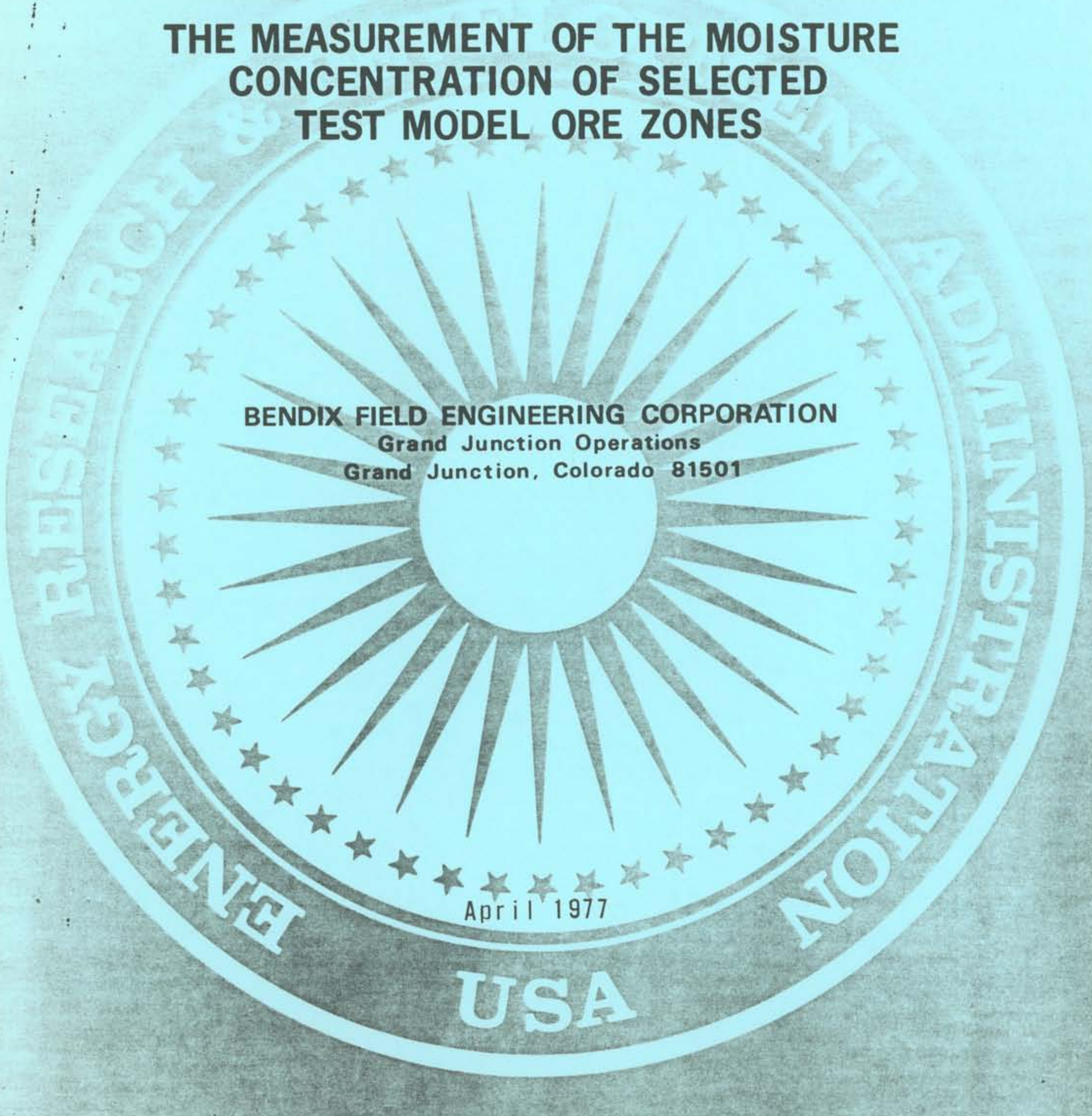
BFEC-1977-3

THE MEASUREMENT OF THE MOISTURE CONCENTRATION OF SELECTED TEST MODEL ORE ZONES



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PREPARED FOR THE U.S. ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION
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Abstract

The three M-barrels (moisture models) and certain uranium test models were logged in August, 1975, with the ^{252}Cf-based neutron-neutron probe. The results, in percent equivalent moisture for the respective test model (in parenthesis), are: 9.4-13.0 %eH_2O(R), 12.1-18.2 %eH_2O(PD), 9.2-14.0 %eH_2O (PW), 10.8 %eH_2O(U1), 14.7 %eH_2O(U2), and 15.1 %eH_2O(U3). A calibration for the ^{252}Cf-based probe was established by logging the M-barrels and using moisture concentration values for these models that were analytically determined in November, 1971. Justification for using old moisture concentration values is presented. The results by chemical analysis of the 27 samples extracted from the M-barrels just prior to logging were judged suspect and therefore not used in establishing the quoted moisture concentrations.

Introduction

Recent on-site tests of a prototype, pulsed 14 MeV neutron logging probe revealed significant differences between measured and quoted moisture concentrations of certain GJO test models. The concern generated by these discrepancies led to the present undertaking.

Initially the planned scope of work was limited to just the R, PD, and PW test models but was expanded opportunistically to include a qualitative assessment of the M-barrels as moisture models and a check of the logging probe calibration determined using the M-barrels. The calibration check was to consist of logging a few additional test models for which moisture concentrations were considered to be well known. Neither the initial nor the added ancillary tasks were planned to be definitive statements on certain parameters of the test boreholes and models: rather, they were undertaken to provide limited interim data. Within this context then, this study was undertaken as a precursor to planned and current studies that promise to be more thorough and comprehensive in scope.

Before continuing and for those unfamiliar with the neutron-neutron probe, the following description of operation may be of some help. The neutron-neutron probe basically measures the hydrogen concentration of the formation. This is so because hydrogen is by far the most effective element in slowing down, by successive collisions (moderation), the fast or energetic source neutrons. Hence it is hydrogen that directly and predominately influences the detected neutron response by scattering the moderated source neutrons back to the detector. If it is assumed that the total pore volume of the formation is saturated with water then the probe's response is taken as a measure of the formation porosity (i.e., the fractional void volume completely saturated with water). In order to minimize variations in response due to lithology, the

ERDA neutron detector (with a 10 inch active length and filled with 20 atmospheres of ^{3}He) is wrapped with cadmium which acts (via neutron capture) to discriminate against the low energy or thermal neutron intensity.

Overview of Procedure

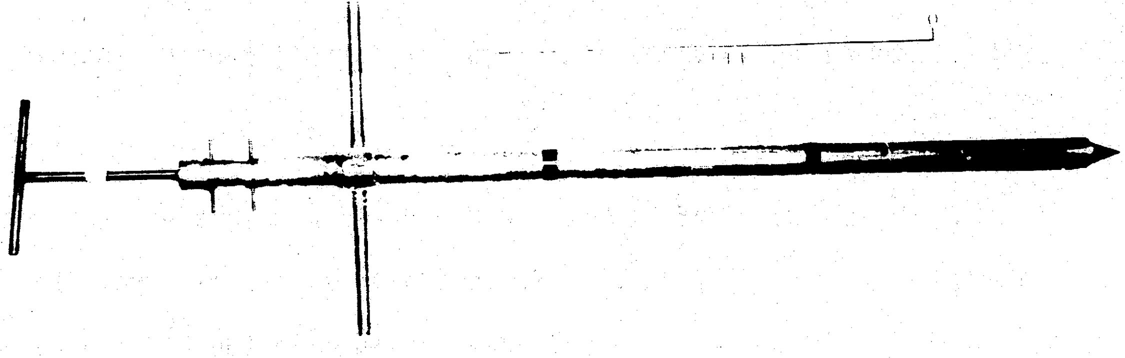
On August 22, 1975, the three M-barrels were opened, sampled, closed and sealed within 1-1/2 hours. Following this, the M-barrels and the test models R, PD, PW, U1, U2, and U3 were logged with the ^{252}Cf-based neutron-neutron probe. A total of 34 logs were obtained. Mr. M. Callihan assisted throughout the data taking.

Sample Taking and Results

For each M-barrel, three approximately equidistant locations on the surface ($\approx 120^{\circ}$ apart) were chosen and three aliquant samples ($\approx$5 gm/sample) were extracted from selected depths at each of these three surface locations. This procedure yielded nine samples per barrel. The depth of the sand and alum $[Al_2 (NH_4)_2 (SO_4)_2 \cdot 24H_2O]$ mixture was approximately 35 inches for each barrel. Thus a sample in the vertical plane was extracted from the top seven inches, the middle seven inches, and the bottom seven inches.

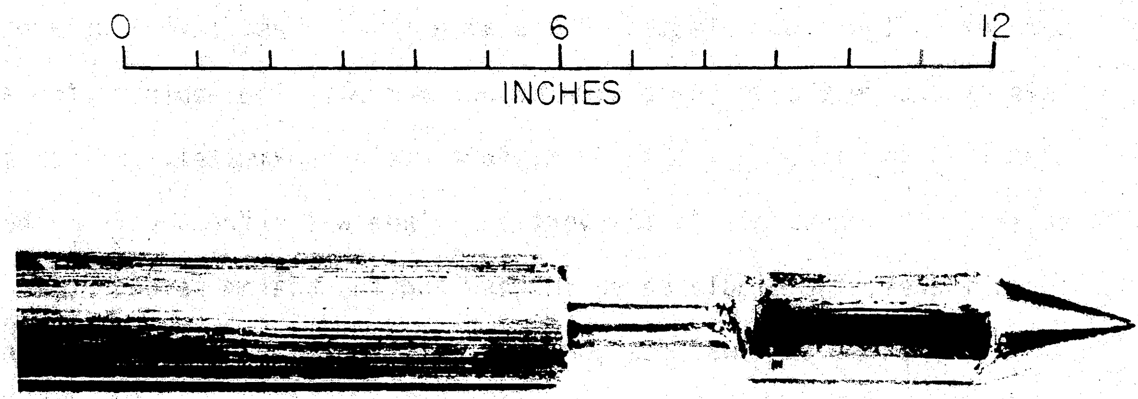
A special tool was designed and fabricated to perform this selective sampling procedure. Two views of the sampling tool are shown in Figure 1. The tool consists of a hollow tube with two large rigidly attached handles; the sample collector, attached by a long shaft with a handle, passes snugly through the tube. The sample collector gathered a sample from a desired location in a manner analogous to coring out the inside of an apple. The sampling procedure consisted of pressing the entire unit, as shown in Figure 1A, down into the sand-alum mixture (one of the small pins shown kept the sample collector from retracting within the tube), removing one of the pins

1A



View of the entire sample extraction unit. The sample collector is within the tube at the left, only the point of it is exposed. Note the two small pins to the right, between the rigid handles of the housing and the smaller handle of the sample collector.

1B



View of the sample collector extended about two inches beyond where it is normally positioned while "coring" a sample. One edge of the slot is sharpened while the other edge is rounded and has a smaller radius of curvature than the collector so as to guide the sample towards the interior of the collector.

Figure 1

and depressing only the sample collector handle, while twisting, to obtain a sample. Figure 1B shows the sample collector extended about two inches beyond where it normally was located while extracting the sample. The sample collector was then retracted within the tube and the entire unit removed from the mixture.

Each extracted sample was placed in a container having a hermetic seal and then labeled according to location within a particular barrel. The sample extraction, labeling, and packaging was made under the supervision of the BFEC Chemistry Department. Arrangements to have the analysis for the hydrogen concentration performed by Accu-labs Research, Inc. (Wheat Ridge, CO), were also made by the Chemistry Department.

The results of Accu-lab's analysis of the 27 samples are given in Table I. Note that equidistant, hence symmetric, surface locations are arbitrarily designated as A, B, and C for each barrel. The averages both vertically and for similar depth intervals (horizontal) are given along with the overall average for each barrel (at the lower right, just below each data set). In Table II the overall averages are compared to values determined in November 1971 at which time the barrel contents were reblended and analyzed.

The large range of values and their lack of correlation in both the vertical and horizontal planes of the M-barrels as shown in Table I was judged sufficient grounds to disregard the results of the chemical analysis. The comparison shown in Table II is not unexpected considering the data base used in arriving at the mean. The large uncertainties associated with these means merely reflects the diversity of the data in Table I. Unfortunately, disregarding these results, nullified one of the planned objectives which was to assess the status of the M-barrels as useful moisture models. The questions on whether settling has occurred within the barrels cannot be addressed. Further,

TABLE I

Hydrogen Analysis of M-Barrel Samples Performed by Accu-Labs Research, Inc.

% Total Hydrogen

M1 Barrel	A	B	C	Averages
Top	0.28	0.43	0.53	/ 0.413 ± 0.126
Middle	0.42	0.26	0.39	/ 0.356 ± 0.085
Bottom	0.32	0.35	0.44	/ 0.370 ± 0.062
Avgs.	0.340 ± 0.072	0.346±0.085	0.453±0.071	0.380 ± 0.086

M2 Barrel	A	B	C	Averages
Top	2.33	2.87	1.87	/ 2.326 ± 0.545
Middle	3.70	1.66	1.97	/ 2.443 ± 1.099
Bottom	1.92	1.51	1.33	/ 1.586 ± 0.302
Avgs.	2.650±0.932	2.013±0.745	1.693±0.328	2.119 ± 0.749

M3 Barrel	A	B	C	Averages
Top	4.21	3.98	4.80	/ 4.330 ± 0.423
Middle	4.08	4.03	5.30	/ 4.470 ± 0.719
Bottom	5.21	3.31	2.29	/ 3.603 ± 1.482
Avgs.	4.500±0.618	3.773 ± 0.402	4.130±1.613	4.134 ± 0.941

Note that A, B, and C are arbitrary designations for the three symmetric locations on the surface of each barrel at which vertical samples were extracted.

TABLE II

Comparison of Results of Moisture Concentration

Barrel	Accu-Labs' Results % Total H	 % eH_2O	% eH_2O (11/71)
M1	0.380 ± 0.086	3.420 ± 0.774	4.40
M2	2.119 ± 0.749	19.071 ± 6.741	12.2
M3	4.134 ± 0.941	37.206 ± 8.469	21.0

without a definitive chemical analysis, any calibration arrived at for the ^{252}Cf-based probe cannot be assigned a statistically significant confidence interval.

In further analysis, it will be shown that the measured response of the ^{252}Cf-based probe is in agreement with older data on the M-barrels using the same probe, taken when the moisture concentrations of the barrels were considered to be well known. Such agreement with extrapolated data gives credence to the belief that the chemical analysis can and should be discarded.

Logging

After the barrels were sealed, they were logged using a scaler sampling rate of 1.0 sec. and a winch speed of 4 ft/min. While the logging was in progress, a neutron detector, positioned on the outer periphery of the barrel and in the midplane normal to the barrel axis, recorded count rates that were approximately 1% or less of the detected response. The data obtained by logging were corrected for deadtime using a value for the time constant τ = 4.946 $\pm$ 0.245 μsec. This value of τ was determined from the logs of U1, U2, and U3 by comparison of logs obtained in the electronically deadtime corrected mode with those obtained using a 1.0 sec scaler sampling rate. The barrels were logged upwards and downwards and although no difference was observed in the data, only those logs taken from the bottom to the top of the barrel are used herein.

The test models were logged with a winch speed of 4 ft/min. in a deadtime corrected mode with print-out every 0.2 foot. As indicated above, some test models were logged with a scaler sampling rate of 1.0 sec.

Calibration

The measured response of the neutron-neutron probe was verified by using data taken with the same probe in 1972 and 1973 which are times after, but moderately close to, the time the M-barrels were reblended and analyzed. These data are given in Table III for the then measured count rate (n) and the dead-time corrected count rate (N). Using the 1972 data and the accepted value for the half-life of ^{252}Cf, the expected count rates for August 22, 1975 (the date of the present measurements) were computed. The results and a relative comparison are given in Table IV. Similar results were obtained using either of the 1973 data sets.

Agreement in Table IV between the measured (N_M) and the expected (extrapolated) response (N_E) to within 5% may be fortuitous, all things considered. The inference that the composition (i.e., the effective water concentration) of the M-barrels is unchanged over the span of four years cannot be definitively made on the basis of this comparison.

Nevertheless, with no other data available, the measured response of August 22, 1975 is used in conjunction with the analysis of November 1971 to arrive at a calibration curve. The results of a parabolic regression analysis are given in Table V and the resulting calibration curve is plotted in Figure 2.

The shape of this curve depends primarily on the source to detector spacing: this distance was 25 cm for the probe used here.

It is interesting to note that parabolic regression coefficients determined (but not shown) for the data tabulated in Table III do not agree with the results in Table V. One expects the coefficient "a" to increase (scale) like $e^{+2\lambda t}$, the coefficient "b" to increase like $e^{\lambda t}$, and the coefficient "c" to remain constant with time. Perhaps this unpredictability in the parameters is due to lack of long-term reproducilbility of the electronics and/or in the setup of the electronics: however there is no evidence to support this speculation.

TABLE III

Data Obtained Using ^{252}Cf Neutron-Neutron Probe

Raw Data Count Rate (sec^{-1})
(Date Measured)

Barrel	n (10/18/72)	n (3/9/73)	n (3/30/73)
M1	16,395	14,741	14,540
M2	13,932	12,510	12,284
M3	10,996	10,012	9,709

Deadtime Corrected* Count Rate (sec^{-1})
(Date Measured)

Barrel	N (10/18/72)	N (3/9/73)	N (3/30/73)
M1	17,843	15,901	15,668
M2	14,964	13,336	13,038
M3	11,629	10,534	10,199

*τ = 4.946 μsec

TABLE IV

Comparison of Expected (N_E) and Measured (N_M) Response of the ^{252}Cf Neutron-Neutron Probe

Barrel	N_{EO} (sec^{-1}) (10/18/72) a)	N_E (sec^{-1}) (8/22/75) b)	N_M (sec^{-1}) (8/22/75)	$\frac{N_M - N_E}{N_M}$ X 100
M1	17,843	8,481	8,840	4.0
M2	14,964	7,112	7,323	2.9
M3	11,629	5,527	5,764	4.1

a) $N_{EO} = \frac{n}{1 - n\tau}$ using $\tau = 4.946$ μsec

b) $N_E \doteq N_{EO} \exp(-\lambda t)$, for $t = 1,038$ days, $\lambda = 7.166 \times 10^{-4}$ (days)$^{-1}$

TABLE V

Parabolic Regression Analysis for the Neutron-Neutron Probe Calibration Curve

Data

Barrel	N(sec^{-1}) measured 8/22/75	(eH_2O in gm/cm^3) measured 11/71	% eH_2O
M1	8,840	0.0703	4.4
M2	7,323	0.1931	12.2
M3	5,764	0.2800	21.0

Functional Form

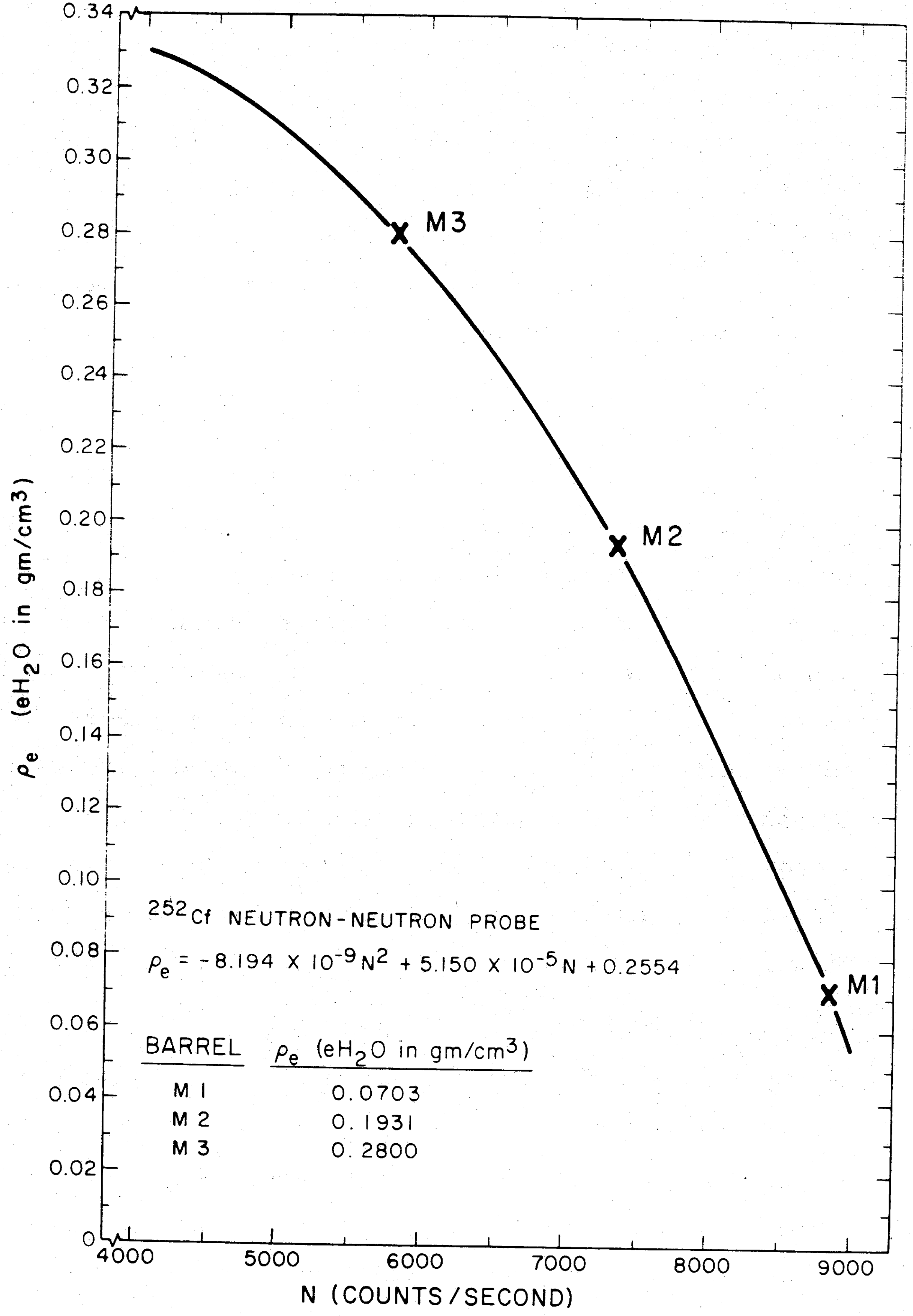
$$\rho_e = aN^2 + bN + c$$

Results

$a = -8.194 \times 10^{-9}$ $sec^2 - gm/cm^3$

$b = 5.150 \times 10^{-5}$ $sec - gm/cm^3$

$c = 2.554 \times 10^{-1}$ gm/cm^3



^{252}Cf NEUTRON-NEUTRON PROBE

$\rho_e = -8.194 \times 10^{-9} N^2 + 5.150 \times 10^{-5} N + 0.2554$

BARREL	ρ_e (eH_2O in gm/cm^3)
M 1	0.0703
M 2	0.1931
M 3	0.2800

Figure 2. Calibration Curve.

Test Model Moisture Concentrations

The logs of the test models R, PD, PW, U1, U2, and U3 are shown in Figures 3 through 8 respectively. For each mineralized zone the percent equivalent moisture concentration (% eH_2O) is indicated. For R, PD, and PW, a scale of values is constructed. The curves through the data are intended merely as a guide and are not quantitative. In viewing the logs of PD and PW it should be noted that their moisture concentrations are similar; a result which is contary to the quoted concentrations of 0.69 and 26.2% respectively.

Finally as a guide to the estimate of the calibration curve accuracy, a comparison of the measured concentrations of U1, U2, and U3 is made with values measured at an earlier time. This comparison is shown in Table VI. The conclusion is ambiguous for lack of the chemical results. The calibration curve may be accurate to 5% as Table IV suggests in which case the moisture concentration of U1, U2, and U3 has increased or, in the other extreme, the moisture concentrations for the M-barrels have changed in which case the calibration is good to 10% accuracy as indicated by Table VI. Of course some combination of these extremes is not ruled out i.e., a variability in the M-barrel concentration along with changes in the U1, U2, and U3 moisture concentrations.

Discussion

The utility of the ^{252}Cf-based neutron-neutron probe is that it can measure hydrogen concentrations in-situ over a relatively large sample volume. Obviously, with regard to the test models, using this probe is preferred over a chemical analysis which requires obtaining costly cores. However, in order to use the neutron-neutron probe it must be calibrated; this is where the need for reliable moisture models arises.

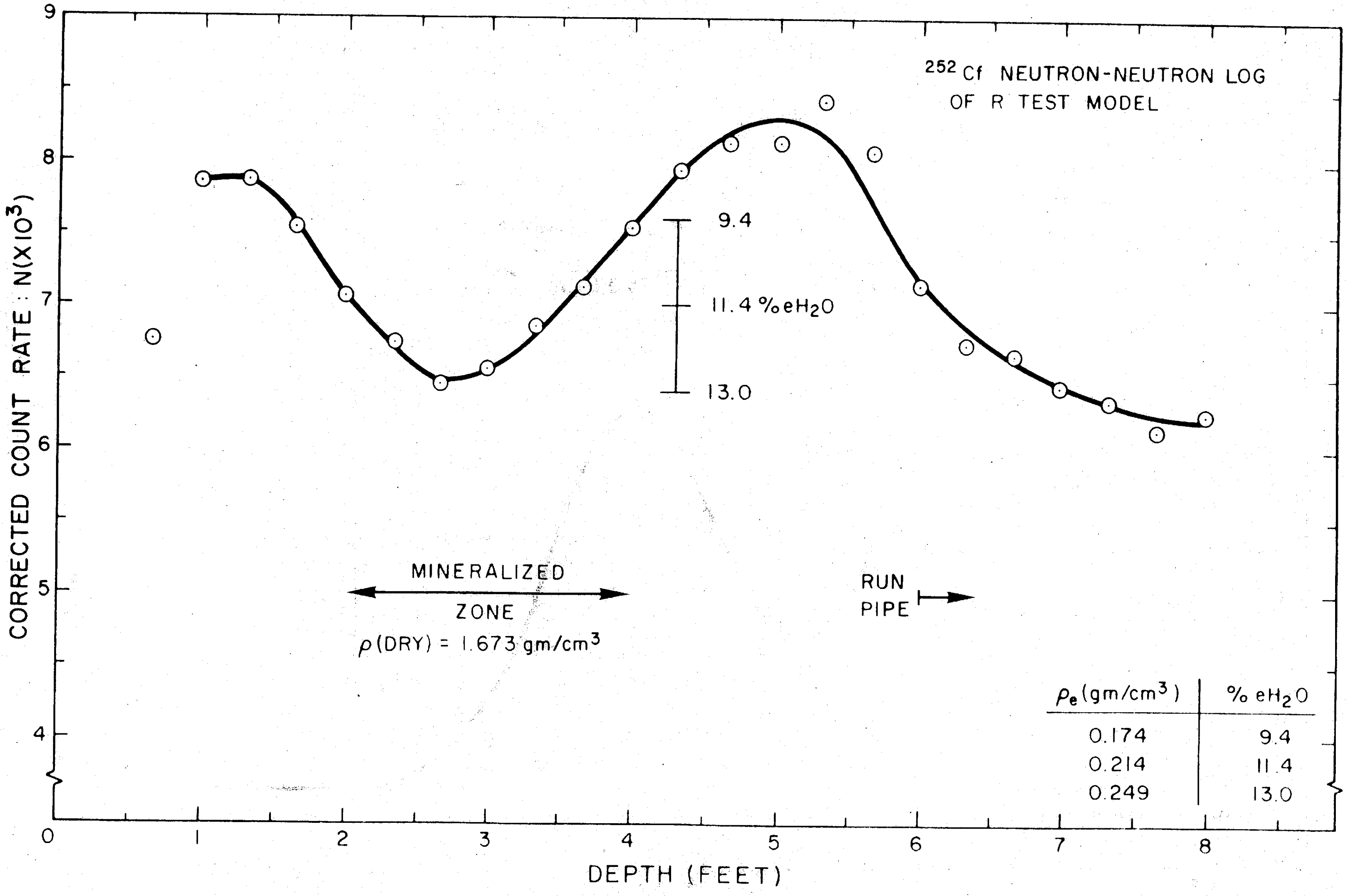


Figure 3. R Test Model

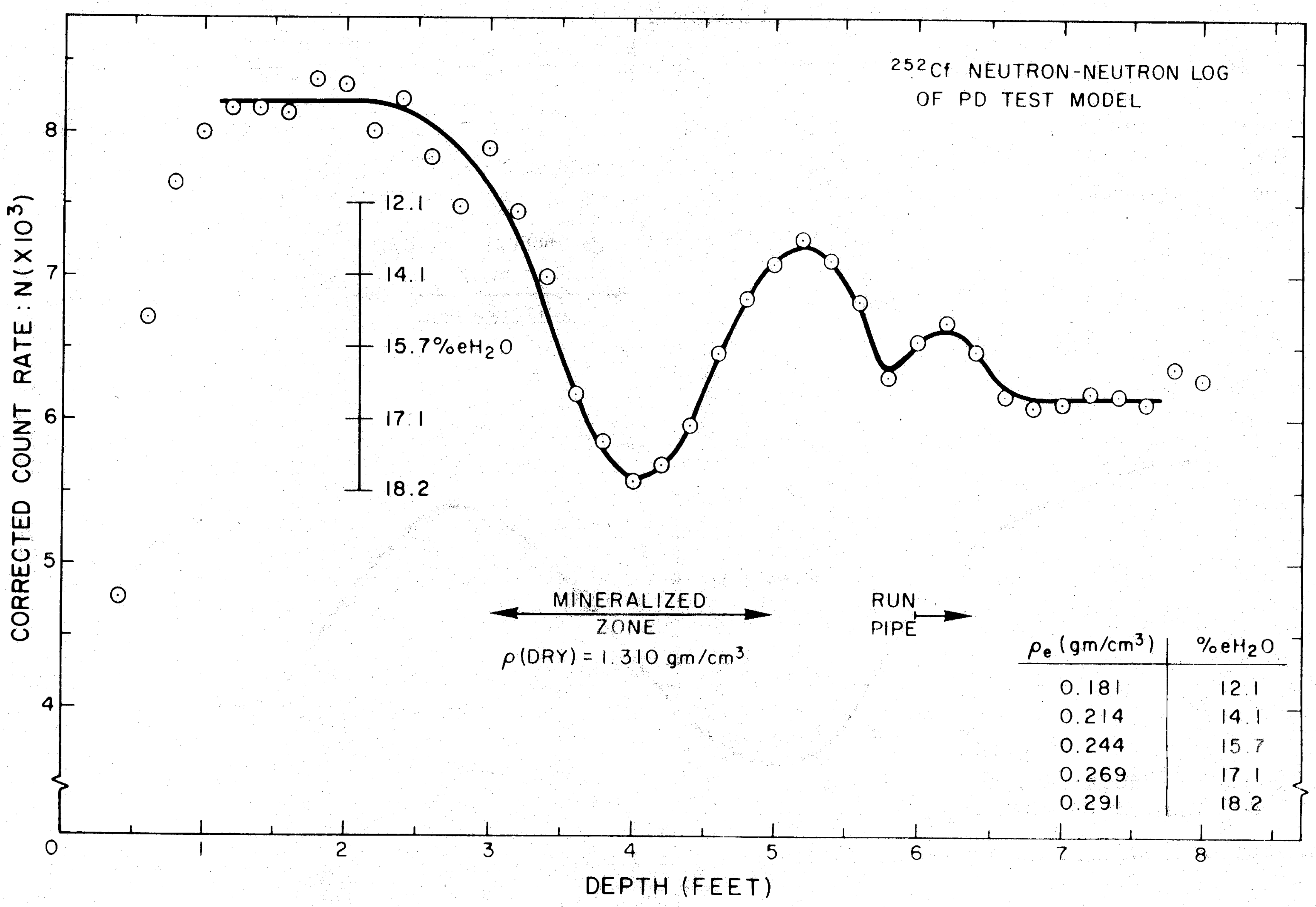


Figure 4. PD Test Model

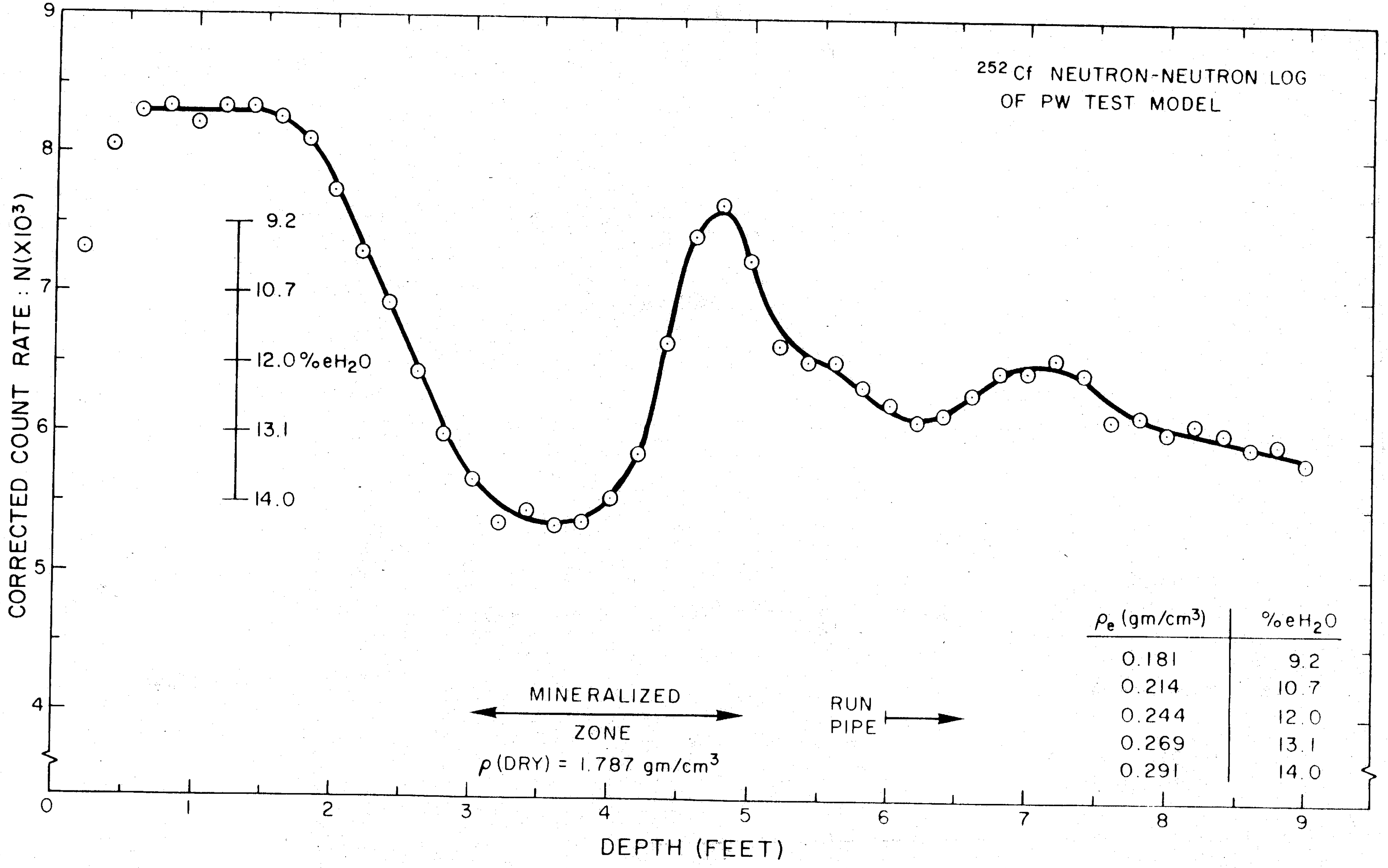


Figure 5. PW Test Model

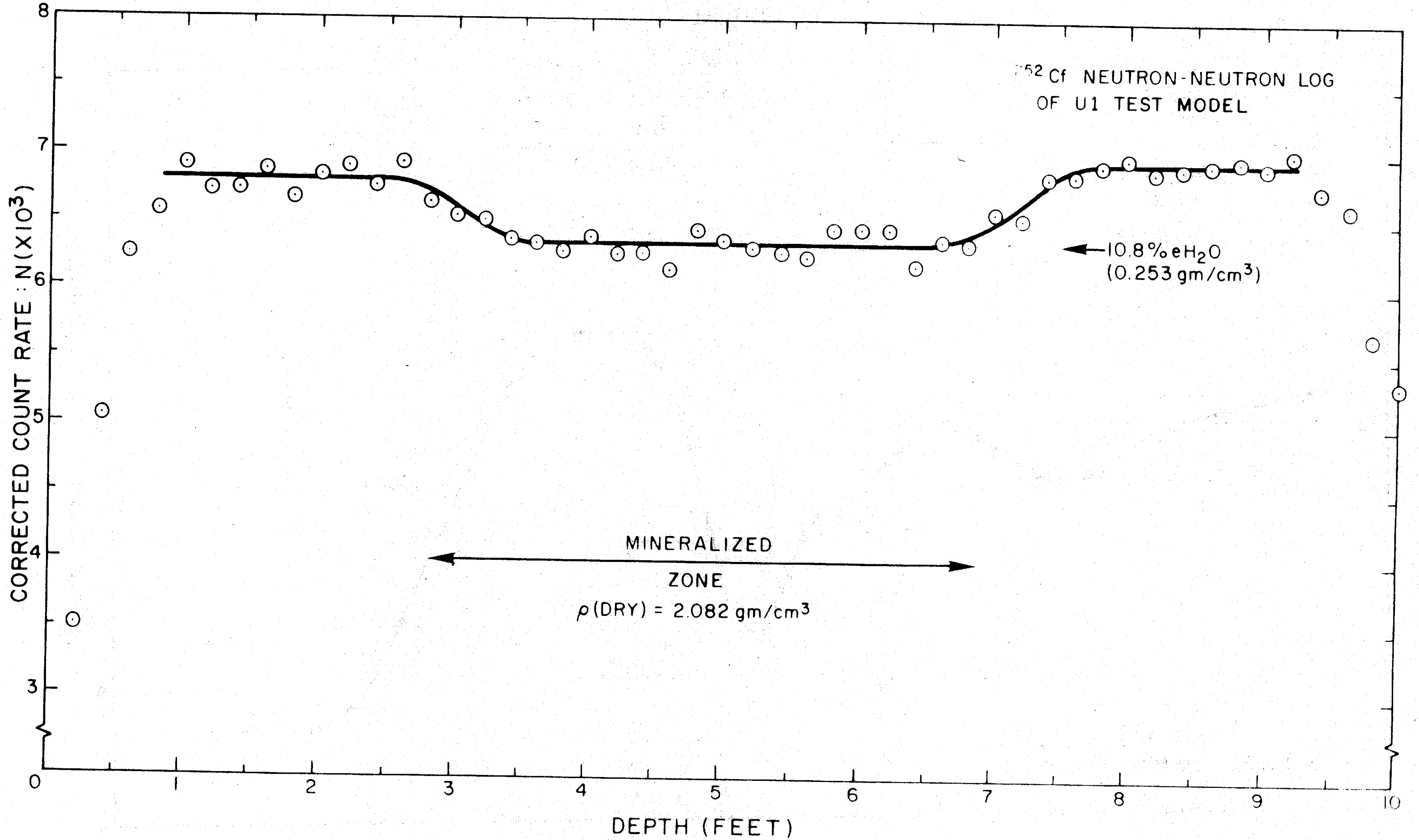


Figure 6. U1 Test Model

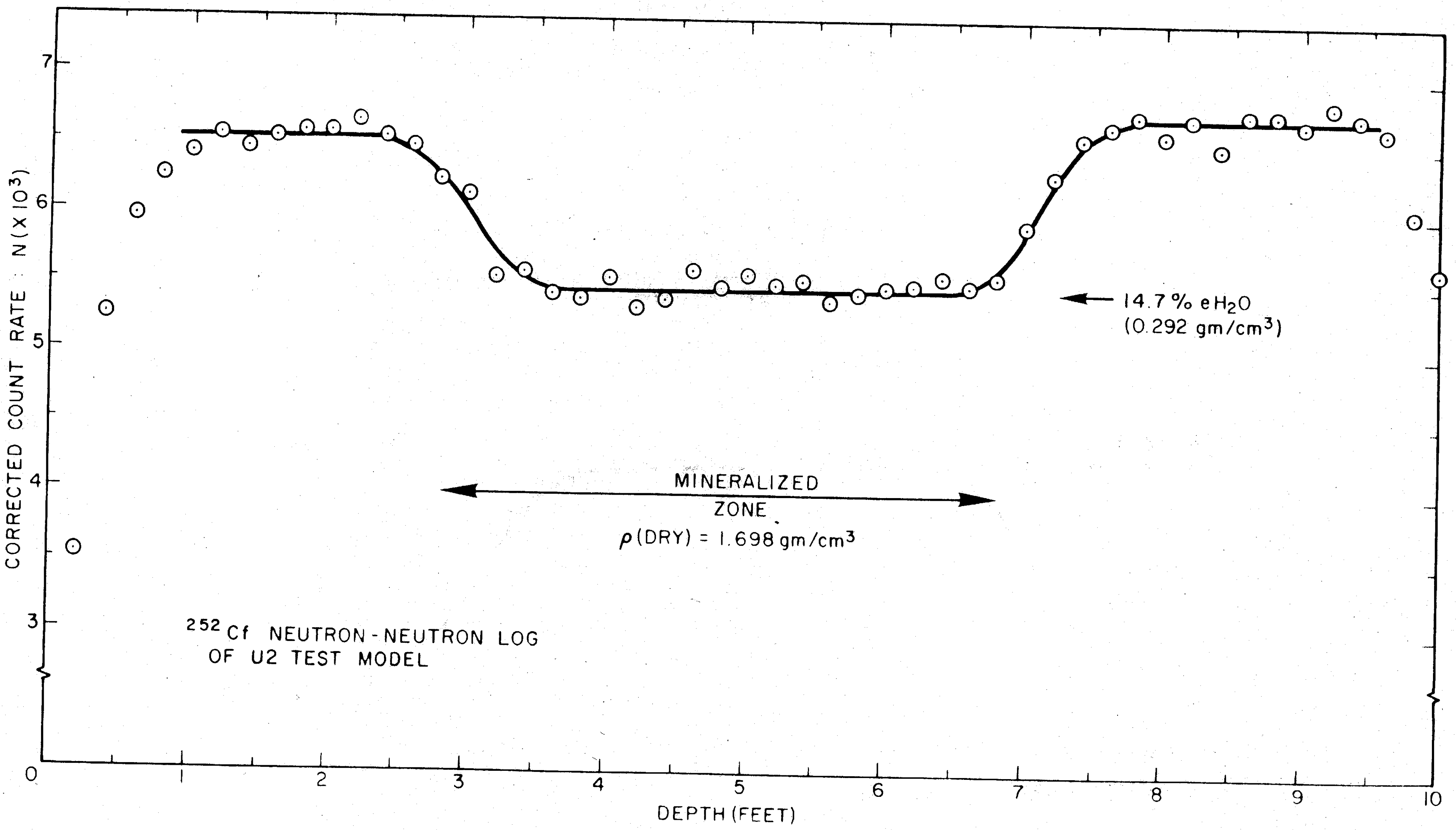


Figure 7. U2 Test Model

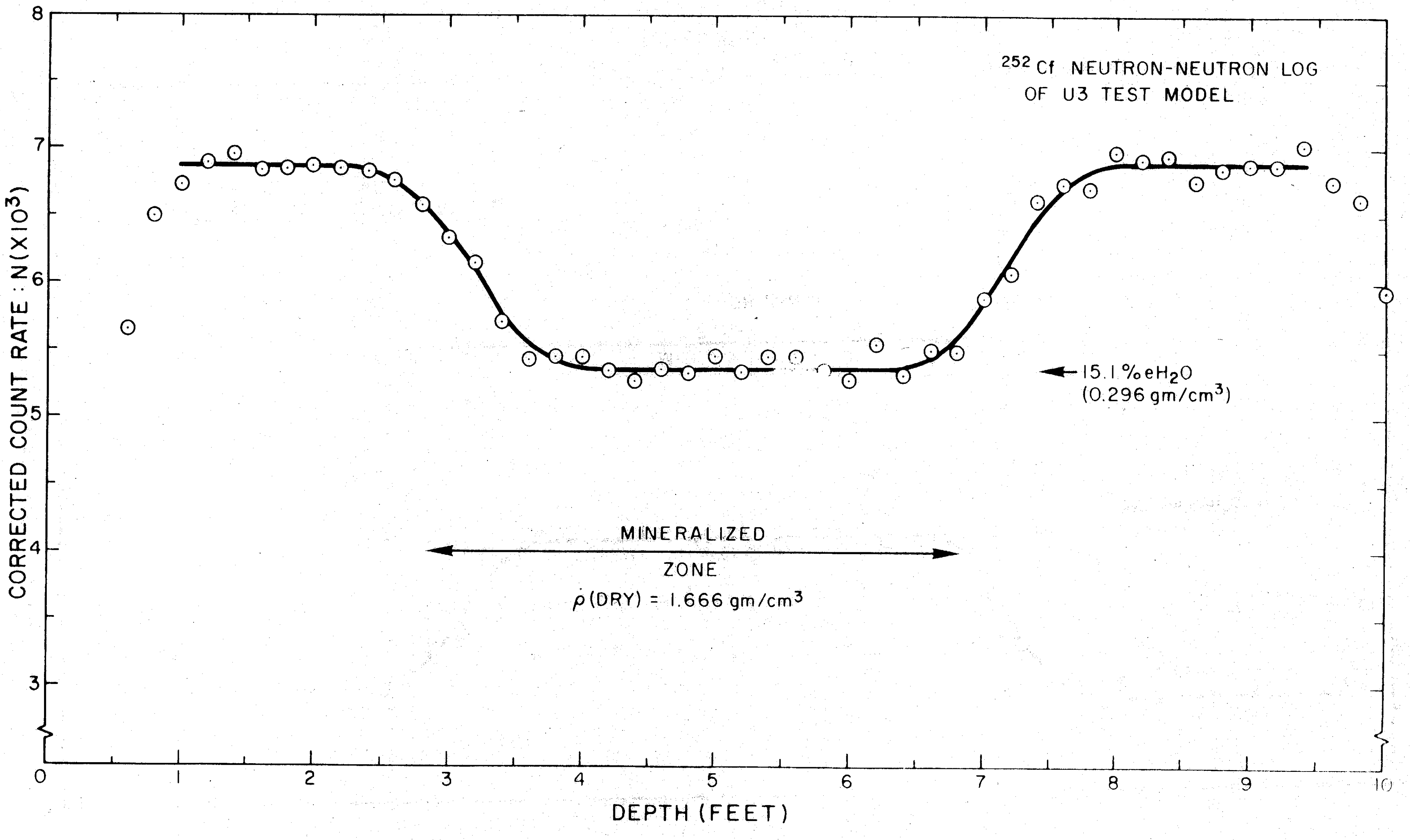


Figure 8. U3 Test Model

TABLE VI

Comparison of Present and Earlier Measured Values of Moisture Concentration in the Ore Zone of the U Models

Test Model	ρ(Dry) (gm/cm^3)	ρ_e(eH$_2$0) (gm/cm^3)	% eH$_2$0 $\frac{\rho_e}{\rho + \rho_e}$ X 100	% eH$_2$0 (old)
U1	2.082	0.2530	10.8	10.1
U2	1.698	0.2922	14.7	14.3
U3	1.666	0.2955	15.1	14.8

It is difficult to speculate the reason for the failure of the chemical analysis, which in this study, was to be the linch pin for the M-barrel calibration. The analysis used viz., the extrapolation of earlier taken data, was to serve as a verification of the chemical analysis and not as the principle means of calibrating the ^{252}Cf-based probe. No definitive statement on the current status of the M-barrels can be made because no data exists to support it. However within this context, certain remarks deserve to be made.

The agreement between the measured and the expected response of the probe as shown in Table IV is encouraging. Perhaps the M-barrels are unchanged from when they were last reblended and analyzed; then again it is possible that the logging probe is insensitive to the limited degree of settling which may have already occurred. In any case, it is still desirable to resample the M-barrels if only to provide input to improved versions of the moisture models. Work elsewhere* is in progress to construct moisture models also using sand and alum although housed in a different geometry.

The PD and PW test models have, for mineralized zones, sealed metal containers in which the ore, moisture, etc., are housed. The results of this work suggest that the dry pit (PD) has taken on moisture while the wet pit (PW) has given up some of its moisture. It is also obvious that the mineralized zones for these test models as well as R, are not thick enough to establish a plateau in the response.

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* J. R. Hearst, "Neutron Logging in Partially Saturated Media", SPWLA Sixteenth Annual Logging Symposium, June, 1975.