RAPID, ROOM-TEMPERATURE QUANTITATIVE ANALYSIS OF OIL IN SPONGE CORE

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A new procedure (U.S. Patent Pending) for the rapid and quantitative extraction of oil from sponge core has been developed. Oil volumes are determined following extraction by optical spectrometry. Previously described methods for extraction of oil from sponge core rely on Soxhlet extraction or high pressure solvent washing techniques. Our method involves high speed disaggregation of the sponge in a pressure tight disposable container. Typically, oil is quantitatively extracted from a sponge sample at room temperature within 20 minutes. Water removal prior to oil extraction is unnecessary.

Methylene chloride is utilized as the extraction solvent in part because of its efficiency as a solvent for oil and partly because it facilitates separation of the sponge from the aluminum liner. In practice, slight solubilization of the sponge in methylene chloride has not been a problem. Blank corrections for extraction of virgin sponge yield an equivalent extracted oil volume of less than 0.2 ml. A mixture of methylene chloride and other solvents, like toluene, can be used, when required, for complete dissolution of specific oils.

Quantification of oil following solvent extraction is based on a standard optical spectrometric calibration technique utilizing several oil-solvent mixtures of known composition. The advantages of this analytical technique include simplicity, rapidity, wide dynamic range and low cost.

INTRODUCTION

Sponge coring is an innovative and relatively inexpensive technique which was introduced to eliminate errors in conventional routine core analysis oil saturation estimates caused by oil loss during coring and core recovery. The process involves installation of a highly absorptive, oil-wet polyurethane sponge liner in the core barrel. The sponge surrounds a recovered core and absorbs any oil that bleeds or migrates out of the core when pore fluids expand as pressure drops during core recovery. The sponge liner is specifically formulated to immobilize oil and prevent its migration through the sponge. Oil saturation data, based on analysis of recovered core can then be corrected for the volume of oil lost from the core, but retained by the sponge. The validity of the oil saturation correction is strongly dependent on the completeness of the oil extraction step and the accuracy of the subsequent measurement of extracted oil volume.

Published methods for extracting oil from sponge require time consuming Soxhlet extraction or complex high pressure washing techniques. Difoggio, et al (1987) and Dangayach (1988) extract oil from sponge in a Soxhlet extractor using a solvent selected from the group consisting of cycloalkanes, ethers and freons. Subsequent quantification of oil volumes was accomplished utilizing near IR spectroscopy or super-critical fluid chromatography. Subsequently, Difoggio (1988) described a high pressure solvent extraction technique which removes oil from sponge by use of a solvent jet directed against the sponge. The solvent is recycled until complete oil extraction is achieved. Calkin, et al (1988) extracted oil from sponge and then quantified the extracted oil volume utilizing a partial distillation process.

ANALYTICAL REQUIREMENTS

Quantification of oil volumes trapped in a segment of sponge core is a two step process. Successful determination of oil volume requires 1) complete extraction of oil from the sponge and 2) accurate measurement of the extracted oil volume. In our process (U.S. Patent Pending), oil is extracted from the sponge at room temperature using methylene chloride as the solvent. The volume of extracted oil is determined by comparison of the visible light absorption properties (wavelength of 440 nm) of the

extracted oil and solvent with standard samples consisting of known mixtures of oil and solvent. Primary advantages of our analytical technique are low cost, rapidity, accuracy, wide dynamic range and sensitivity. Oil in sponge samples is completely extracted in 20 minutes. The subsequent optical spectrometric quantification of the extracted oil volume requires a few seconds per sample.

THE OIL EXTRACTION PROCESS

A section of sponge core liner and core is shown schematically in Figure 1. The aluminum core liner includes aluminum fingers which prevent movement of the sponge during coring. The liner and core are cut into 6-in long sections. The samples are then cut in half lengthwise (axially) in preparation for analysis. The core halves are removed from the cut liner and subsequently processed in the usual way for measurement of oil and water saturation, porosity, permeability, etc. The two halves of the sponge core liner are processed together to extract and quantify an oil volume which can be precisely correlated with the corresponding routine core analysis-derived oil and water volumes.

Corresponding sponge core liner sections are placed in metal extraction vessels in contact with approximately 1 liter of methylene chloride solvent. The sponge is soaked in the solvent for several hours (24 hours if the sponge core was frozen at the wellsite). The initial solvent soak causes the sponge to swell significantly, thereby, greatly simplifying the subsequent separation and removal of the sponge from the aluminum liner. After separation of the sponge from the liner, the sponge is cut into cubes and left in the original metal extraction vessel. The aluminum liner sections are carefully rinsed with fresh solvent and discarded. The solvent in the extraction vessel is then carefully adjusted to a final volume of 2 liters by addition of fresh solvent.

Oil extraction is accomplished in 20 minutes at room temperature by shredding the sponge cubes in the oil extraction vessels using a high speed rotary cutter. The extraction vessels are sealed by application of metal covers fitted with rotary seals and associated cutters as shown in Figure 2. The rotary cutters are designed to shred the sponge, thereby, completely releasing contained oil to the solvent, and to homogenize the oil-solvent mixture. Experience has shown that a 20 minute cycle satisfies these

objectives.

OIL VOLUME ANALYSIS

Following extraction of oil from a sponge sample, an oil-solvent sample is retrieved from the bottom of the extraction vessel using a syringe fitted with a 0.45 micron membrane filter housed in a syringe filter holder. An aliquot of the oil-solvent mixture is transferred to an optical cell and the optical absorbance of the sample is obtained using a standard visible light optical spectrometer. The measured absorbance of a sample is compared to standardized oil-solvent mixtures to arrive at the final estimate of extracted oil volume.

Derivation of a suitable optical absorbance calibration curve requires a sample of stock tank oil representative of the oil in the cored reservoir intervals. A typical calibration curve is illustrated in Figure 3. The dynamic range of a calibration suite depends to a large degree on the API gravity of the oil. Heavy, darker oils have a narrower calibrated range than lighter oils. It is also prudent to consider dilution of samples which fall near the upper end of a calibration curve to maintain analytical accuracy.

VALIDATION OF THE SPONGE CORE ANALYTICAL PROCEDURE

A suite of 17 water saturated sponge core samples, injected with known oil volumes via syringe, were obtained from major U.S. oil companies. The 6-in long, 4-in diameter samples were subjected to the oil extraction and analytical procedures outlined above. Results of the evaluation are provided in Table 1 and Figure 4. The plotted data were blank corrected for the apparent oil volume produced by slight dissolution of virgin, oil-free sponge in methylene chloride solvent. The equivalent oil volume due to partial dissolution of the sponge material was less than 0.2 ml in all cases.

Ultimate sensitivity of the analytical procedure is strongly dependent on the stability and sensitivity of the optical spectrometer. It was possible to detect oil volumes of 0.04 ml or greater using a Bausch & Lomb Spectronic 88 visible light spectrometer. Modern dual beam digital instruments should be capable of even better resolution. In

comparing known to measured oil volumes, this study found an average error of 5.6% for the extraction-analytical procedure. The greatest relative error was found for samples with the lowest or trace amounts of oil as would be expected.

CONCLUSIONS

A new technique for quantifying the volume of oil extracted from sponge core has been developed. The procedure, which is carried out at room temperature, is fast, economical and accurate. A complete sponge core analysis program can generally be concluded before the corresponding routine core analysis program is completed.

REFERENCES

Calkin, C.L. and Ellington, W.E., 1988, Method for analyzing solvent extracted from sponge core: U.S. Patent 4785661.

Dangayach, K.C.B., Difoggio, R. and Ellington, W.E., 1988, Method for determining the amount of oil in a sponge core: U.S. Patent 4787983.

DiFoggio, R., Ellington, W.E. and Dangayach, K.C.B., 1987, Method for determining the amount of oil in a sponge core: European Patent Application 0227192A2.

DiFoggio, R., 1988, Solvent dispenser for removing oil from sponge core: U.S. Patent 4771634.

FIGURE 1.
SCHEMATIC ILLUSTRATION OF A SPONGE CORE

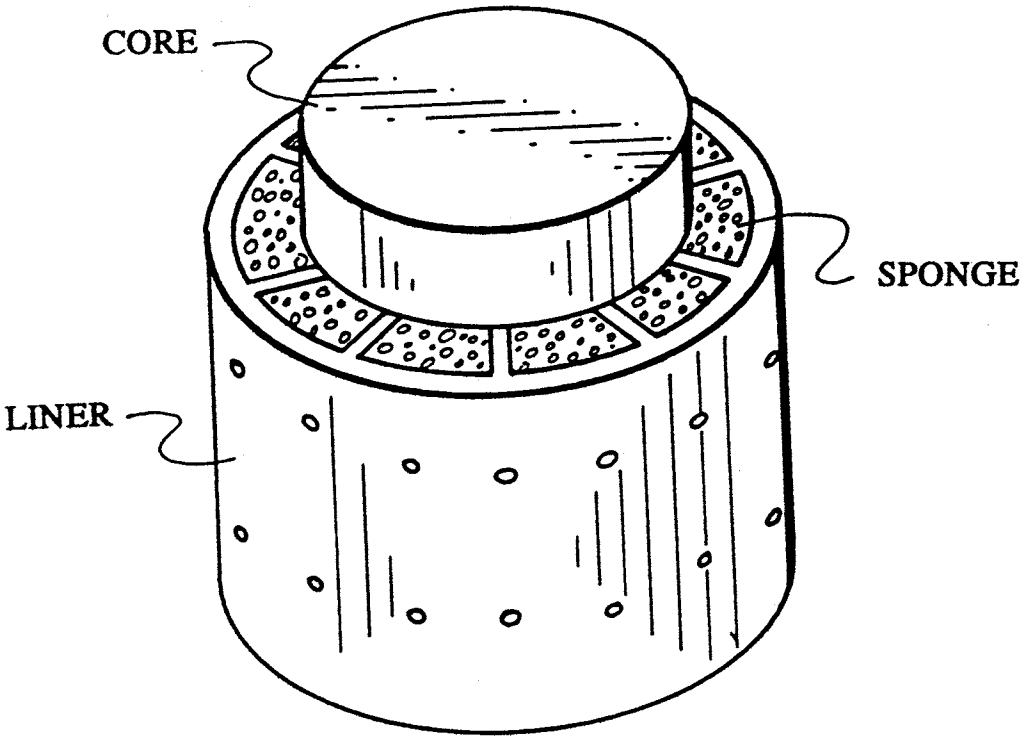

FIGURE 2.
SPONGE CORE OIL EXTRACTION APPARATUS

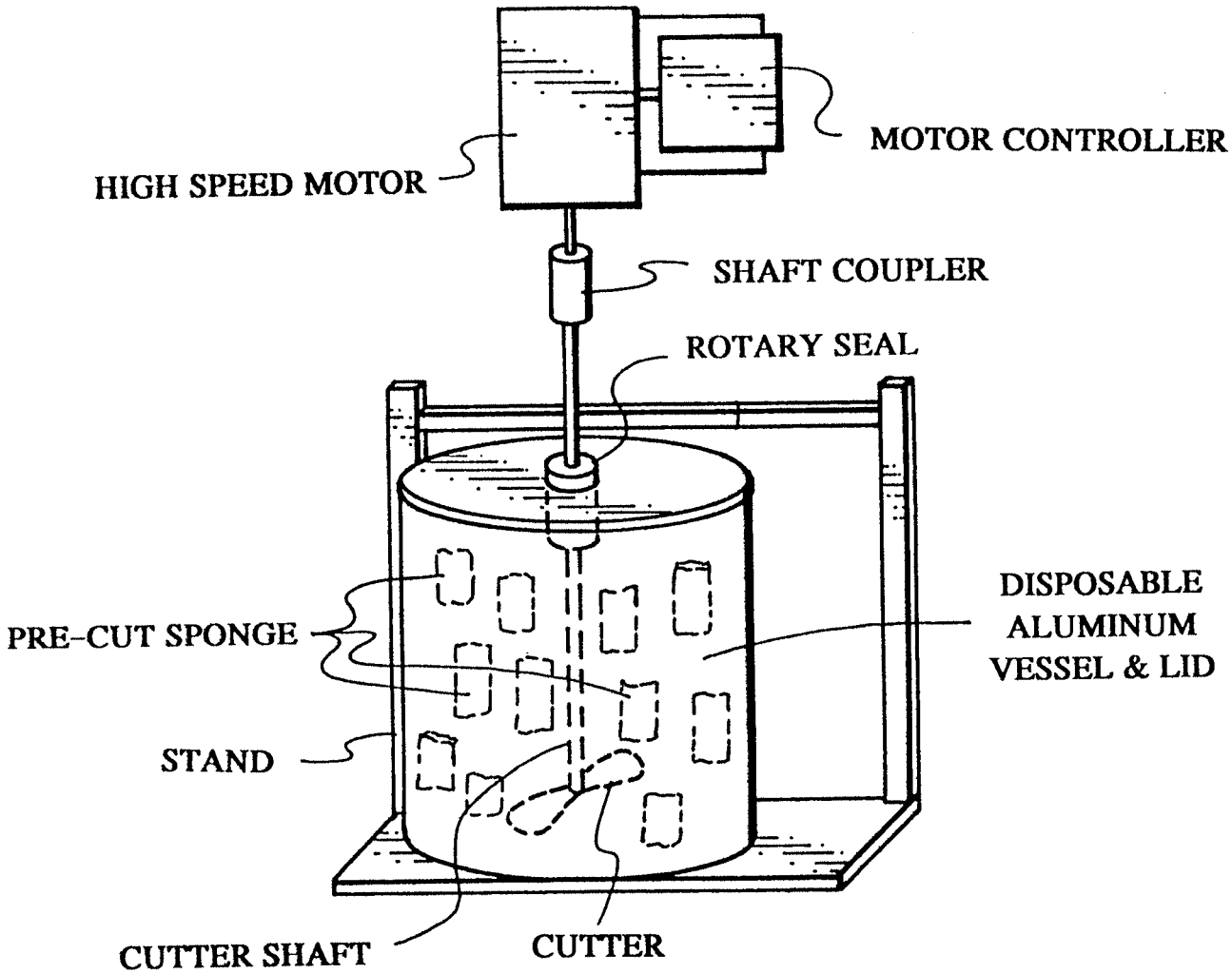

FIGURE 3.
OIL VOLUME -- ABSORBANCE CALIBRATION

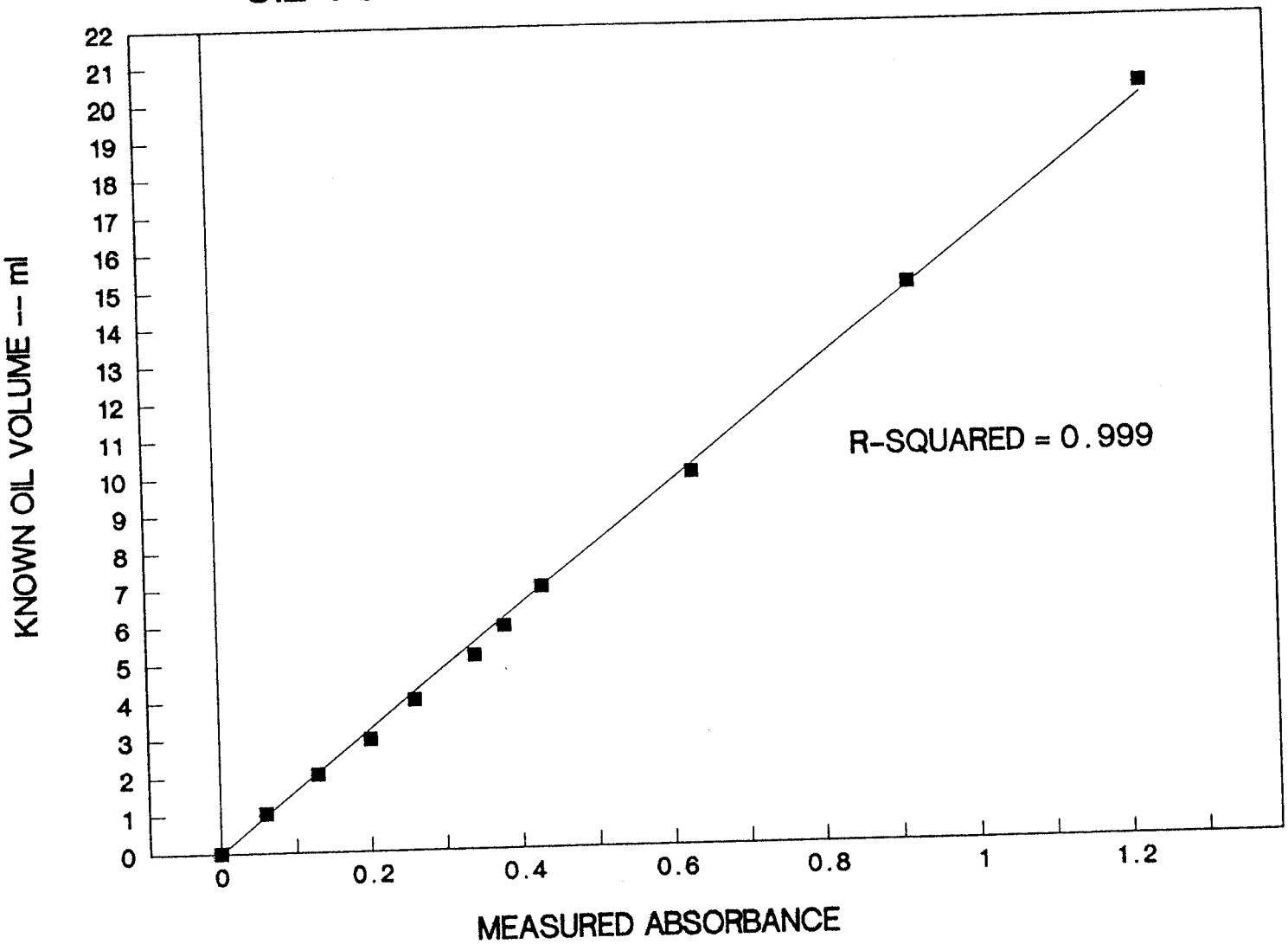

TABLE 1.
SPONGE CORE ANALYTICAL DATA

OIL #1			OIL #2			OIL #3		
KNOWN OIL VOL (ml)	MEASURED OIL VOL (ml)	PERCENT ERROR (%)	KNOWN OIL VOL (ml)	MEASURED OIL VOL (ml)	PERCENT ERROR (%)	KNOWN OIL VOL (ml)	MEASURED OIL VOL (ml)	PERCENT ERROR (%)
2.04	2.07	-1.47	3.01	2.88	4.32	1.62	1.47	9.26
1.95	2.01	-3.08	2.94	2.80	4.76	8.72	9.3	-6.65
3.22	3.29	-2.17	4.29	3.86	10.02	0.48	0.35	27.08
2.99	3.10	-3.68	4.54	4.15	8.59	3.88	3.75	3.35
5.85	5.90	-0.85	7.13	7.07	0.84	16.48	17.16	-4.13
5.98	6.03	-0.84	6.77	6.54	3.40			

AVERAGE ERROR = 5.56 %

FIGURE 4.
SPONGE CORE ANALYTICAL DATA