fracture was not known. The equivalent aperture is essentially the width of the fracture if that width is conceptualized as being wide enough such that the real surface roughness of the fracture becomes quite small. A parallel-plate model is used to approximate the fracture configuration to determine its permeability.

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<sup>5</sup>Bennett and Anderson (13) measured waterhead in feet. A conversion factor of 144  $(P - P_o)/\gamma_w$  is inserted into equation 1 to convert  $H_o$  to units of pounds per square inch to be consistent with other units in this report.

For the falling-head test, the average equivalent aperture is calculated by

$$e = \{ [r_o^2 / (2n\Delta t)] (12\mu_w / \gamma_w) [\ln (H_{o_1} / H_{o_2})] \ln [R / R_o] \}^{1/3}. \quad (5)$$

Substituting the units used in this report, the above equation for e becomes

$$e = \{ [(r_o^2 \text{ ft}^2) / (2n \Delta t \text{ min})] [12 (1/47,880 \text{ lbf} \cdot (\text{s}/\text{ft}^2)) / (62.4 \text{ lbf}/\text{ft}^3)] [\ln (H_{o_1} / H_{o_2})] [\ln (R/r_o)] [1/(60 \text{ s}/\text{min})] \}^{1/3},$$

where  $H_{o_1}$  and  $H_{o_2}$  = test interval pressures at  $t_1$  and  $t_2$ , respectively, psi;

$t_1$  and  $t_2$  = initial and final times, respectively, corresponding to a set of permeability data;

and  $\Delta t = t_2 - t_1$ , min.

Simplifying the above equation yields

$$e(\text{in}) = 3.223 \times 10^{-3} \{ [r_o^2 / (n\Delta t)] [\ln (H_{o_1} / H_{o_2})] [\ln (R/r_o)] \}^{1/3} \text{ ft}. \quad (6)$$

All other variables have the same meaning and are measured in the same units as in the equation for the constant-head case.

Under pressure-drop conditions,

$$k \text{ (single fracture, intrinsic)} = (\mu_w / \gamma_w) [r_o^2 / (2n \Delta t e)] [\ln (H_{o_1} / H_{o_2})] [\ln (R/r_o)]. \quad (7)$$

After the units used in this report are inserted, a substitution is made for e, and the equation is simplified, it is found that

$$k = 8.654 \times 10^{-7} \text{ ft}^2 \{ [r_o^2 (\ln (H_{o_1} / H_{o_2})) \ln (R/r_o)] / (n\Delta t) \}^{2/3}.$$

This expression gives the intrinsic permeability of the average single fracture in the test zone in units of square feet. To find k in terms of millidarcies, it is derived that

$$k = 8.146 \times 10^7 \text{ mD} \{ [r_o^2 (\ln (H_{o_1} / H_{o_2}))] [\ln (R/r_o)] / (n\Delta t) \}^{2/3}. \quad (8)$$

Two intervals of hole UH2 were chosen for in situ permeability testing. Both intervals had produced methane gas flows from the hole during core-drilling operations. The test interval length, the separation between the two inflatable packers, was identical for both tests: 5.5 ft. The first test zone was located between 63 and 68.5 ft. This portion of the hole was seen to be transected by fractures during logging; a total of 13 were counted. A constant-head test was conducted within this interval. Water was used as the test fluid for this and all other permeability tests conducted at the site during the study. The 63.0- to 68.5-ft section was tested at pressures of 565 to 600 psig.

Graphs made from the experimental data show how both equivalent aperture and fracture permeability varied with increasing pressure. In the shallow zone test (fig. 15A), both the equivalent aperture and the permeability decreased with increasing pressure. It is difficult to account for this behavior. Test pressures were below the hydraulic fracture gradient as determined by Briedenhof (14). The personnel on-site indicated that the pump used during the test was functioning at maximum flow capacity in generating the 600-psig pressure. Although the gauge at the collar of the hole registered 600 psig, an unstable pressure differential within the tested interval could account for the performance shown in figure 15A. Those portions of the test section at the greatest distance from the hole collar may not have

been pressurized to the test pressure because test fluid was removed via fracture permeability more rapidly than pressurized water was flowing in. If this condition did exist, it would eventually produce falling pressure at the gauge, but the test duration of about 68 min may not have been long enough to record a pressure drop at the gauge. The data do show that permeability and fracture aperture decreased with time, with the test pressure beginning at 560 psi and ending at 600 psi. The unusual relationship observed between fracture aperture and permeability and fluid pressure may also be attributable to alteration of the in situ stress field in the vicinity of the shaft by shaft-sinking operations. The capacity of the induced fracture reservoir may have been too large for the test fluids to fill.

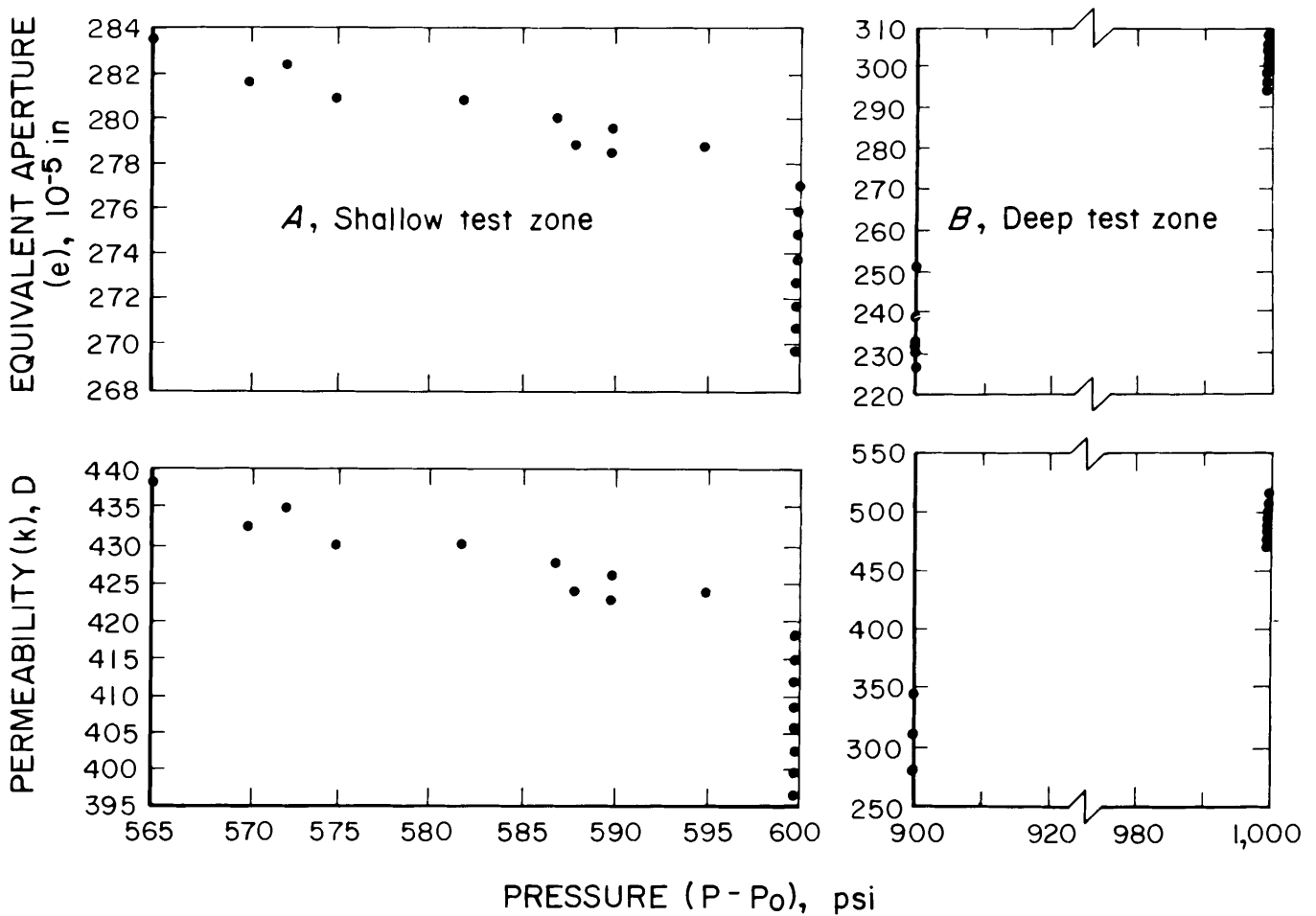


FIGURE 15.—Hole UH2: variation of average equivalent fracture aperture and permeability with fluid pressure as measured by constant head.

The deeper test interval in hole UH2 extended from 115 to 120.5 ft. The constant-head test was maintained at 900 and 1,000 psi. Figure 15<sup>B</sup> presents the equivalent aperture and average fracture permeability, graphed against absolute pressure in the test interval. Although only two pressures were maintained during this test, there does appear to be an increase in equivalent aperture that ranges from less than 0.0023 to over 0.0025 in

at 900 psi and from over 0.0029 to just under 0.0031 in at 1,000 psi. Some of these separations overlap those determined in the 63.0- to 68.5-ft interval at lower pressures. Permeabilities in the deeper test section of hole UH2 ranged from about 280 to over 500 D. At lower pressures in the shallower test section of UH2, the determined permeabilities ranged from about 395 to 435 D.

DISCUSSION

Figure 6 presents geologic data for each of the vertical holes. The horizontal distance between hole collars is not given, but depth is shown to scale as distance in feet below the Upper Void Level mine floor. The desorption sample number in the leftmost column. The next column gives lithologic information about the core. The relative lithological abundance is given by the number or density of symbols indicative of the appropriate constituent. The next column is a histogram showing oil yields as determined by modified Fischer assay by Dickinson Laboratories, Inc. The assay sample sizes were generally 2 ft or less. The measured assays are given in figure 16. A high degree of lateral continuity in grade is apparent. These data were normalized to match the desorption sample interval by a weighted average. Sample mass was determined by multiplying the volume of the oil shale sample by its

specific gravity. Specific gravities were calculated using Smith's empirical relationship between oil shale density and oil yield (15).

The next column to the right on figure 6 is another histogram showing the 40-day index of methane content per unit mass as measured by desorption in a closed container. The column on the right gives the location and quantity of fractures in the core. Distinctions are made between fractures and bedding plane separations. Also, different types of core breaks are accounted for. Where fractures occur too close together to map accurately, a symbol (shown in the key) gives information about fracture density.

Figure 6 allows the qualitative evaluation of interdependence between several factors. In general, gas flows emanated from fractures, vugs, fracture planes coated with kaolinite, and core loss zones. The relationship between gas

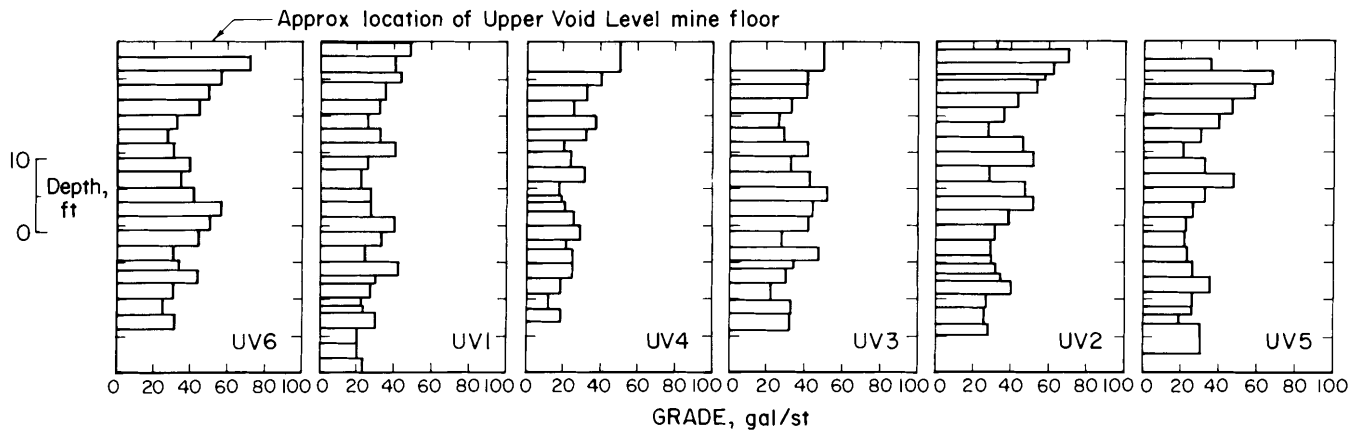


FIGURE 16.—Actual measured assay values for vertical holes before samples were weight-averaged to match the desorption sample interval.



flows during drilling operations and gas enrichment measured by MDM gas desorption testing was tentatively upheld. It may be that methane flows are better correlated with the form or structure of the methane-emitting zone than with its desorption gas content or chemical composition. This is a somewhat appealing premise because methane that readily flows from a drill hole is probably mobile and exists in a free gas state or dissolved in water before penetration by drilling operations. It would not be necessary for this gas to remain near its source. However, reservoirs that produce gas appear to have been very limited in volume as they bled off within the duration of a single shift. Although the permeability data represent only an initial step toward understanding the reservoir characteristics of oil shale ore bodies, they do indicate high permeability, hundreds of darcies, within the fracture network of gas-producing zones. Although data from this study on the permeability of unfractured oil shale are not yet available, previous workers have found the unfractured rock to have a permeability on the order of microdarcies.

It has been postulated that an association might exist between the amount of methane enrichment and the oil yield richness of oil shale from the same sample upon retorting. Predictions of oil shale specific gravity based on oil yield imply a relationship between assays and the actual amount of organic matter contained in a sample. The histograms in figure 6 do not qualitatively support an association between methane content and oil yield. A quantitative approach was attempted by graphically representing methane desorbed per unit mass in terms of oil yields, in figures 17A and 17B. An association is not supported by either of these plots. Similar graphs were prepared for other gases with similar results. Nitrogen was quite unreactive. CO<sub>2</sub> generally increased slightly in volume but was not highly reactive, and the calculated quantities of CO<sub>2</sub> are less reliable with increasing test time as additive experimental errors increase.

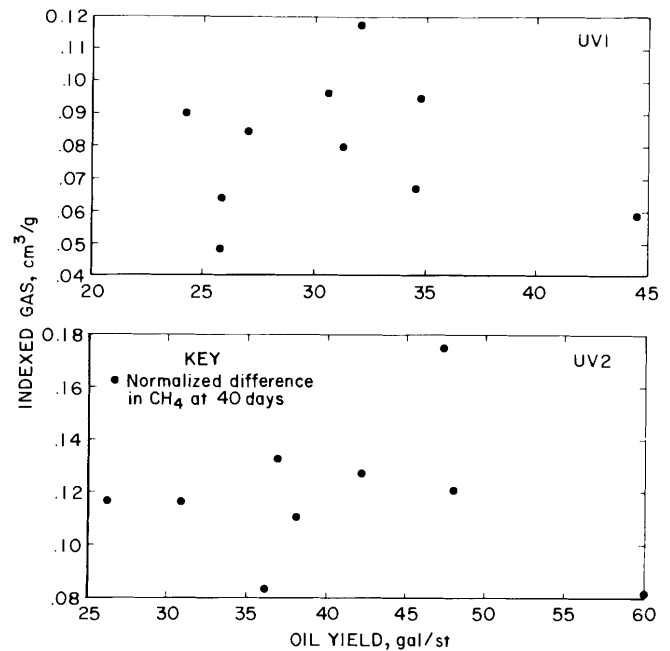


FIGURE 17.—Correlation between methane content at 40 days of desorption and oil yield for holes UV1 and UV2.

Oxygen can also be problematic in terms of additive experimental error because its volume decreases over time. Graphs of oxygen per unit mass against oil yield did not show them to be correlative.

The parameter shown in figure 6 that directly and most consistently varies with methane content appears to be the occurrence of asphalts or bitumen and sometimes pyrite. Asphalts in the context of this study refer to streaks or small, rounded inclusions of bituminous material found in the core. The fine-grained, dispersed nature of this material suggests that the organic materials in the oil shale have been the source of the bitumen formation. The exact mechanisms for this formation are not totally understood; however, associations between bitumen and oil shale are common in the Uinta Basin.

Three primary factors are recognized as influencing the type or chemical composition of bitumen produced in environments of oil shale genesis. The first factor is suggested by petrologic investigations of coal. It has been shown that

the specific botanical composition of coal progenitors is an important factor in the physical and chemical makeup of the coal formed. The progenitors of Green River oil shale and rare algal coals are similar, so it is anticipated that both oil shale and coal share this primary control. Oil shale does contain far more mineral matter than conventional coal. The other recognized primary factors of bitumen formation are related to the chemical environment of the ancient lake of oil shale deposition. It has been inferred that the lake waters varied chemically through time, and differing bitumens are preferred on the basis of the salinity of the environment where they form (16). The third primary control of bitumen formation is the availability or depletion of oxygen during deposition and burial of the bitumens' precursors. The degree of reduction in the lake waters favors the chemistry of certain bitumens (16).

A secondary control might result from differences in the chemical and physical stabilities of the bitumens and/or their precursors. These stabilities could determine the presence or absence of the substances now found in the rock. The pyrite sometimes associated with these bitumens probably resulted from reduction of sulfate to sulfide, which combined with iron from mineral matter, similar to pyrite formation in coal-forming swamps.

Figure 18 shows the frequency distributions of methane contents indexed to 72 h of desorptions for the total sample population (n) in this study. The mean (X) of the samples is  $0.0316 \text{ cm}^3/\text{g}$ , with a standard deviation (S) of  $0.0169 \text{ cm}^3/\text{g}$ . Experimental malfunctions reduced the total sample population from 134 to 119 at 72 h and to 100 at 40 days. The bimodal distribution of the vertical holes has not been accounted for. The slightly higher degree of methane enrichment of the horizontal hole samples might be attributable to shorter lost-gas times or to gas enrichment of the horizon(s) drilled by the horizontal holes as opposed to the diversity of oil shale beds

and methane contents penetrated by the vertical holes. These postulates are not substantiated.

Figure 19 presents methane contents of the sample population on a 40-day time index. The mean of the group is  $0.114 \text{ cm}^3/\text{g}$ , with a standard deviation of  $0.0505 \text{ cm}^3/\text{g}$ . A bimodal distribution for the vertical hole samples and a slightly higher degree of methane enrichment for horizontally derived samples is apparent. Figure 20 shows the 50 horizontal hole samples indexed to 125 days of methane desorption. Similar to data shown in the previous two figures, the overall distribution is skewed to the left, toward the lower end of the scale. The data of figure 20 have a mean of  $0.195 \text{ cm}^3/\text{g}$  and a standard deviation of  $0.071 \text{ cm}^3/\text{g}$ .

The composition of gas released from oil shale samples can be estimated from this study. The gases routinely analyzed by gas chromatography included Ar,  $\text{N}_2$ ,  $\text{O}_2$ ,  $\text{CO}_2$ ,  $\text{CH}_4$  and, less frequently, CO and  $\text{C}_2\text{H}_6$ . Of the seven oil shale samples discussed in the section "Results of Modified Direct Method Testing," five produced smooth and regular gas evolution and/or reaction curves indicative of reliable experimental execution.  $\text{CH}_4$ ,  $\text{CO}_2$ , and  $\text{C}_2\text{H}_6$  were the only gases showing predictable increases in volume. Nitrogen showed an increase in some instances and a decrease in others. Nitrogen volume did not increase in a regular trend between sample points. Overall changes generally accounted for less than 10 pct of the  $\text{N}_2$  starting volume. When  $\text{CH}_4$ ,  $\text{CO}_2$ , and  $\text{C}_2\text{H}_6$  were normalized to 100 pct, the five samples averaged 91.56 pct  $\text{CH}_4$ , 8.43 pct  $\text{CO}_2$ , and 0.01 pct  $\text{C}_2\text{H}_6$ .

Comparisons can be made between the gas contents of oil shale samples and the gas contents of a variety of other rock samples. The methane content means and ranges of all oil shale samples are given for the time indices of 3, 40, and 125 days in figure 21. The units of gas content are cubic centimeters per gram. All three time indices for oil shale samples overlap with high-volatile bituminous coal (e.g., Pittsburgh Coalbed).

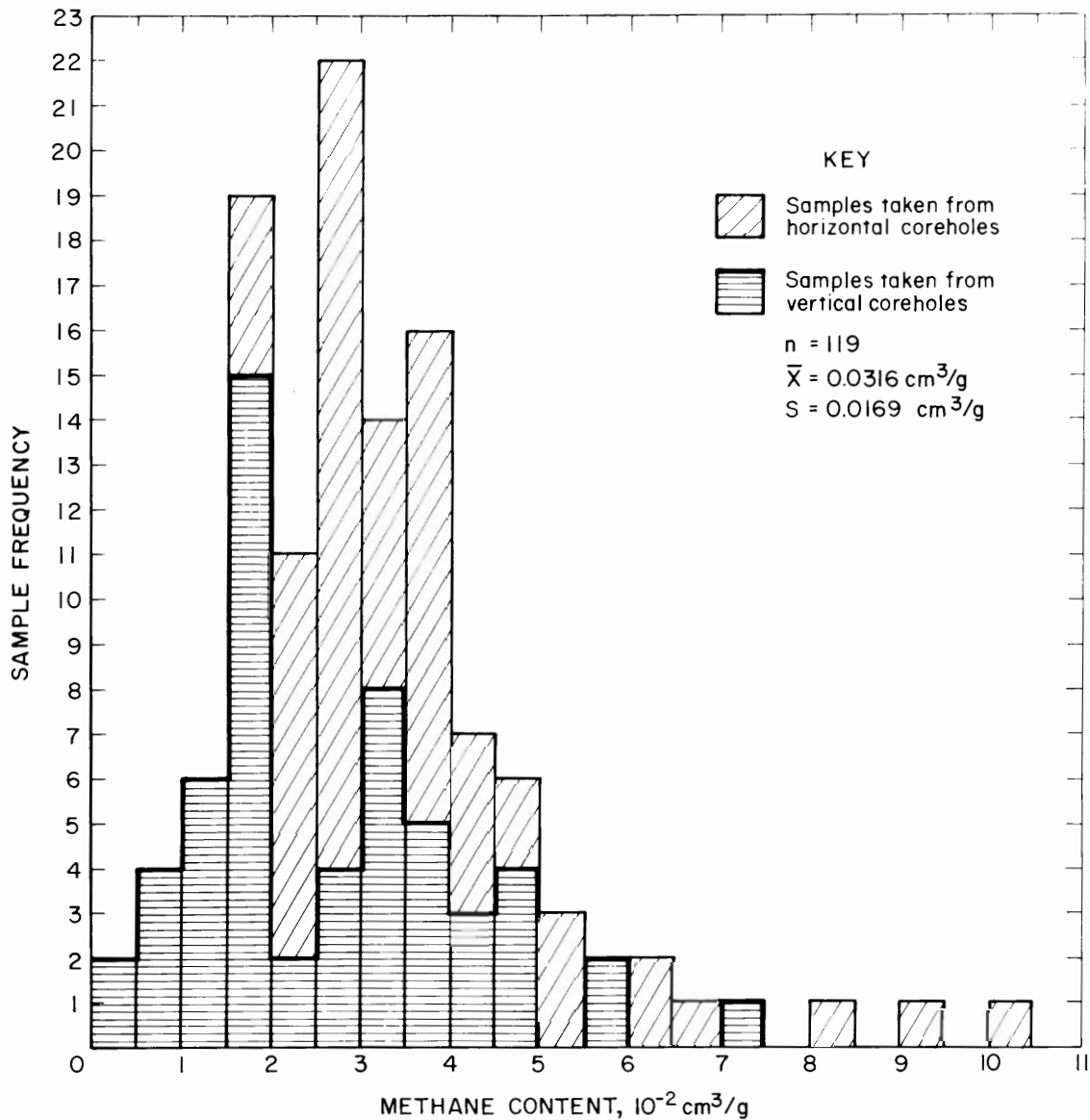


FIGURE 18.—Methane contents of samples at 3 days of desorption showing vertical corehole sample distribution and total sample distribution.

However, the logarithmic horizontal scale shows the mean of high-volatile bituminous coal to be an order of magnitude higher than the mean gas content of oil shales reported for 40- and 125-day time indices and two orders of magnitude higher than the mean gas content of oil shale samples at the 3-day time index. Gas contents of Cathedral Bluffs oil shale samples at the 3-day time index are quite similar to outburst-type domal salt in both mean and range. This does not indicate outbursting tendencies in oil shale. It does show a resemblance between the

volume of gas released from the gassiest domal salt type categorized by the Bureau and the amount released in the shortest time duration of oil shale gas-content testing reported here. There is overlap shown between the ranges of the 3- and 40-day time indices of oil shale gas contents and the ranges of the other two salt types.

It is not possible to account for the differing gas-content test methods for salt, coal, and oil shale. The MDM technique used on the oil shale samples

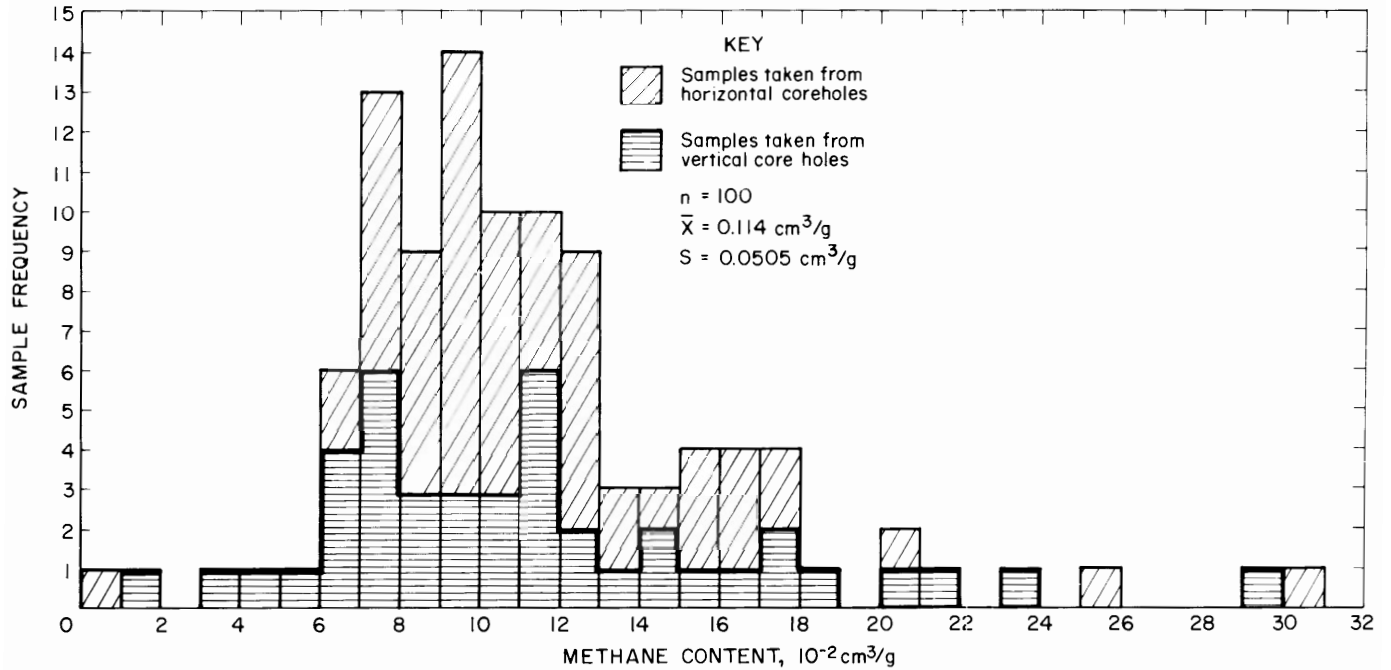


FIGURE 19.—Methane contents of samples at 40 days of desorption showing vertical corehole sample distribution and total sample distribution.

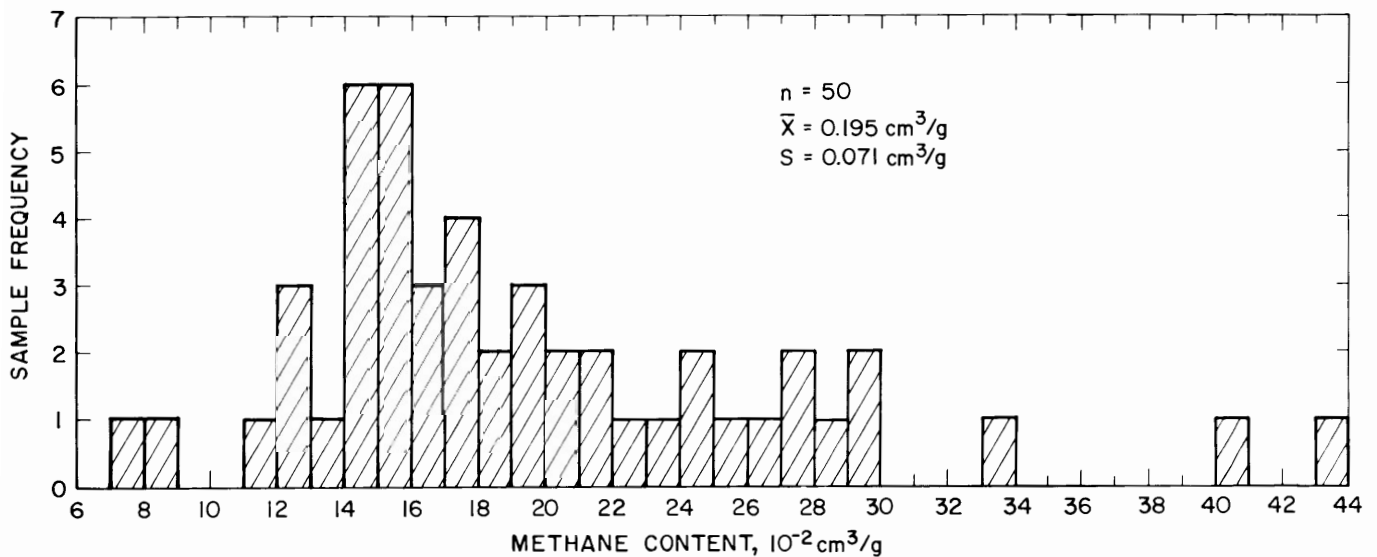
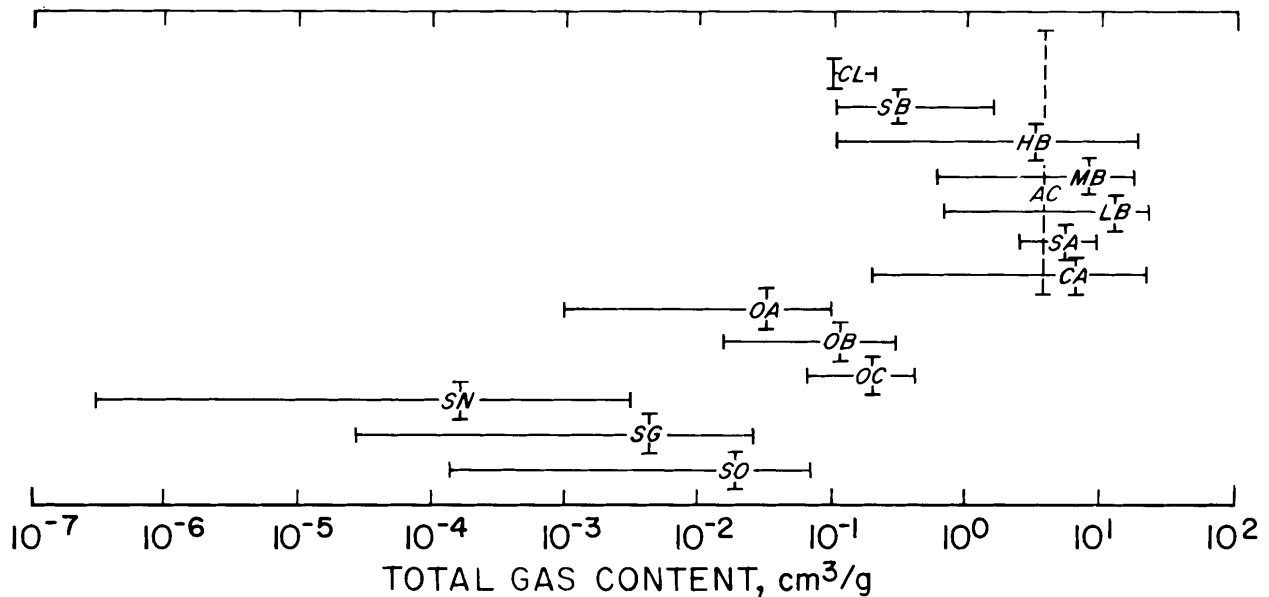


FIGURE 20.—Methane contents of samples at 125 days of desorption showing horizontal hole sample distribution.

involves no destructive sample treatment and measures gas released from the sample under essentially atmospheric conditions after the rock is removed from in situ conditions. Dissolution gas-content testing for evaporites measures gas released only by destructive sample

treatment. The direct method data reported for coal samples in figure 21 are obtained generally by a few months of desorption testing until the process has greatly declined, after which the coal samples are crushed and the gas released is measured and added to the total.



## KEY

U.S. coal

- CL Lignite
- SB Subbituminous
- HB High-volatile bituminous
- MB Medium-volatile bituminous
- LB Low-volatile bituminous
- SA Semianthracite
- CA Anthracite
- AC Average

Green River Formation oil shale, Piceance Basin

- OA 3 days of desorption testing
- OB 40 days of desorption testing
- OC 125 days of desorption testing

U.S. domal salt

- SN Gulf Coast domal rock salt, normal
- SG Gulf Coast domal rock salt, anomalous, impure, gassy
- SO Outburst salt

FIGURE 21.—Mean and range of methane contents released from assorted rock samples.

## CONCLUSIONS

The occurrence of methane when mining oil shale is supportable theoretically and empirically. However, there is not a great deal of agreement on the amount of methane released upon mining a given quantity of oil shale. Data from the Horse Draw Mine probably represent some of the best information available on this subject; the average arrived at was 42 ft<sup>3</sup>/st (1.3 cm<sup>3</sup>/g). In using these data, it is useful to consider that the Horse Draw Mine was designed to recover dawsonite and nahcolite in addition to oil shale. This required mine workings to be developed below the dissolution surface, where the saline minerals are preserved. These horizons are below many oil shale

beds usually of major interest to the oil shale mining industry, including the Mahogany Zone. The geologic character of the oil shale is very different above the dissolution surface in the Piceance Basin, where vuggy porosity and water movement through fractures are relatively commonplace. The stratigraphic differences between the Cathedral Bluffs and Horse Draw Mines probably inhibits the sharing of methane data between the two sites.

A number of geologic factors have been suggested as being important in the control of methane enrichment in oil shale. This report has attempted to address some of them. There is no evidence for a

significant correlation between oil yield by modified Fischer assay and methane content. The existence of certain local structural features such as fractures and vugs may provide channels for gas movement in oil shale. These features are sometimes associated with methane enrichment in oil shale but appear to be more significant in controlling the movement of free gas in the ore body. The feature having the most persistent direct relationship with gas enrichment was the occurrence of bitumen and possibly pyrite in the rock. The bitumen occurred as streaks and small rounded inclusions of solid material in the rock matrix.

The amount of methane contained in fresh oil shale samples was measured by a new technique known as the modified direct method (MDM). The method is described in detail in the appendix. This test measures the STP volume of gas released from a rock sample that is removed from in situ conditions to essentially atmospheric conditions without crushing or similar destructive action on the sample. Quantifying STP gas volumes each time a sample is taken during the duration of the test produces information on rates of gas desorption and gas reactions. It has been shown that methane is released relatively rapidly when a sample is first removed from the in situ environment, but this rate quickly declines, probably in less than 24 h. It is recommended that in using the test, (1) if the objective is to measure total gas content, infrequent gas sampling be done, with samples taken most frequently at the beginning of the test and (2) if rates of gas releases or reactions are the objective, then samples be taken much more often; the methane desorption rate can be expected to change rapidly immediately following removal of the sample from the host rock.

This study represents a detailed drilling program successfully completed from underground mine workings. A surface-set diamond drill bit with a semiround crown and internal discharge was the best choice for core drilling in the Piceance Basin, in order to maintain water pressure in penetrating vugs filled with

tuffaceous or argillaceous material. Use of a split inner barrel is suggested. A drill with independently variable thrust and rotational controls imparts maximum flexibility to the driller for contending with inhomogeneities varying from comparatively hard and somewhat elastic oil shale to fractures, and to vugs or fractures filled with loose or weakly lithified material. A drill unit powered by 25 hp or more is appropriate for all but very short hole drilling.

Permeability data presented here should be considered preliminary. The most reliable data generated during this study suggests that very high permeabilities can exist in the fracture network, especially in zones that produced gas flows during drilling. The permeabilities given here ranged from about 300 to 500 D. Evidence also suggests that gas reservoirs as they exist in oil shale formations are very limited in volume, perhaps because of the narrow apertures of the fracture system.

Methane contents per unit mass were calculated from MDM for three time intervals of gas desorption. Methane contents were calculated at 72 h to simulate a typical amount of time muckpiles would remain underground. The mean value of 119 samples was  $0.0316 \text{ cm}^3/\text{g}$ . Methane contents were calculated at 40 days to get higher gas values without excluding samples removed from drill holes completed near the termination of the study. This population numbered 100 oil shale samples, and the mean gas content was  $0.114 \text{ cm}^3/\text{g}$ . At 125 days, the longest time for which gas contents were indexed, a mean of  $0.195 \text{ cm}^3/\text{g}$  was determined for 50 samples. All sample populations deviated somewhat from normal distributions, with the majority of samples being skewed slightly toward the low end of the scale and a few samples forming a tail in the more gas-enriched direction.

Comparisons can be made between the gas contents of oil shale samples and other rock samples. Rock sample testing has determined that methane released from Mahogany Zone oil shale from the Piceance Basin is far less than that evolved from U.S. high-volatile bituminous coal. Test

results also indicate that oil shale mining may produce slightly more methane than is generally released in domal salt mines. However, dissimilarities in gas-content testing methodology are highly significant to these comparisons. Coal and salt gas determinations attempt to measure the total gas contained. MDM testing for oil shale samples measures

gas released without destructive sample treatment. One can only speculate on how much gas remains in the oil shale even after 125 days of gas desorption, as reported here. Under no circumstances would it be advisable to apply data reported here to oil shale occurring in other basins or within differing stratigraphic units.

#### REFERENCES

1. Robinson, W. E. Origin and Characteristics of Green River Oil Shale. Ch. in *Oil Shale*, ed. by T. F. Yen and G. V. Chilingarian. Elsevier, 1976, pp. 61-79.
2. Russell, P. L. History of Western Oil Shale. Cent. Prof. Adv., East Brunswick, NJ, 1980, 152 pp.
3. Cole, R. D., G. J. Daub, and B. E. Weichman. Geology of the Horse Draw Naphcolite and Oil-Shale Mine, Piceance Creek Basin, Colorado. Paper in 15th Oil Shale Symposium Proceedings (Golden, CO, Apr. 28-30, 1982). CO Sch. Mines, Aug. 1982, pp. 15-28.
4. Sapko, M. J., J. K. Richmond, and J. P. McDonnell. Continuous Monitoring of Methane in a Deep Oil Shale Mine. Paper in 15th Oil Shale Symposium Proceedings (Golden, CO, Apr. 28-30, 1982). CO Sch. Mines, Aug. 1982, pp. 320-340.
5. Teichmuller, M. Origin of the Petrographic Constituents of Coal. Sec. in *Coal Petrology*. (Engl. transl.). Gebruder Borntraeger, West Berlin and Stuttgart, 3d ed., 1982, pp. 219-294.
6. Hartley, J. A., and T. N. Beard. A Standard Technique for Handling, Marking, and Logging Oil Shale Core. Paper in 16th Oil Shale Symposium Proceedings (Golden, CO, Apr. 13-15, 1983). CO Sch. Mines, Aug. 1983, pp. 81-98.
7. Kissell, F. N., C. M. McCulloch, and C. H. Elder. The Direct Method of Determining Methane Content of Coalbeds for Ventilation Design. BuMines RI 7767, 1973, 17 pp.
8. Murray, D. K., and J. D. Haun. Introduction to the Geology of the Piceance Creek Basin and Vicinity, Northwestern Colorado. Ch. in *Guidebook to the Energy Resources of the Piceance Creek Basin Colorado*, ed. by D. K. Murray. Rocky Mt. Assoc. Geol., 1974, pp. 29-40.
9. Yen, T. F., and G. V. Chilingarian. Introduction to Oil Shales. Ch. in *Oil Shale*, ed. by T. F. Yen and G. V. Chilingarian. Elsevier, 1976, pp. 1-12.
10. Eugster, H. P. Origin and Deposition of Trona. *Contrib. Geol.: Trona Issue*, v. 10, No. 1, 1971, pp. 49-56.
11. Stellavato, N. Results of the Geologic Mapping Program During Shaft Sinking and Subsequent Station Development at C-B Tract. Paper in 15th Oil Shale Symposium Proceedings (Golden, CO, Apr. 28-30, 1982). CO Sch. Mines, Aug. 1982, pp. 115-136.
12. Kuchta, J. M., M. Hertzberg, R. Cato, C. D. Litton, D. Burgess, and R. W. Van Dolah. Criteria of Incipient Combustion in Coal Mines. Paper in 15th Symposium (International) on Combustion (Tokyo, Japan, Aug. 25-31, 1974). Combustion Inst., Pittsburgh, PA, 1974, pp. 127-136.
13. Bennett, R. D., and R. F. Anderson. New Pressure Test for Determining Coefficient of Permeability of Rock Masses. U.S. Army Eng. Waterways Exp. Stn., Tech. Rep. GL-82-3, July 1982, 41 pp.
14. Bredehoeft, J. D., R. G. Wolff, W. S. Keys, and E. Shuter. Hydraulic Fracturing To Determine the Regional In Situ Stress Field, Piceance Basin, CO. *Geol. Soc. America Bull.*, v. 87, No. 2, 1976, pp. 250-258.
15. Smith, J. W. Theoretical Relationship Between Density and Oil Yield for Oil Shales. BuMines RI 7248, 1969, 14 pp.

16. Bell, K. G., and J. M. Hunt. Native Bitumens Associated With Oil Shales. Int. Ser. Monogr. Earth Sci., v. 16, 1963, pp. 333-366.
17. Darton, N. H. Occurrence of Explosive Gases in Coal Mines. BuMines B 72, 1915, 248 pp.
18. Bertrard, C., B. Bruyet, and J. Gunther. Determination of Desorbable Gas Concentration of Coal (Direct Method). Int. J. Rock Mech. and Min. Sci., v. 7, 1970, pp. 43-65.
19. Cheng, K. C., K. K. Feng, and R. Augsten. Test Procedure for Methane Desorption From Coal By Direct Method. Can. Explos. Res. Lab., CANMET Rep. ERP/MRL 81-63 (TR), Aug. 1981, 18 pp.
20. McCulloch, C. M., J. R. Levine, F. N. Kissell, and M. Deul. Measuring the Methane Content of Bituminous Coalbeds. BuMines RI 8043, 1975, 22 pp.
21. Curl, S. J. Methane Prediction in Coal Mines. IEA Coal Res., London, 1978, 77 pp.



## APPENDIX.--MODIFIED DIRECT METHOD PROCEDURE

Several procedures have been used by the Bureau to study the occurrence of gas in coal and oil shale. A method reported by Darton in 1915, essentially examined the gas composition of the atmosphere of a sealed can or bottle containing lump or crushed coal at various points in time (17).<sup>1</sup> The original atmosphere of the container was either ambient atmosphere or a vacuum. The method reported by Darton measured only gas released upon crushing the coal sample. Oxygen sorption and/or reaction by coal was also examined and in some cases was considerable.

A frequently used procedure for measuring the amounts of gases released from coal uses displaced water volume and yields the algebraic gas volume of a sealed container with a rock sample. This technique is known as the direct method and is described in references 7, 18, and 19. The gas volume can be composed of one or more gases that have either desorbed and thus contributed "positive" volumes or sorbed and/or reacted, contributing "negative" volumes. Essential, but generally not practiced, is the sampling of the container atmosphere so the relative proportions of the individual gas species can be examined. A direct method type of test without gas composition information yields an approximation of the gas contents and desorption rates of the rock sample, which can be erroneously small because of the negative volume effects of sorption and/or reaction of gases with the sample in the sealed container. Standard temperature and pressure (STP) corrections to these desorbed gas volumes are applied in general but not always.

The method described in this report, MDM, uses the ideal gas law equation to determine the STP gas volume in the sealed sample container and a gas sample of the container atmosphere to compute the volume of each gas species under

consideration at any point in time. The main advantages of the MDM are (1) desorption or release and/or reaction rates of individual gas species can be accurately determined and (2) the determination of these rates can be performed on samples of smaller size or lower gas contents than in previous direct method practices. These advantages allow a more accurate and flexible characterization of multiple gas source variables where the direct method yields information on the changing overall volume of the total gases contained. This method can be performed in laboratory, field, and underground settings.

PROCEDURE, CALCULATIONS,  
AND EXPERIMENTAL APPARATUSProcedure

The main purpose of the MDM is to obtain gas volumes over a period of time to characterize gas desorption and reaction rates. Gas should be sampled more frequently when a rock sample is first sealed into the container and less frequently at later times. Coal samples are usually sampled six to eight times within the first 2 h, a week later, and once a month for each of the next 2 to 6 months.

The buildup of pressure in the sealed sample container should be minimized so that the desorption process is not inhibited. However, taking samples of small volume changes can introduce potentially large cumulative errors. By controlling pressure and atmosphere composition in the sealed container, desorption, sorption, and reaction phenomena can also be studied at different conditions.

Basic Calculation

The essence of this procedure is the calculation of gas composition and volume (corrected to STP conditions) in the sealed sample container at a given point in time. This quantity of gas is compared with the container atmosphere at a prior point in time, and the gas species volume differences are accumulated.

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<sup>1</sup>Underlined numbers in parentheses refer to items in the list of references preceding the appendix.

Using the ideal gas law, the volume at STP conditions of gas species  $x$  in a container at time interval  $n$  ( $V_{x,n}$ ) is determined by

$$V_{x,n} = \left\{ \frac{[(P_{atm} + P_{vent,n} + dP_n) V_g]}{(T_{g,n}) (P)} \right\} (T) ([x]_n), \quad (A-1)$$

where  $P_{atm}$  = atmosphere pressure, mm Hg;

$P_{vent}$  = underground mine ventilation pressure differential over  $P_{atm}$  from surface if samples are sealed underground, mm Hg;

$dP$  = differential pressure of container atmosphere with respect to  $(P_{atm} + P_{vent})$ , mm Hg;

$T_g$  = temperature of gas atmosphere in container, K;

$V_g$  = free space for gas atmosphere to occupy in container,  $\text{cm}^3$ ;

$T$  = standard temperature (273 K);

$P$  = standard pressure (760 mm Hg);

and  $[x]$  = volume fraction of gas species or group  $x$  in container atmosphere,  $\text{cm}^3/\text{cm}^3$ .

#### Experimental Apparatus

A typical configuration of the apparatus is presented in figure A-1. The experimental apparatus used in demonstrating this method was estimated to perform within the following limits:

1. Barometer:  $\pm 1$  to 2 mm Hg.
2. Thermometer:  $\pm 0.5$  to  $1.0^\circ$  C.
3. Differential pressure gauge:  $\pm 1$  to 2 pct full scale.
4. Evacuated container and gas sample port for gas sample or detector giving

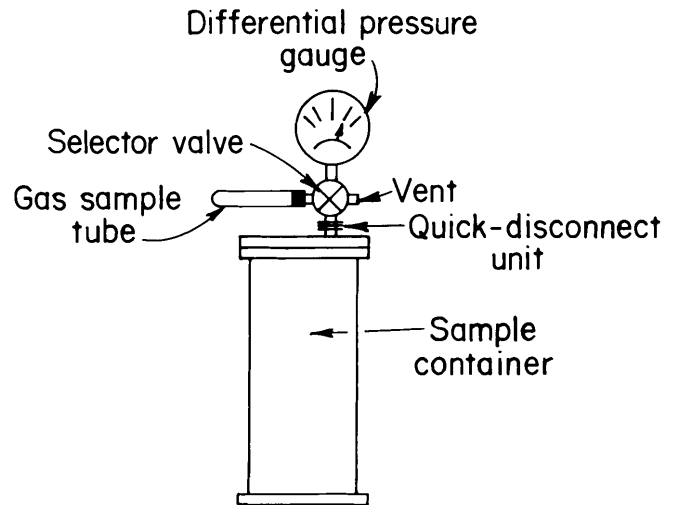


FIGURE A-1.—Generalized apparatus.

compositional analysis:  $\pm 1$  to 2 pct of value.

5. Sample container with at least one inlet and valve with volume measured:  $\pm 1$  to 2 pct of total volume (determined by weight of water required to fill container with and without sample to obtain sample and free-space volumes).

The Bureau's experience with gas sampling of rock samples has found the design of experimental apparatus to be of paramount importance in obtaining accurate results. Hematological test tubes and needles have proven very reliable as containers for sampled gas and for transference of released gas. A diaphragm-type pressure gauge is used to measure partial pressures in the oil shale sample containers.

Magnahelic pressure gauges are available in a variety of ranges and are built to be accurate to within 2 pct of the full scale measured. Sample containers for the oil shale core were constructed of 2.5-in-diameter schedule 40 PVC pipe. It is desirable for the diameter of the container to be somewhat larger, in this case by slightly more than 0.5 in, than the core diameter, to facilitate insertion and removal from the container. Schedule 80 PVC caps were tapped and fitted with quick disconnects for closing the open end of the container underground. The opposing ends had been

closed by the gluing of schedule 40 caps at the Bureau. Hansen, Swagelock, and Milton-type quick disconnects were used on the core containers.

#### BASIC DATA COLLECTION CYCLE

To obtain a container atmosphere data point at a given time:

1. Record date and time of data point determination.
2. Measure atmosphere pressure ( $P_{atm}$ ) and temperature of the gas in the container ( $T_g$ ), which can be considered to be ambient if the storage area for containers does not undergo large and/or rapid temperature changes.
3. Measure barometric pressure. If the test site is located underground (e.g., coal mine) and only the surface atmospheric pressure is known, then the ventilation pressure differential ( $P_{vent}$ ) with respect to surface must be measured.
4. Connect a differential pressure gauge to the sample container, and measure an initial differential pressure with respect to ambient pressure ( $dP_i$ ).
5. Purge the sample circuit with container gas if  $dP_i \geq 0$  or with an inert gas if  $dP_i < 0$ , then take a gas sample of the container atmosphere. This gas sample is for gas composition analysis.
6. If assurance is required that an appropriate volume of gas from the container was obtained, take an intermediate differential pressure measurement ( $dP_{inter}$ ) after the gas sample, and approximate the volume ( $V_{sample}$ ):

$$V_{sample} = [(dP_i - dP_{inter})(V_g)] / (P_{atm})$$

7. After gas sampling, if  $dP_{inter}$  is--
  - a. Positive, then bleed excess differential pressure ( $dP_f$ ) to atmospheric pressure and measure. This terminates measurements at this data point.
  - b. Zero or negative, terminate data point measurement;  $dP_f = dP_{inter}$ . If any gases are injected or air leaks into the container, another gas composition sample must be taken and a final  $dP_f$  taken to terminate the measurements.

A sample data collection form is presented as figure A-2.

#### Data Reduction

The first step in the data reduction process is to determine the atmosphere composition when the sample is first sealed. The ambient atmosphere is sampled for composition, and the temperature and pressure are measured. The free space in the container with the rock sample is known, and the STP-corrected volumes of the gases sealed in the container with the sample are calculated by equation A-1 and represent the initial conditions.

Gas samples of the container atmosphere, as well as measurements of the corresponding pressures and temperature, are taken periodically, and the gas species STP volumes are calculated by equation A-1 for each sampling period. In each sampling period, measurements are taken both before and after release of excess gas pressure. The current initial gas volumes in the container are compared with the final gas volumes from the previous sampling period: The individual gas volumes before any release of excess gas pressure are subtracted from those calculated at the end of the previous sampling period after release of excess pressure. These differences represent the changes in gas species volumes that have taken place since the end of the previous sampling period. The volume changes are applied to the cumulative gas volumes that began with the initial conditions when the container was sealed. The cumulative gas species volumes can then be presented graphically as a function of time. The STP gas volumes should be normalized to either a unit mass (cubic centimeters per gram) or volume of rock (cubic centimeters per cubic centimeter) basis.

The MDM testing discussed in this report was performed underground at the Cathedral Bluffs Mine, where the test environment was at a nearly constant temperature over the duration of measurements. However, core removed from surface drilling in a separate study was

Mine or property: \_\_\_\_\_ Corehole: \_\_\_\_\_ Sample No.: \_\_\_\_\_  
 Lithology: \_\_\_\_\_ (24 chars. entered in DB)  
 Core interval: from \_\_\_\_\_ ft to \_\_\_\_\_ ft Sample length: \_\_\_\_\_ ft Core diameter: \_\_\_\_\_ in  
 Date-time core: begin \_\_\_\_\_ end \_\_\_\_\_ contained \_\_\_\_\_  
 Container: \_\_\_\_\_ Container volume: \_\_\_\_\_ cm<sup>3</sup> Lost time: \_\_\_\_\_ h

Date - dd-mm-yr	Time hh:mm	Cum. time, h	Patm, mm Hg	Pvent, mm Hg	dPi in H <sub>2</sub> O	Gas n	dPinter in H <sub>2</sub> O	dPf in H <sub>2</sub> O	Temp, °C	Comments (24 chars.)
:	:									
:	:									
:	:									
:	:									
:	:									
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Sample volume: \_\_\_\_\_ cm<sup>3</sup> Sample wt.: \_\_\_\_\_ g Nominal free space: \_\_\_\_\_ cm<sup>3</sup>  
 True free space: \_\_\_\_\_ cm<sup>3</sup>

FIGURE A-2.—Sample data-collection form.

tested and stored at a surface site, and results were not totally satisfactory because of the differing rates at which the gas, core, and container equilibrate with changing air temperatures. A constant-temperature environment is recommended.

Unaccounted for with the MDM are the gases lost between the time the rock sample is taken and when it is sealed in the container. Essential to determining the lost gas is determining the length of time that gas has been desorbing from the sample before it is sealed in the container. Previous researchers have calculated the lost-gas time for a vertical drill hole filled with water or drilling fluid by assuming that desorption begins when the sample has traveled half the distance out of the drill hole (20). In using this technique, the lost gas can be determined graphically by plotting the cumulative gas volumes on a graph with the square root transformation applied to the time axis and drawing a linear extrapolation to "zero" time.

Lost gas was not calculated for data presented in this report, but it is

advisable that a lost-gas technique be applied to MDM experiments in any study where the rock sample is exposed to atmospheric conditions for more than about 1 h before being sealed in a container. The necessary data are available to perform a lost-gas correction on the Cathedral Bluffs results, although some question does remain concerning the initiation of lost-gas conditions in this study.

The one component of a rock's gas content not yet addressed is the gas remaining in the sample after the monitoring of gas activity in the sealed container has been terminated. To determine this, some form of crushing of the sample can be performed in a sealed container with an inert (nitrogen) atmosphere (7, 21). The released gas volumes are determined with the above method. Since it takes relatively great lengths to liberate this remaining gas from the rock, it seems probable that this component of the total gas content of the rock is not significant in mine emission or gas drainage studies.