

WATER CONTENT OF FRESH CONCRETE MEASURED BY NEUTRON RADIATION

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Experiments were made to study the use of neutron radiation to measure the water content of fresh concrete. Hydrogen in the water causes neutrons to be reduced in energy and backscattered to a detector that counts low-energy neutrons only. Thus, with proper control, the neutron count is proportional to water content in a standardized sample of concrete. Standard $\frac{1}{2}$ -ft³ concrete measures were modified to receive the nuclear probe (a tube carrying source and detector) and contained the concrete sample. The apparatus was calibrated with laboratory-made concretes having a wide range of water contents. Water content of concrete samples taken from truck-mixed batches measured between 0.92 and 1.11 times the average batched unit quantity of water. Readings taken with the probe located in different positions relative to polyethylene models of the sample showed a large influence of the spatial relation of source, detector, and sample. These tests demonstrated the applicability of the nuclear method for measuring water content of fresh concrete but indicate that refinements are needed to increase the precision obtainable.

•THE USE of neutron radiation to measure moisture content of soils and other materials is well established, and commercially available apparatus for this purpose is convenient to use, rugged, and reliable. Experiments were made with such instruments to determine the feasibility of measuring directly and promptly the water content of freshly mixed portland cement concrete. In this procedure, a sample of freshly mixed portland cement concrete is irradiated with high-energy neutrons, and a portion of these are thermalized (reduced to a low-energy state) by collision with the hydrogen nuclei in the water. The resulting radioactive backscatter is measured and with proper calibration is an indicator of water content of the tested sample.

The determination of the water content of fresh concrete can be useful in controlling its quality and in providing assurance that it will be of the desired strength before it is placed. The slump test, although quick and direct, is not adequate for measuring water content or water-cement ratio because it is affected by factors other than water. The Dunagan (1) and Willis-Hime (2, 3) procedures for measuring the constituents of fresh concrete and the oven drying of samples to measure water content (4) have certain virtues, but they all require a relatively long time to complete after sampling. The use of backscattered thermal neutrons to measure water content of soils (5, 6, 7), asphalt content (8), and water content of concrete (9) is described elsewhere. Further experiments on concrete are described here.

FACTORS AFFECTING NEUTRON COUNTS

The radioactive source produces high-energy neutrons that collide with the nuclei of the elements in the sample surrounding the source. Three kinds of interaction take place (11). One is a reduction in energy by successive collisions with hydrogen nuclei, the process called thermalization, and at any time a certain proportion of the total

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available neutrons will be thermal neutrons at the lowest energy state of 0.025 eV. The second interaction is reflection from collision with heavier nuclei, which causes high-energy neutrons to rebound with essentially no loss in energy and to be moving in all directions. However, many neutrons escape collision and reflection and, hence, cannot interact in other ways. Third, neutrons can be absorbed by reaction with other nuclei, producing isotopes and other radiation; such reactions depend on the energy state of the neutron. Thus, the number of neutrons available to react with hydrogen and become thermal is the number emitted by the source less the number that escape or react with other nuclei. Of the number of neutrons available, the number that will become thermal depends on the amount of hydrogen. Hence, a count of thermal neutrons, taken under controlled conditions, is an indication of the amount of hydrogen present in the sample.

Thermal neutrons are counted by detectors that consist of tubular elements containing boron trifluoride gas, enriched in the boron-10 isotope. A high electrical potential (1,400 to 1,500 volts) is maintained between electrodes in the gas. When a thermal neutron strikes the boron-10 nucleus of the boron trifluoride molecule, an alpha particle is ejected. The electrically charged alpha particle is highly ionizing in the boron trifluoride gas, causing a pulse between the electrodes. This pulse is amplified and triggers an electronic counter capable of summing all such pulses that occur within an accurately measured interval of time.

Several factors, as well as the amount of hydrogen present, affect the number of thermal neutrons counted. The intensity of the source, of course, governs the total number of neutrons available in a given time. Because neutron emission is not steady but rather occurs in bursts, a sufficiently long time is needed to get an essentially constant emission in the counting time interval. The size and shape of the detectors, their distance from the source, and their orientation to the source affect the proportion of thermal neutrons that will be intercepted by the detectors and hence counted. It is essential that the electrical potential in the detectors and the amplifier gain remain constant so that a calibration made under one set of these parameters will be applicable during use of the apparatus. Obviously, the electrical circuitry must be adequately shielded against external events that could produce pulses to activate the counter.

If the volume of material surrounding source and detector is large enough, the thermal neutron count will depend on the total amount of hydrogen within the volume affected by the radiation. The size of this volume, in which detectable thermalization occurs, increases as the hydrogen atom concentration decreases. On the other hand, a sample of smaller size and shape will permit a larger share of high-energy neutrons to escape, and, hence, the count of backscattered thermal neutrons will be smaller than would be obtained from a larger sample having the same concentration of hydrogen per unit volume. Thus, the size and shape of the sample and the positions of source and detector tubes relative to the sample must be held fixed to provide an accurate calibration. It is important also that there be no water or other hydrogen-containing material outside the sample but within the volume of detectable thermalization.

The composition of the medium may contribute to the thermal neutron count in addition to the count arising from the hydrogen atoms in the water. If hydrogen is present in any form, for example, in organic matter or hydroxides, it will be counted the same as hydrogen in water. Thermalization caused by other lightweight elements will be detected as well. On the other hand, certain elements that may be present cause a reduction in count inasmuch as they are capable of reacting with and absorbing neutrons. Neutrons so absorbed are not available to be thermalized and counted. Heavy elements present in the material reflect neutrons, preventing their escape and increasing the proportion of those emitted that are available for thermalization and detection. These potential sources of error in measuring water content can be eliminated if counts are taken on the dry ingredients of the concrete. Background counts determined on dry materials are subtracted from the counts obtained on concrete samples to give the net count indicative of water content.

APPARATUS AND PROCEDURE

Two pieces of apparatus were used in these tests. This equipment was commercially available and was designed for moisture measurement in soil at depths below the surface.

Each consisted of 3 parts—probe, shield, and scaler—with electrical cable connecting probe and scaler. The probe consisted of a closed metal tube a little more than 1 in. in diameter and about 1 ft long that contained the neutron source, detector, and amplifier. The lower end of the probe contained shielding to prevent the escape of radiation from the bottom end of the probe. When not in use to test a sample, the probe was housed in the shield to protect the operator from radiation. The scaler provided the electrical power to charge the detector and operate the amplifier and pulse counter. The counters were provided with automatic timing switches to limit the counting time interval.

The neutron source in probe A was 100 millicuries of americium-beryllium, which emitted fast neutrons at a rate of 2.08×10^5 /sec. The source in probe B was a 4.09-millicurie source of radium-beryllium that emitted 4.91×10^4 fast neutrons/sec. Boron trifluoride detectors were used in both probes.

The experimental procedures adopted were designed to be used without modification to the commercially available apparatus. This required that the source and detector in the probe be located within the concrete test sample. It was also felt desirable to transfer the source in the probe from its shield to the test position within the sample without exposing the operator to direct radiation. The apparatus used is shown in Figure 1. It consists of a modified unit weight measure containing the concrete sample with a central access tube in which the probe was placed. The shield was supported above the concrete by an extension sleeve on the access tube. The scaler was located on a nearby bench.

Unit weight measures of 1-ft³ and $\frac{1}{2}$ -ft³ capacities were provided with a central steel tube welded to the reinforced base. This tube was just large enough to accommodate the probe, keeping it free from contact with the concrete. This prevented wear on the probe and possible jarring of the electronic elements that might occur if the probe were in contact with the concrete. It also eliminated the need to clean the probe and protected the operator from unnecessary exposure to radiation. The tube was cut off $\frac{3}{4}$ -in. below the level of the top of the measure and provided with a cap so that the top of the tube would not interfere with filling, compacting, and striking off the measure. The volumes of the modified measures were calibrated so that accurate unit weight values could be calculated.

Preliminary tests were made to ascertain shielding requirements, optimum operating voltages for the probes, standard count readings, variation in count rate with length of counting time, and background counts in air and in empty containers. Radiation safety surveys showed that no special shielding was needed. The standard count was taken with the probe in a fixed position relative to its shield so that comparative readings could be taken from day to day with the probe under exactly the same conditions. This provided a check on the operation of the entire system—source, probe, and scaler. A malfunction in the operation of the equipment would, therefore, show up as a change in the standard count. Background counts taken with the probe in the air and in the empty containers showed such low readings that no significant errors occurred from this source (9).

The procedure followed in taking nuclear readings was simple and straightforward. The sample material was prepared, and the measure was filled, compacted, leveled off, and weighed. The access tube was then uncapped, the tube extension sleeve was affixed, and a plywood cover was placed over the measure to minimize evaporation. The probe in its shield was placed in position over the measure, and the probe was lowered into the access tube. The operator then walked away from the unit, started the count on the scaler, and remained several feet away until the count was complete. The count and its elapsed time were recorded, the probe was drawn up into the shield, and the assembly was returned to its case until needed for the next observation. Standard counts, with the probe in its shield, were taken at the beginning and at the end of each period of work in the laboratory.

Samples of cement and oven-dried sand gravel were tested to determine whether these ingredients of the concrete would affect the neutron counts. The measures were filled with each of these materials in turn, and counts were taken. The thermal neutron counts were so low that no corrections were necessary for thermalizing elements in the dry constituents of the concrete in these experiments.

CALIBRATION OF PROBES

The probes were calibrated with concrete and mortar mixes covering a wide range in water contents. This procedure gave a direct calibration on concrete materials and also provided data to judge the accuracy and reliability of the calibrations. Water content varied from about 9 to 15 lb/ft³ (30 to 50 gal/yd³) although usual concrete mixes are found in the lower end (30 to 40 gal/yd³) of this range. However, when the largest range was used that could be obtained without undue segregation of the mixtures, the calibration could be established more definitely.

The results of the calibrations for water content are shown in Figures 2 and 3. The abscissas are total water content, including absorbed water, and the ordinates are thermal neutron count ratio. The quantity of water in the measure was found by proportion of weights from the known quantity of water batched, the total batch weight, and the sample weight. Weight of water per cubic foot was calculated from the calibrated volumes of the measures and converted to units of gallons per cubic yard. Neutron counts are expressed in terms of the thermal neutron count ratio, N_a , which is the ratio of the test count divided by the standard count for the same time interval. Each plotted point represents a test batch of concrete. Linear regression curves were fitted to the data by the method of least squares, and these lines together with their equations are shown in the figures. The dashed lines are the limits of the 95 percent confidence interval. It is 95 percent certain that the actual water content of a tested sample as determined by this procedure and on these materials lies within the limits of the confidence interval shown. The index of correlation, I , for each curve shows the per-unit proportion of the data used for the curve that are within 1 standard deviation from the curve. The somewhat greater spread of the data points at higher water contents is probably caused by segregation of aggregates or bleeding in these wetter mixes.

These calibration curves show the effect of the sample size. Neutron counts taken on $\frac{1}{2}$ -ft³ samples were considerably less than those taken on the 1-ft³ samples. Within the range of water contents covered, a much larger proportion of the neutrons emitted by the source escaped thermalization and detection when the smaller sample was used.

TRUCK-MIXED TESTS

A field study of the homogeneity of ready-mixed concrete (10) offered an opportunity to try the neutron measurement of water content against carefully controlled full-sized, truck-mixed batches of fresh concrete. This study was conducted by the engineering staff of the National Ready-Mixed Concrete Association to ascertain the effects of size of batch, mixing revolutions, mixing speed, and loading methods on the homogeneity of concrete within a batch. Unit weight, air content, slump, coarse aggregate content, and compressive strength tests were made on samples taken at intervals of about 15, 50, and 85 percent of the discharge of each batch. Homogeneity of the batch was judged by the closeness of agreement between the test results for the 3 samples. In these tests, truck mixers were charged with loads ranging from 7 to 9 yd³. Different methods of charging were used, and the factors under study were varied. The quantities of materials charged were measured accurately on new equipment in a batching plant constructed for this test program. Some batches showed a high degree of homogeneity; others failed in one respect or another to meet the ASTM C 94 criteria.

Water content was measured by thermal neutron count on samples of concrete taken from 46 truck-mixed batches in this program. A sample of about 2 ft³ was taken after 85 percent of the mixer load was discharged. Counts were taken on $\frac{1}{2}$ ft³ of this sample; probe B was used, and the same procedure as described above was followed for calibration of the probe.

The results of these experiments are given in Table 1. Each entry in the table is the test for 1 truck-mixed batch. The results are presented in ascending order of batched water content. It is not the order in which the tests were made, nor were 2 batches at the same water content loaded into the mixer or mixed in the same way.

The batched water content was calculated from the total weights of materials loaded into the mixer, with correction for the air content. The measured water content was

Figure 1. Arrangement of apparatus for measuring radiation in concrete samples.

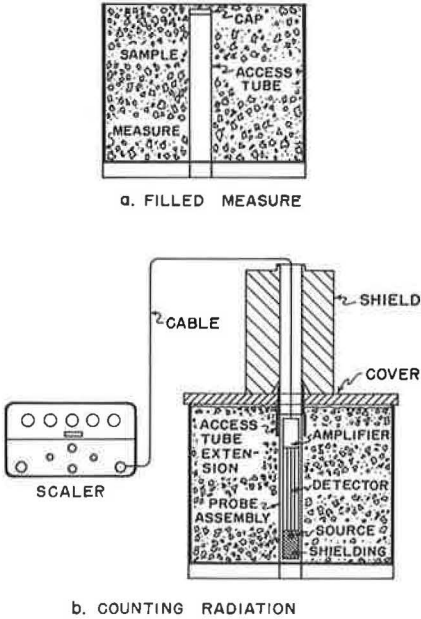


Figure 2. Thermal neutron count ratio versus water content, probe A.

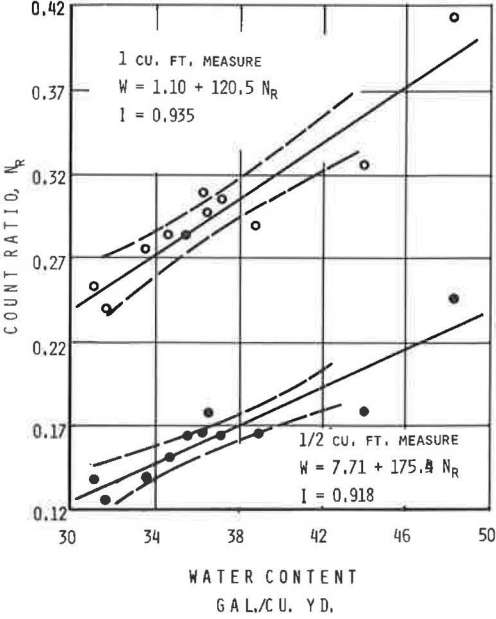


Figure 3. Thermal neutron count ratio versus water content, probe B.

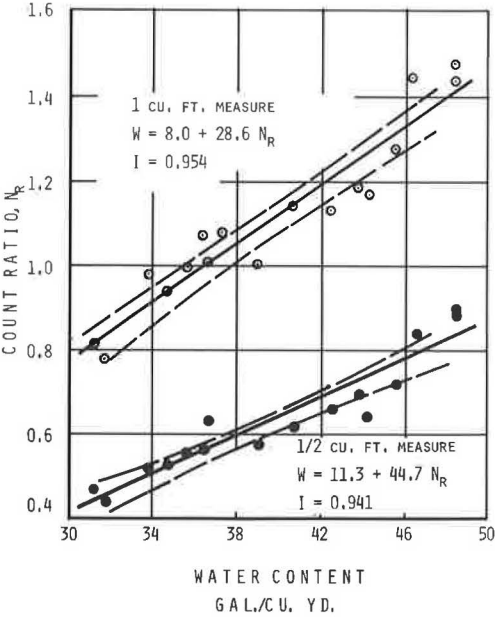


Table 1. Batched and measured water content of truck-mixed concrete.

Gal/Yd ³			Gal/Yd ³		
Batched	Measured	Ratio	Batched	Measured	Ratio
32.7	34.8	1.07	35.6	34.5	0.97
33.0	34.8	1.06		34.6	0.97
33.3	35.3	1.06		35.7	1.00
	36.6	1.10		37.3	1.04
	36.9	1.11		34.3	0.95
33.7	35.3	1.05	36.0	34.6	0.96
	35.4	1.05		35.2	0.98
	36.3	1.08		35.3	0.98
	36.3	1.08		35.8	0.99
	36.8	1.09		36.7	1.02
34.0	35.3	1.04		37.8	1.05
34.3	33.2	0.97	36.2	34.3	0.95
	35.8	1.04		35.2	0.97
	36.0	1.05		36.7	1.01
	36.0	1.05		33.8	0.92
	37.4	1.09	36.5	33.8	0.92
35.0	35.2	1.01		35.0	0.96
35.3	35.2	1.00		35.9	0.98
	35.4	1.00	36.9	33.9	0.92
	36.0	1.02		34.4	0.93
	37.4	1.06		34.6	0.94
	37.4	1.06		34.7	0.94
	38.9	1.10		36.6	0.99

Note: Average value of M/B ratio, 1.019; standard deviation in M/B ratio, 0.055, equivalent to 1.9 gal/yd³ water content.

determined from neutron counts taken with probe B and $\frac{1}{2}$ -ft³ samples, and the calibration data shown in Figure 3 were used. For comparison, the ratio of measured water content to batched water content is shown.

The correspondence between measured and batched water contents was not so good as had been hoped prior to the tests. Discrepancies as large as 10 percent were greater than one would want in a useful quality control procedure for fresh concrete. However, there are a number of potential sources of error in the procedure used here, as are discussed later, and these data were sufficiently encouraging to indicate the desirability of refining the method and apparatus to give more accurate measurements.

Another test of the validity of the water content measurements was made on some of the samples from the truck-mixed tests. Out of the total number of batches on which neutron counts were made, 6 showed a high degree of uniformity as judged by the other tests (such as slump and strength) that were performed. This was an indication that the water and cement were distributed uniformly throughout the batch; hence, the water-cement ratio found from the samples tested was a valid measure for the batch. Samples taken after about 15, 50, and 85 percent of the discharge were tested for water content by neutron measurement. Three 6- by 12-in. cylinders for compressive strength tests at 7 days were made from each of these 3 samples.

The results of this comparison are shown in the plot of compressive strength versus water-cement ratio in Figure 4. Four sets of points are shown. The crosses are plots of the average compressive strength for all cylinders of the batch against water-cement ratio as calculated from the weights of materials batched. The solid circles represent the average strength of the 3 cylinders made from the sample at 15 percent of discharge plotted against the water-cement ratio calculated from the average cement content and the water content found by neutron measurement on this sample. Similarly, the open circles and triangles represent these determinations for samples at 50 and 85 percent of discharge respectively. Complete readings were not taken on 2 batches; hence only the observed points are shown.

These data showed the results of the neutron-measured values to be consistent with the average strength versus water-cement ratio results for all batches. The range and average in water-cement ratios were a little higher for the individual samples than for the calculated batch averages. This may indeed be a correct indication of a small lack of homogeneity in these batches.

An attempt was made to measure water content directly on the concrete discharged into a 2-wheel buggy. A closed-bottom steel tube to accommodate the probe was welded to a 14-in. square steel plate. The tube was forced into the concrete in the buggy until the plate rested on the surface of the concrete with no air or free water trapped under it. The probe and shield were then placed on the plate, and the probe was lowered into the tube in the concrete. The arrangement was geometrically similar to the relation between probe, shield, and samples that pertained in the unit weight measures; the source was located at the same distance below the surface of the concrete in both cases. A separate sample of concrete was taken at the same point of discharge from the mixer as was each concrete buggy sample. This sample was compacted in the $\frac{1}{2}$ -ft³ measure, and neutron count readings were taken for comparison.

Thirteen batches were tested in this way for both the buggy and $\frac{1}{2}$ -ft³ samples; probe B was used for all. The ratio of count in buggy to count in $\frac{1}{2}$ -ft³ measure in these cases ranged from 2.04 to 2.67, and the other values were rather uniformly distributed between these extremes. The readings taken in the buggy were more than twice those in the $\frac{1}{2}$ -ft³ measure because the buggy contained a much larger volume of concrete to act on the neutrons. The variation in the ratio of count in buggy to count in measure was disappointingly large. Two causes of the variation were possible: segregation in the concrete in the buggy and, more important, the fact that the buggy was not filled to the same depth each time. This indicated that the size of sample in the buggy was not large enough to contain all of the zone of detectable thermalization around the probe. Hence, if measurements of water content are to be attempted in such vehicles or containers, it is necessary to standardize test conditions and determine a calibration for them.

COMPARISON BETWEEN PROBES

Measurements were taken with both instruments on the same sample or on samples of the same concrete in many of the laboratory and field experiments. The results of these observations are shown in Figure 5, where the readings (thermal neutron count/min) of probe B are the abscissas and those of probe A are the ordinates. Some points on this curve represent data taken on polyethylene layers in the container, as described in the following section. The agreement between the 2 instruments is very good for the entire range of water contents in both the laboratory and field tests. This is to be expected because the geometric relations between source, detector, and sample are similar for both probes.

EFFECTS OF SAMPLE GEOMETRY

An effort was made to study some of the variations observed in measurements on concrete. Tests were conducted on probe A by using polyethylene as the hydrogen-bearing medium in the $\frac{1}{2}$ -ft³ measure. The polyethylene was in the form of disks $\frac{1}{2}$ in. thick, with an inner 2-in. diameter hole to fit around the central tube in the measure and with an outer diameter $\frac{1}{4}$ in. less than the inner diameter of the measure. Different numbers of disks were arranged in the measure in several different spacings, and neutron counts were taken with the probe at several elevations in the measure.

A comparison between polyethylene and water is shown in Figure 6. One pound of water contains the same amount of hydrogen as 0.777 lb of polyethylene. Check readings were made by partly filling the measure with 21.3 lb of water, coming to a depth of 8 in., for which the neutron count ratio was 1.15. Then, 14 polyethylene disks, weighing 16.64 lb (an equal weight of hydrogen) and being 7 in. deep in the measure, gave a neutron count ratio of 1.12. This slight difference is caused by the difference of 1 in. to which the detector tubes were covered by the thermalizing medium.

Readings taken with different amounts of polyethylene in the form of 7, 11, and 14 disks uniformly spaced through the depth of the measure are shown in Figure 7. Neutron count ratios are plotted for each case as abscissas. The ordinates of the diagram are the locations of the radioactive source as the probe was raised by $\frac{1}{4}$ -in. increments. With 7 disks, the neutron count ratios vary from 0.23 with the source $1\frac{5}{8}$ in. above the bottom of the measure to about 0.20 with the source $4\frac{5}{8}$ in. above the bottom of the measure. Larger ratios were found for the other cases with more polyethylene in the measure. The relative magnitudes of the count ratios for the same probe position are not linearly proportional to the weight of polyethylene in the measure. The fact that the count ratios increased disproportionately with increased weight of polyethylene indicates that the greater spacing between disks allowed more neutrons to escape when fewer disks were used.

The results of the third set of measurements using polyethylene are shown in Figure 8. In these experiments, a 4-in. thick layer (of 8 disks) of polyethylene was located at each of 3 positions in the measure: bottom, middle, and top. For each position of the thermalizing medium, the probe was raised so that the source moved through the full height of the measure. Neutron counts were taken at many different heights of source to give data for the 3 curves plotted.

With the polyethylene on the bottom of the measure, the neutron count ratio varied from about 0.50 with the source in its lowest position to a very small value as the probe was raised to midheight of the measure, and then to an increased value as more of the detector in the probe became encased in the shield. With the polyethylene in the middle position, the ratio increased as the source entered into the polyethylene, decreased as the amount of polyethylene between source and detectors decreased, then increased again as more of the probe was shielded.

In all of these tests, the detector tube entered the shielding as the probe was raised in the measure. Consequently, high-energy neutrons were thermalized within the shielding and were counted by the part of the detector tube within the shield.

Figure 4. Compressive strength for observed water-cement ratios, truck-mixed samples.

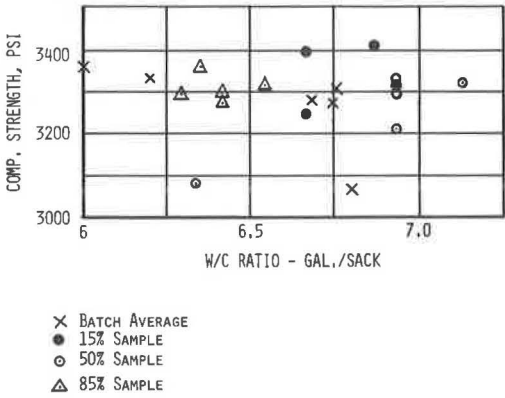


Figure 5. Comparison of probes.

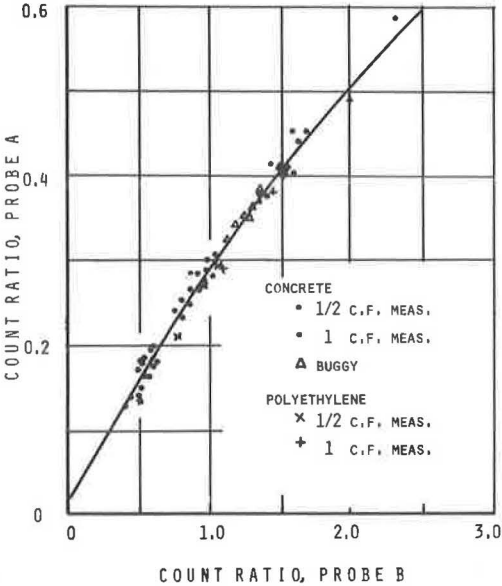


Figure 6. Comparison of count ratios for equal weights of hydrogen.

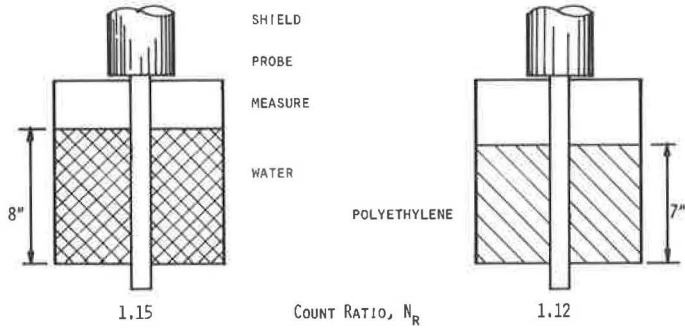


Figure 7. Effect of position of source on count ratio.

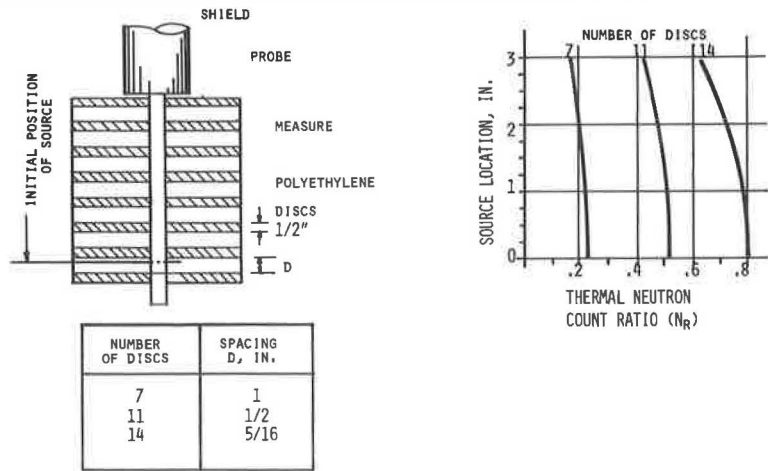
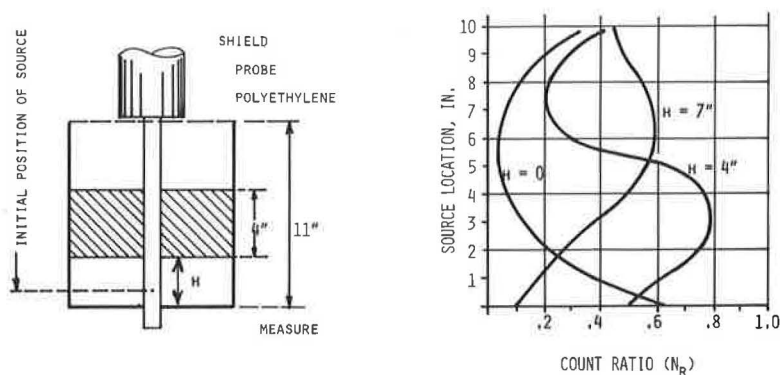


Figure 8. Effect of position of thermalizing material on count ratio.



DISCUSSION OF RESULTS

The results obtained thus far show that water content can be measured by this procedure of neutron irradiation and counting of the thermalized neutrons in a standardized sample. However, the results are not so precise as desired in a quality control procedure for concrete, and it is necessary to examine the possible sources of error or uncertainty in the procedure. It was expedient to use existing readily available equipment in these experiments to explore the feasibility of the idea. Changes in technique, apparatus, or test conditions might then be made to improve the precision of the results.

The size of the concrete samples must be larger than any used here to eliminate the size effect from the calibration. This would be impractical; hence, a standardized size and shape of concrete sample are essential.

Certain aspects of the problem of securing a representative sample of concrete have particularly significant effects on this method for finding water content. Variations in the composition of the sample, as caused by aggregate segregation or by bleeding, can cause error because the water content of the material nearest the source is most effective in the thermalizing process. Further, because mortar always will be closest to the tube containing the probe, the water content measured may be somewhat larger than the average for the batch. Aggregate size, although not studied experimentally here, will undoubtedly have an influence on the calibration.

Erroneously high neutron counts may be caused by water or other hydrogen-bearing material near the measure and probe. It was noted that the readings were increased if the operator stood near the measure. Similarly, water on the floor or in the earth beneath the measure can be detected and will increase neutron count readings. The influence of temperature on the apparatus is another source of discrepancy; calibrations should be checked for the temperature at which the apparatus will be used. It is possible under some conditions of local electrical static that the electrical shielding of the amplifier may be insufficient, and false counts may be produced.

The results reported here are encouraging, but somewhat better precision than was found in these tests is desirable. Improved procedures and apparatus are needed to provide better protection from external influences, calibration that accounts for the effects of elements other than hydrogen, and arrangement of the sample, source, and detector to eliminate local effects caused by the position of aggregate particles.

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