

APPLICATION OF THE ROCK-EVAL III OIL SHOW ANALYZER TO THE STUDY  
OF GASEOUS HYDROCARBONS IN AN OKLAHOMA GAS WELL

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# APPLICATION OF THE ROCK-EVAL III OIL SHOW ANALYZER TO THE STUDY OF GASEOUS HYDROCARBONS IN AN OKLAHOMA GAS WELL

## Introduction

Pyrolysis has been employed as a tool for characterizing potential petroleum source rocks as early as 1915 by White(1), in the 1960's by Hunt, Juntgen and Van Heek, and Bajor, and in the 1970's by LePlat and Noel, Giraud, Gransch and Eisma, Bordenave, Barker, Claypool and Reed, and Espitalie among a few (2,3,4,5,6,7,8,9,10,11).

Development of commercial pyrolysis instrumentation, such as the Rock-Eval I, began in the early 1970's and allowed consistent analysis of rock samples for source rock characterization (11). Additional refinements such as microprocessor control of operation and automatic sampling were incorporated into second generation Rock-Eval II instruments (12). Both commercialization and automation have made pyrolysis a routine tool in the expedient characterization of whole rock samples.

Clementz et al. (13) reported the difficulty of assessing kerogen quality when total organic carbon (T.O.C.) data were unavailable during a wellsite evaluation of pyrolysis data. Computing hydrogen and oxygen indices and plotting a modified van Krevelen kerogen typing diagram for kerogen characterization cannot be accomplished without T.O.C. data. However, the new generation of pyrolysis instrumentation, the Rock-Eval III Oil Show Analyzer (4), includes the capability for T.O.C. analysis of whole rock samples and also, the detection of light, gaseous and condensate hydrocarbons (14).

A comparison of Rock-Eval systems is given in Appendix I. Appendix II explains Rock-Eval parameters and Appendix III shows various computations made from Rock-Eval data.

## Background

The newest Rock-Eval instrument provides typical parameters ( $S_1$ ,  $S_2$ ,  $S_3$ , and  $T_{max}$ ) but adds a  $S_0$  peak yield. As such the  $S_1$  peak is thermally separated into two separate peaks. The first peak includes those free hydrocarbons eluted into the detector during a 90°C isotherm for one to five minutes. Depending on sample quality and content this includes  $C_1$ - $C_8$  petroleum compounds. The second peak includes those hydrocarbons being thermally distilled between 90°C and 300°C with a three minute isotherm at 300°C.

The  $S_0$  plus  $S_1$  yield obtained from the Rock-Eval III thermal distillation process compares to the chemical extraction process as shown in Figure 1. Variations in solvent and thermal extraction yields are dependent on sample collection and storage techniques, solvent system, analytical procedures and the sample itself. In general the loss of light end hydrocarbons by chemical extraction (primarily loss due to sample preparation and solvent evaporation) is less in the Rock-Eval technique. However, extraction of high molecular weight hydrocarbons is less complete in the Rock-Eval (15,16) as some petroleum is carried over into the  $S_2$  peak yield. Despite these differences both results are indicative of the concentration of petroleum in the sediment sample and may be used to predict hydrocarbon nature.

Determination of the nature of these free hydrocarbons, that is, whether they are indigenous or migrated, may be accomplished by comparison to the organic carbon content of the same sample. Utilization of extractable hydrocarbons to T.O.C. ratios as an indication of migrated hydrocarbons was reported by Baker (17). Anomalous ratios, i.e., the presence of substantial quantities of extractable hydrocarbons per weight percent T.O.C., is indicative of migrated hydrocarbons. Similarly, comparison of  $S_0$  and  $S_1$  yields to T.O.C. may allow discernment of migrated hydrocarbons when viewed with the well profile or field data. Care must be exercised that contamination of samples from drilling operations has not occurred.

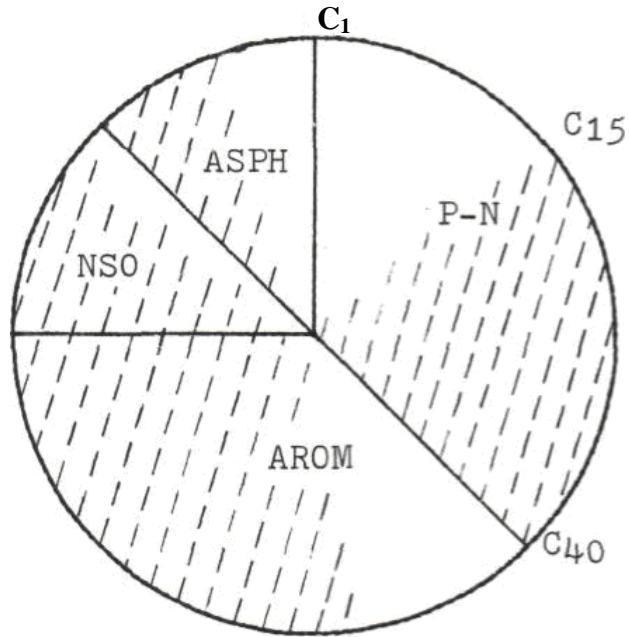
The Rock-Eval III T.O.C. determination allows the analysis of whole rock samples without prior acid treatment to remove inorganic carbon. The T.O.C. process consists of two steps: (a) thermal distillation and pyrolysis and (b) oxidation.

In the pyrolysis process each whole rock sample is first thermally distilled releasing free hydrocarbons followed by pyrolysis (cracking) of kerogen (and, if present, thermal extraction of high molecular weight hydrocarbons). The total hydrocarbon yield from these steps is normalized to carbon content (pyrolyzed carbon, PC) with units adjusted to weight percent carbon, i.e.,

$$\text{PC or } \frac{k(S_0+S_1+S_2)}{10} \quad (S_0, S_1, S_2 \text{ in mg hydrocarbon per gram of whole rock})$$

The value of the constant, k, is 82%, which is the average percent of carbon from thermally extracted and pyrolyzed hydrocarbons (18).

**(a) Solvent Extraction Yield**  
(dashed lines indicate approx. yield)



**(b) Thermal Extraction Yield (S0 + S1)**  
(dashed lines indicate approx. yield)

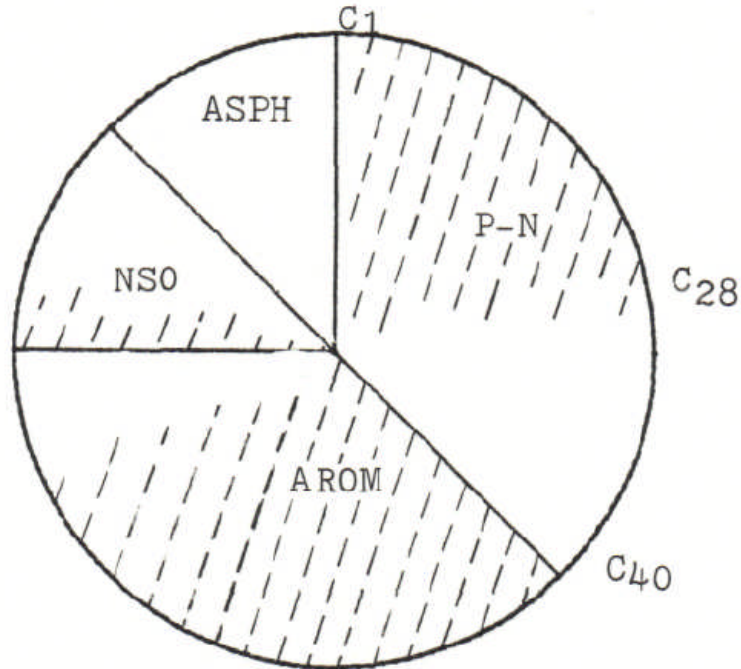
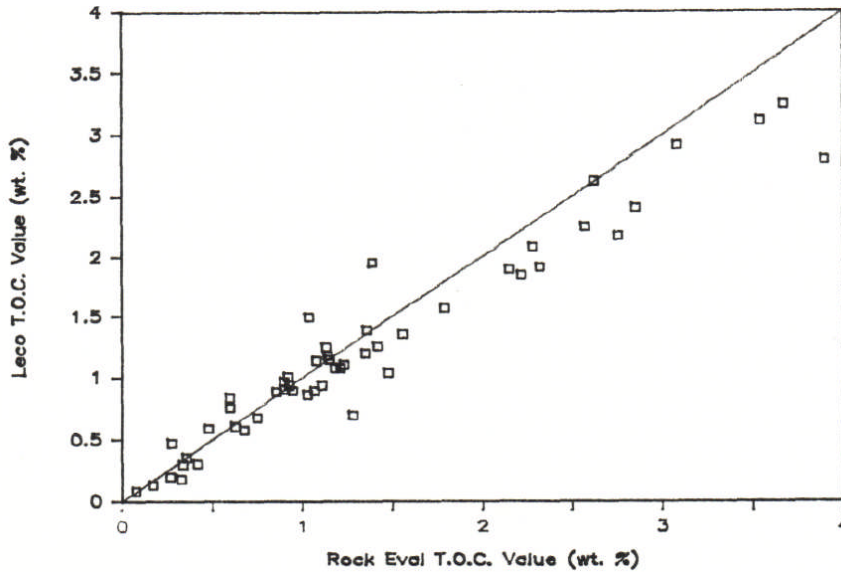


Figure 1

Diagrammatic sketch of extraction yield vs. Rock-Eval thermal extraction yield (S0+S1).  
Illustrated extract composition is P-N (37.5%), AROM (37.5%), NSO (12.5%), and  
ASPH (12.5%)

**(a) Comparison of T.O.C. Data**  
(T.O.C. Values 0-4 %)



**(b) Comparison of T.O.C. Data**  
(T.O.C. values 4-80 %)

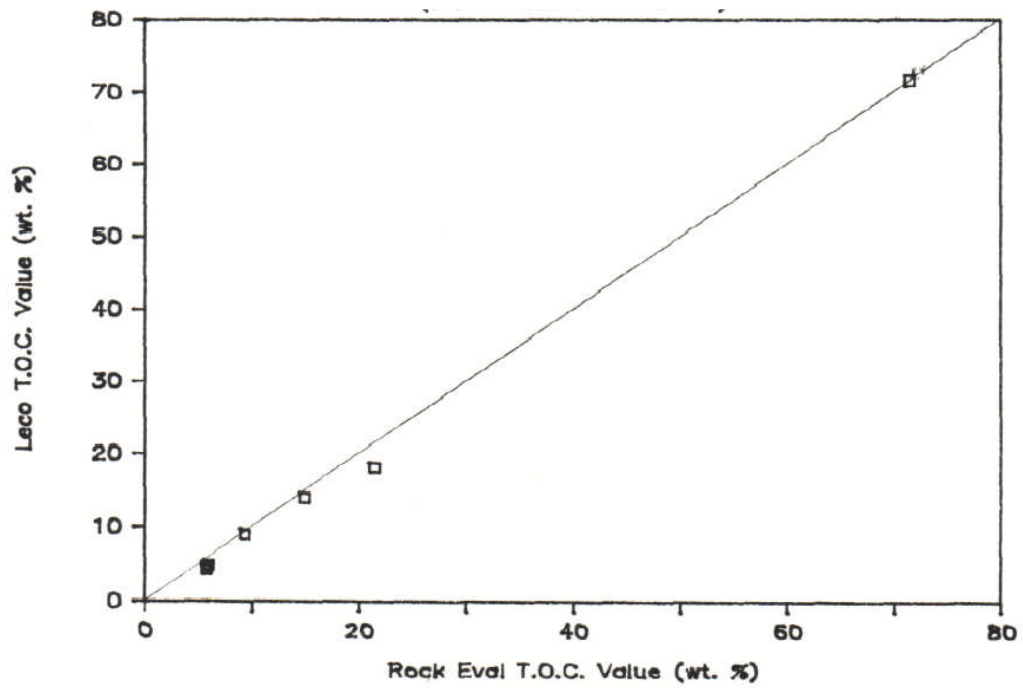


Figure 2

The sample is automatically transferred to a second oven where the second process of T.O.C. determination occurs, the oxidation of any carbon remaining after pyrolysis, which forms carbon monoxide and carbon dioxide. Carbon monoxide is catalytically converted to carbon dioxide for trapping and detection by a thermal conductivity detector. The oxidation oven temperature is kept just below 600°C. The portion of carbon oxidized to carbon dioxide in the second oven constitutes the residual carbon, RC, adjusted also to weight percent carbon, i.e.,

$$\frac{RC}{10} \text{ or } \frac{S_4}{10} \text{ (S}_4 \text{ in mg carbon/g whole rock)}$$

The computation of T.O.C. is then

$$\text{T.O.C. (wt. \%)} = \text{PC} + \frac{RC}{10} \qquad \frac{0.82(S_0+S_1+S_2) + S_4}{10}$$

Carbonate disassociation into inorganic carbon dioxide and its mineral oxide does not interfere in the oxidation process since it has been disassociated in the pyrolysis process. The maximum temperature for Rock-Eval pyrolysis is 600°C while the oxidation oven is operated just below the 600°C threshold, thereby avoiding any interference in organic carbon due to additional inorganic carbon dioxide generation.

Numerous independent T.O.C. correlation studies have been completed. Comparison of Rock-Eval T.O.C. data to data from the standard Leco combustion method by the Institut Francais du Petrole (IFP) had a correlation coefficient of 0.99 (least squares method).

In our applications laboratory fifty nine samples were analyzed using the Rock-Eval T.O.C. system. The results were compared to T.O.C. values obtained from over ten separate laboratories each having their “own” standard method of handling samples for organic carbon analysis. These samples were of various lithologies including several very high percentage carbonate rocks. The data are plotted in figure 2 from the data in Table I. Overall, the correlation coefficient was also 0.99 (least squares method).

The Rock-Eval T.O.C. formula does not include organic carbon dioxide (S3) released during the pyrolysis process (18). Carbon from organic carbon dioxide release usually amounts to less than five percent of the T.O.C. even in oxidized sediments. The Rock-Eval III Oil Show Analyzer does not trap and detect the S3 peak; however, in the Rock-Eval II plus

T.O.C. system, S<sub>3</sub> may be included in the T.O.C. computation by separately adding the S<sub>3</sub> contribution normalized for carbon content and adjusted to weight percent carbon, i.e.,

$$\frac{0.27 (S_3)}{10} \quad (S_3 \text{ in mg CO}_2/\text{g of rock})$$

### Kerogen Type and Thermal Maturity Plot

To determine organic-matter type from Rock-Eval III data, a plot of the Hydrogen Index versus T max is required. This type of kerogen characterization plot takes into account both capability to generate hydrocarbons and maturity (18,19).

### Gas Well Study

#### Operator and Location

Sandfer Oil & Gas Company's Cruson No. 1-13 in Custer County, Oklahoma, Anadarko Basin, was logged between 8000 and 15150 feet. Cuttings were typically analyzed at thirty to sixty foot intervals using a Rock-Eval III.

#### Sample Preparation: Wellsite

Cuttings were collected at the shaker and washed through a ten mesh sieve. The cuttings were then sealed under water in plastizer-free plastic bags to minimize the loss of light gases by evaporation and to prevent any residual drilling fluids from drying onto the cuttings.

#### Sample Preparation: Laboratory

When received in the laboratory individual samples were washed with water until clear water elution was seen. The cuttings were dried at room temperature for approximately fifteen minutes. The picked, unpulverized cuttings were weighed (approximately 100 mg) and analysis initiated.

Espitalie et al. reported the effects of variations in drying time on S<sub>0</sub> yield (18). Selected samples containing S<sub>0</sub> range hydrocarbons were dried for 15, 60 and 120 minutes. At both 60 and 120 minutes complete loss of S<sub>0</sub> hydrocarbons was observed. Crushing and grinding of selected, preserved cuttings also caused complete loss of S<sub>0</sub> hydrocarbons. No experimentation was attempted on shorter drying times.

### Geochemical Log and Data Tables

Using the data generated from the Rock-Eval, a geochemical profile of the well was prepared as shown in figure 3 from the well data in Table II. The capability to determine T.O.C. allows computation of the Hydrogen Index as well as S0 to T.O.C. and S1 to T.O.C. ratios.

### S0 Analysis

Hydrocarbons evolved during the 90oC isotherm were detected initially at 9540 feet and S0 detection was made in various sections through total depth. A maximum was detected at 10070 feet in a porous sandstone section, but no S0 hydrocarbons were detected in the gas show range of the Pennsylvanian Middle Red Fork Sandstone.

The source of these light hydrocarbons was presumed to be from either hydrocarbons trapped in interstitial pore spaces or adsorbed hydrocarbons from indigenous generation or migration.

Comparison of S0 values to the mud gas log were inconsistent perhaps due to sampling techniques especially sample storage and transit time to the applications laboratory. In any case the correlation between S0 and mud gas was not seen as reported elsewhere (19). However, one section of the well, from 13210 to 13420 feet, yielded both S0 hydrocarbons and mud gas indication of light hydrocarbons. This section was below the gas find at 13000 feet.

Study of S0 to T.O.C. and S0 to S1 ratios for the complete well showed no correlation. However, individual sets of data throughout the oil and gas window exhibited high positive correlation of S0 to T.O.C. indicating that these light hydrocarbons are indigenous.

On the other hand, one set of S0 data from a well section exhibited no correlation with T.O.C. These S0 hydrocarbons were considered to be migrated hydrocarbons.

Table III shows sections where there was S0 detection but no mud gas detection. S0 to T.O.C. and S1 to T.O.C. correlations were computed in an attempt to show their indigenous or migrated nature. Note that the number of data sets is small in these intervals.

	S0: T.O.C.	S1:T.O.C.
(1) 9890 – 10310	0.846 (indigenous)	0.955 (indigenous)
(2) 12350 – 12620	0.343 (indeterminate)	0.831 (indigenous)
(3) 14330 – 14540	-0.023 (migrated)	0.793 (indigenous)
(4) 14660 – 14810	0.886 (indigenous)	0.889 (indigenous)

Table III



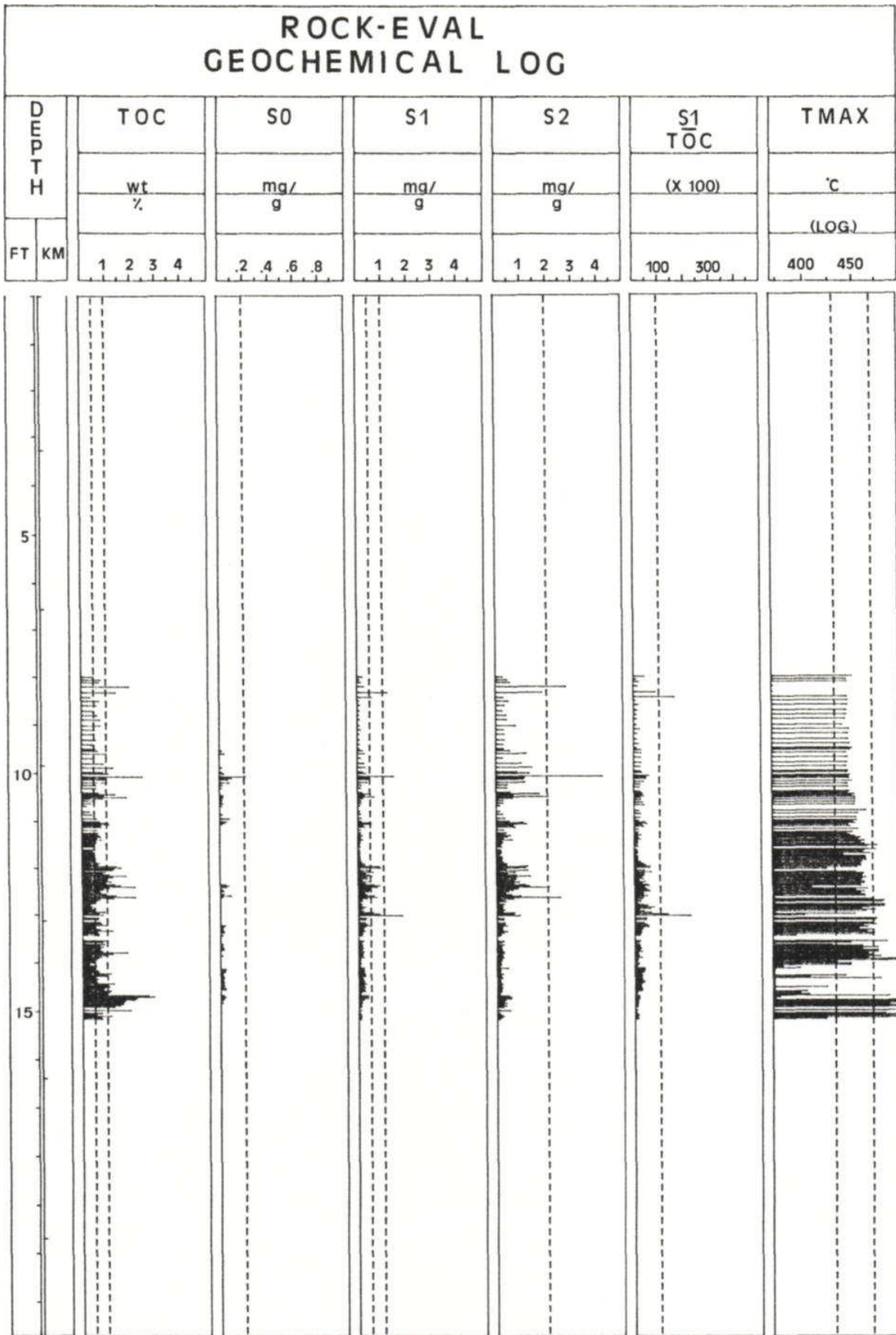


Figure 3 (a)

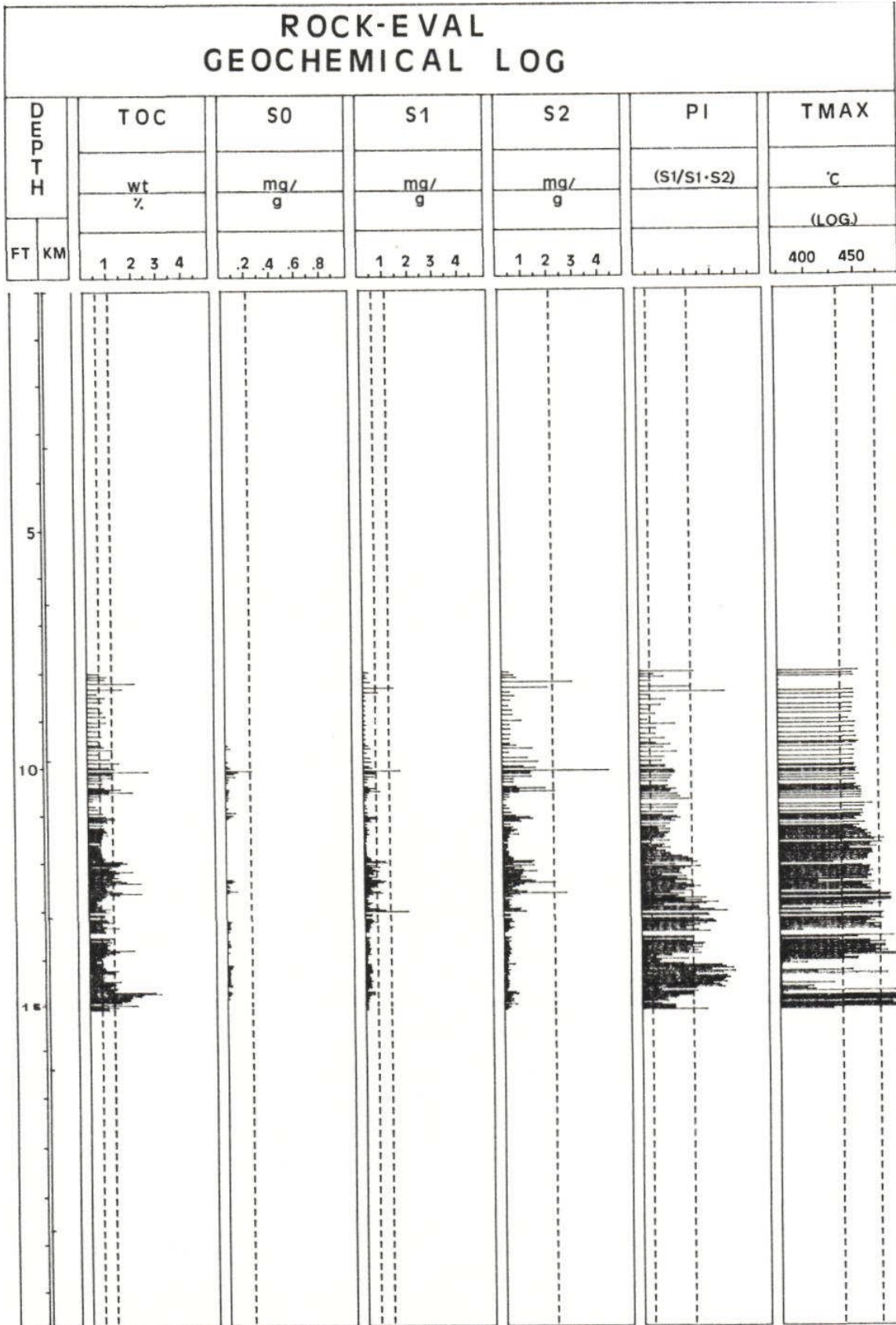


Figure 3 (b)

In very mature sections determination of hydrocarbon nature becomes more difficult. Cracking of bitumen itself may result in anomalous S0 to T.O.C. ratios. Therefore, correlation of S0 to S1 and S0 plus S1 to T.O.C. ratios may help elucidate the nature of the S0 hydrocarbons. Table IV shows the same four sections shown in Table III with the aforementioned correlations.

		S0: S1	S0+S1: T.O.C.
(1)	9890 – 10310	0.054 (indigenous)	0.945 (indigenous)
(2)	12350 – 12620	0.614 (indigenous)	0.806 (indigenous)
(3)	14330 – 14540	0.047 (migrated)	0.753 (indigenous)
(4)	14660 – 14810	0.919 (indigenous)	0.895 (indigenous)

Table IV

This data appear to confirm our suspicion that the 14330-14540 section S0 hydrocarbons are not related to the indigenous bitumen. However, the S0 hydrocarbons detected in sections (1) and (4) appear to be closely related to the indigenous bitumen, probably resulting from cracking of higher molecular weight hydrocarbons. The weighting of S1 made the S0+S1:T.O.C. ratio uninformative in this particular well.

A section of prime interest was the Middle Red Fork Sandstone reservoir section from 12950 to 13300 feet. The mud gas log revealed high percentages of methane, ethane, propane, and butane but no S0 hydrocarbons were detected. This reservoir section was a porous sandstone and no S0 hydrocarbons were retained on the cuttings analyzed.

However, in this same section S1 values were significant. S1 correlations to T.O.C. were made in this and other organic rich sections. The results are summarized in Table V with S1 versus T.O.C. correlations shown.

		S1: T.O.C.
(1)	9800 – 10520	0.934 (indigenous)
(2)	11870 – 12650	0.678 (indigenous)
(3)	12940 – 13000	-0.095 (migrated)

Table V

Sections 1 and 2 most likely contain hydrocarbons resulting from indigenous generation while section 3 appears to be indicative of migrated hydrocarbons.

The average value of the S1 to T.O.C. ratio for the complete well was 0.25. At the top of the Middle Red Fork Sandstone (12970 feet) the ratio was 1.23; at the 13000 foot reservoir level the ratio was 2.14 – the highest S1 to T.O.C. value in the well.

Study of S1 to T.O.C. ratios in other sections of the well revealed one additional section of interest that was otherwise undetected. At 8430 feet, near the top of the Douglas Sandstone, the S1 to T.O.C. ratio was 1.59 – the second highest value in the well. Future wells will be tested in this interval.

#### Gas Zone Source Rock Identification

In the immature and oil window sections of a well, both S2 and T.O.C. are indicative of the potential of a petroleum source rock to generate hydrocarbons. However, in the zone of gas formation conventional pyrolysis parameters offer few insights into the nature of the originally deposited organic matter. The metamorphic processes that have occurred cause near complete dehydrogenation of the organic matter and rearrangement into stable pre-graphitic structures. Pyrolysis yields are very low due to these thermal effects.

In cases of thoroughly cooked organic matter, only the residual carbon analysis is indicative of the original quality of the sediment as a petroleum source rock (20). For example, in the Cruson well studied, the transition zone between the oil and gas window is approximately 12650 to 13670 feet. The mean S2 value in this transition zone is 0.24. In the gas window (>13670 feet) the mean S2 value is 0.18. In the same sections the mean T.O.C. values are 0.56 and 1.01 respectively, with the T.O.C. resulting primarily from the oxidation of carbon remaining after pyrolysis. During catagenesis these may have been good oil or gas source rocks. Upstructure locations may still be in the oil window and could be sites for future exploration.

#### Thermal Maturity

Using T max values to study maturity, the oil and gas zones as well as immature zones may be outlined. Drilling to predetermined depths or formations may not result in the complete penetration of the oil window. Utilizing T max as a guide systematically through the depth of a well will allow identification of the top and bottom of the oil window.

Anomalies in T max may assist in the interpretation of pyrolysis data by directing attention to the S2 pyrogram (flame ionization detector trace). Oil accumulations usually cause deflection of T max values from the oil window range (435 – 465) to the 300 to 400 degree range. This is commonly the result of free, high molecular weight hydrocarbons cooking off in the S2 peak. This was noted in the Cruson

Well where oil window T max values dropped to 398 degrees at the 13000 foot reservoir section. Contamination by mud additives could cause similar effect. On the other hand, reworked organic matter may cause T max values to be greatly inflated. For example, T max values in the 530 to 600°C range may be recorded. When S2 values are extremely low, i.e. less than 0.20, T max values are not reliable. Other anomalies in T max values may be caused by mixed kerogens, extreme variation in sample weights, and the rate of temperature programming. Note that in the new generation Rock-Evals, T max values are calibrated against a known standard and are not dependent on sensor positioning on the pyrolysis oven.

## Conclusions

The Rock-Eval T.O.C. system provides an expedient T.O.C. analysis without decarbonation and other chemical or physical preparation of the sample. The technique is reliable and accurate showing excellent correlation to conventional methods. In deep wells where the organic matter has been thoroughly cooked, Rock-Eval T.O.C. data give some insight to the original generation potential of these post mature sediments.

The analysis of S0 hydrocarbons provides information on the presence of light, gaseous and condensate hydrocarbons in non-reservoir well sections. Carefully prescribed and consistent sample handling procedures must be established and maintained for meaningful analysis. Additional study on fresh wellsite cuttings or a laboratory study of canned cuttings is needed. Correlation of S0 analysis to conventional laboratory cutting gas analysis and to Rock-Eval S1 analysis may be appropriate.

Study of S0 to T.O.C. and S1 to T.O.C. ratios is a useful analytical tool in evaluating whether hydrocarbons are indigenous or migrated. It is often possible to ascertain whether detected hydrocarbons are related to indigenous kerogen or bitumen derived or are the result of migration.

T max values must be carefully interpreted in order to accurately predict maturity and kerogen type (in conjunction with HI). Interpretation is enhanced by a review of S2 values and properly attenuated programs.

When carefully reviewed and applied on a local or basin-wide level, the data from the Rock-Eval may assist in the evaluation of source beds and their potential, detect otherwise unrecognized zones of production potential, determine depths for testing operations, define the oil window, and allow correlation of wells for both exploration and production purposes.

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## COMPARISON OF ROCK-EVAL SYSTEMS

	RE I (1977)	RE II (1979)	RE II + TOC (1982)	RE III OSA (1982)
S0	-	-	-	x
S1	x	x	x	x
S2	x	x	x	x
S3	x	x	x	-
T max	x	x	x	x
T.O.C.	-	-	x	x
Automated	-	x	x	x

## DEFINITIONS

S0	Gaseous to condensate range hydrocarbons thermally liberated at a 90 degree C isotherm
S1	Free hydrocarbons (comparable to extractable hydrocarbons) thermally distilled to a 300 degree C isotherm
S2	Hydrocarbons released during the pyrolysis of kerogen between 300 and 550 or 600 degrees C with a linear temperature gradient usually between 25 and 30 degrees C per minute
S3	Organic carbon dioxide released between 300 and 390 degrees C
T max	The temperature at which the maximum rate of yield of S2 hydrocarbons is obtained
T.O.C.	The total organic carbon in weight percent

Appendix I and II

## COMPUTATIONS UTILIZED WITH ROCK-EVAL III OIL SHOW ANALYZER

Gas Production Index (GPI)	$S_0 / (S_0 + S_1 + S_2)$
Oil Production Index (OPI)	$S_1 / (S_0 + S_1 + S_2)$
Total Production Index (TPI)	$(S_0 + S_1) / (S_0 + S_1 + S_2)$
Hydrogen Index (HI)	$S_2 / \text{T.O.C.} \times 100$

## ADDITIONAL COMPUTATIONS UTILIZED BY ROCK-EVAL USERS

Production Index (PI)	$S_1 / (S_1 + S_2)$
Quality Index	$S_2 / S_3$
Oxygen Index (OI)	$S_3 / \text{T.O.C.} \times 100$
Pyrolyzed Carbon (PC)	$0.082 (S_0 + S_1 + S_2)$
Petroleum Potential	$S_1 + S_2$
Migration Index	$S_0 / \text{T.O.C.}$
	$S_1 / \text{T.O.C.}$

COMPARISON OF T.O.C. DATA

ROCK-EVAL T.O.C. DATA	LECO T.O.C. DATA
.42	.31
1.39	1.95
.60	.84
.28	.47
.60	.76
1.03	.87
1.18	1.08
.08	.08
1.21	1.08
.33	.18
3.67	3.25
.27	.20
3.08	2.92
.68	.58
2.15	1.89
71.44	71.65
2.62	2.63
2.85	2.41
5.75	4.34
2.22	1.84
.36	.36
4.01	3.02
1.79	1.57
1.36	1.39
2.32	1.91
2.75	2.17
3.90	2.80
1.11	.94
.75	.68
1.35	1.20
.92	1.01
14.93	14.05
.34	.30
1.42	1.26
1.28	.70
.17	.13
5.79	4.81
2.28	2.08
21.46	18.19
9.36	9.07
3.54	3.12
1.48	1.04
5.96	5.03
1.04	1.50
.90	.97
.48	.59
.86	.89
1.08	1.14
.90	.91
.93	.94
.63	.61
1.14	1.18
1.56	1.36
1.15	1.15
.95	.90
1.07	.90
1.23	1.11

1.13	1.25
2.57	2.25

Table I

# AAPG EXPLORER

## Still Active After 40 Years

