DETERMINATION OF CEMENT IN CONCRETE BY ACTIVATION ANALYSIS WITH CALIFORNIUM-252

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Results from neutron activation analysis of in-place and plastic concrete samples are presented. Data were obtained by equipment suitable for and operated under field conditions. The system described for determination of cement content of in-place concrete includes a 35-microgram Cf-252 source, portable activation/shield assembly with remote operating cable, and commercially available detector and electronics. An analysis of in-place concrete is accomplished in 22 minutes. Results for plastic concrete were obtained with a system designed for soil-cement mixtures. Using a 140-microgram Cf-252 source, an analysis could be completed in 9 minutes with an accuracy of ±5 percent of the amount of cement for normal cement contents. A system for analysis of samples of plastic concrete, cores, and soil-cement is described that can be moved to field sites in a trailer. Most existing methods for determination of cement content of concrete suffer because they are too slow, use too small a sample to be representative, and must be done in a laboratory. The only other field measurement technique being studied utilizes low-energy photon scatter. This technique uses only a thin layer of the available sample and fails to achieve necessary accuracy when aggregate varies in size distribution or heavy element content. Neutron activation analysis offers a rapid, simple, field-operational procedure for measurement of cement content. Besides these advantages, activation analysis allows the use of large, representative samples and offers considerable freedom from interferences.

•EXPOSURE of a sample of concrete to neutrons produces measurable quantities of a number of radioactive isotopes. By controlling the neutron energy spectrum, time of activation (neutron bombardment), decay time, measurement time, and instrument settings, certain of the radioactive products representing cement content can be emphasized. For a fixed set of conditions, radioactivity and composition are directly related. The strict relationship between radioactivity at the end of neutron activation and composition is

$$A = \frac{(\text{weight of element})\ (6.02 \times 10^{23})\ abc\ \{1 - \exp\ [-0.693\,(t/T)]\}}{\text{atomic weight of element}}$$

where

A = activity in disintegrations per second;
a = abundance of the reacting isotope of the element;
b = bombarding neutron flux in neutrons per square centimeter per second;

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c = cross section (or probability of reaction) in square centimeters;
t = activation time; and
T = half-life of radioisotope produced.

This relationship is simplified for fixed experimental parameters such as sample size and geometry as well as those mentioned above:

$$\text{Counts} = K\,(\text{percent cement})$$

where Counts = disintegrations measured by instrumentation and K = a constant for the fixed conditions selected. Such a relationship permits formation of a graph relating counts and cement content. Standard samples treated exactly like samples for the fixed experimental parameters generate the graph for a specific set of components.

EXPERIMENTAL PARAMETERS

Table 1 gives the radioactive materials produced in appreciable quantities by short-duration neutron bombardment. Of these observed radionuclides, ^{49}Ca represents cement content better than any other. In areas using siliceous aggregate and sand, the ^{49}Ca is indicative of only the cement content. By instrumental discrimination against gamma energies below 2.5 MeV, only ^{49}Ca and ^{24}Na produce counts. By using short neutron bombardment, decay, and counting times, ^{49}Ca activity greatly exceeds ^{24}Na activity. With large neutron sources, the typical analysis schedule includes a 5-minute neutron bombardment or activation, 1-minute decay for transfer of the sample to counting instrumentation, and a 5-minute counting or measurement period. For small neutron sources or small samples, the schedule may lengthen to 10 : 1 : 10 minutes for activation : decay : count.

The counting follows the procedures and instrumentation established for soil-cement mixtures (2, 3, 4). A 12.7 × 12.7-cm NaI(Tℓ) crystal detects the gamma radiation. Thermal insulation, shock mounting, and neutron shielding protect the crystal for field use. The large crystal gives the sensitivity necessary for detection of the 3.08-MeV gamma radiation from the small quantity of ^{49}Ca produced. Smaller crystals can be used, but they require substantially larger neutron sources along with longer activation and counting times.

The associated electronic instrumentation consists of a tube base with high-voltage divider network fitted to the detector and connected to a single-channel-analyzer scaler system by a single coaxial cable. The scaler system provides high voltage for the detector operation and permits selection of the gamma energies to be included in the measurement. The discriminator of the single-channel analyzer rejects gamma energies below those of the Ca-49. A convenient prepackaged scaler system is the Eberline Instrument Co. model MS-1. With a little electronic modification to obtain better temperature stability, the MS-1 system operates adequately for laboratory and field use since it can accept either 110 VAC or 12 VDC (auto battery) power. The model MS-1 includes solid-state electronics, large LED display, and oscillator timing system in a compact, lightweight package.

The most compact, high-output, and constant-yield neutron source available for activation of the concrete samples in the field is Cf-252. The Cf-252 decays by alpha emission and spontaneous fission. The fission produces a broad energy spectrum of neutrons. This broad energy spectrum of neutrons permits deep penetration into the sample. Figure 1 shows the thickness of a sample producing useful information on cement content. Figure 2 shows the increase in analytical signal with sample area at constant thickness. These data mean that the analytical information comes from several kilograms of sample material. Such a sample has a good chance of being representative of the bulk of material.

The inner construction of the activation/shield assembly for analysis of in-place

Table 1. Radioactive isotopes produced in concrete by short-duration neutron bombardment.

Radionuclide	Half-life	Gamma Energy (MeV)
^{28}Al	2.3 minutes	1.78
^{49}Ca	8.8 minutes	3.09, 4.05
^{42}K	12.4 hours	1.52
^{27}Mg	10.0 minutes	0.84, 1.02
^{56}Mn	2.58 hours	0.84, 1.81, 2.13
^{24}Na	14.8 hours	1.37, 2.75

Figure 1. Thickness of a concrete sample that produces useful information on cement content.

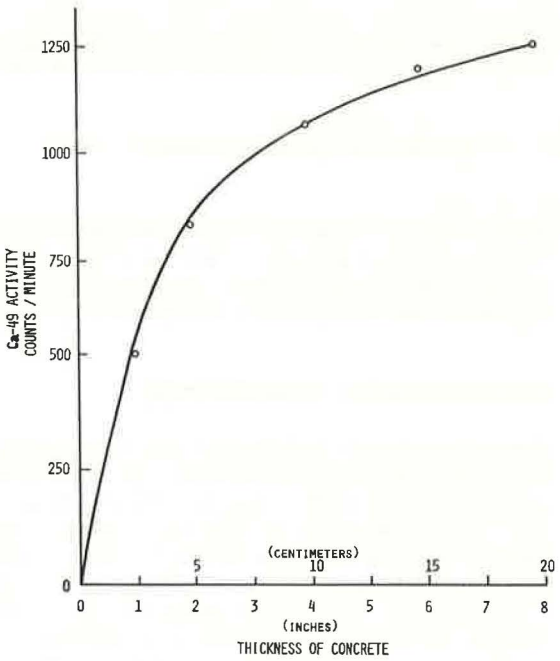


Figure 2. Increase in cement content signal with sample size for concrete slabs 12.5 cm thick.

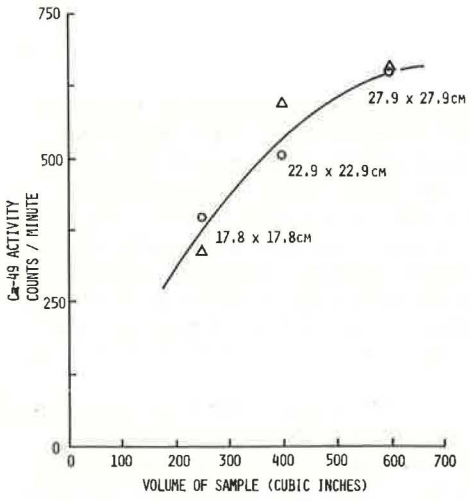


Figure 3. Cross-sectional diagram of activation/shield assembly for analysis of in-place concrete. (WEP = 60 percent water and 40 percent resin.)

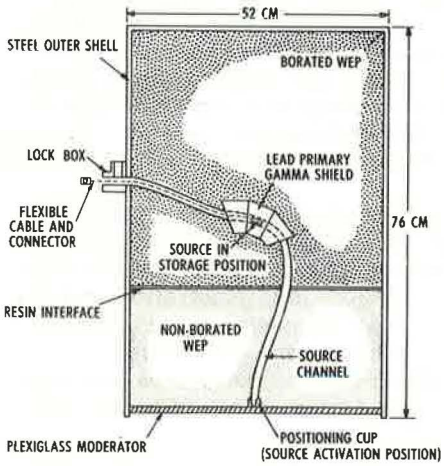
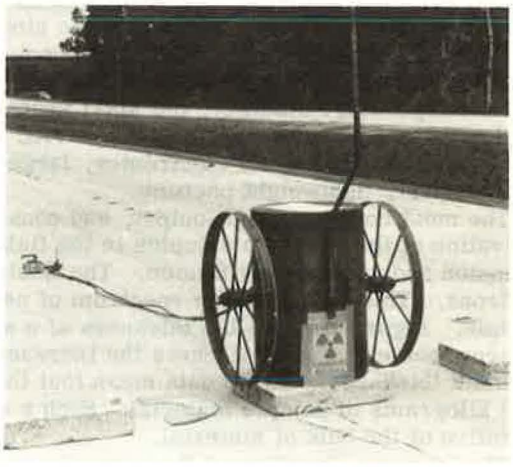


Figure 4. Activation/shield assembly in activation position on a concrete calibration slab; remote crank-out and cable are visible behind the assembly.



concrete is shown in Figure 3. This assembly rolls to the site of the analysis and stands on end for activation of the surface of the concrete. The activation with neutrons begins when the Cf-252 source (about 35 μg) moves from storage position to a point 1.25 in. (3.2 cm) from the end of the assembly. The source moves when the operator turns a crank attached to the source by a flexible cable (such as that used in isotope radiography). The crank and cable arrangement removes the operator to a safe, low-radiation exposure position remote from the activation position. Figure 4 shows the activation/shield assembly in an activation position on top of a calibration slab of concrete. Calibration slabs are 20 × 20 × 5 in. (56 × 56 × 12.5 cm) in size. The remote crank-out and cable are visible behind the assembly. Figure 5 shows the construction of the detector system containing the 3 × 3-in. (12.7 × 12.7-cm) NaI(Tℓ) crystal.

The source size, 35 μg of Cf-252, arose from the need to have an easily portable system combined with a need for rapid analysis. The present system can be moved and operated by 1 man, although a 2-man crew is desirable. Any larger source of Cf-252 would require a shield too heavy for easy use.

The activation/shield assembly for plastic concrete must house a source large enough for rapid results. A compromise between speed and source size (cost and shielding) resulted in the assembly shown in Figure 6. This assembly holds a 150-μg Cf-252 source. As noted, this assembly also uses a moving source. The source moves to an activation position below the sample on a wheel when the operator turns a crank on the side of the shield. Samples sit on the top of the unit inside a series of "donuts" to accommodate samples ranging from 2-liter cylindrical cardboard cartons to those contained in large polyethylene buckets. These larger samples are over 20 cm in diameter and are about as large as can be conveniently handled. Although this assembly can be moved on rollers, its portability will be confined to a small trailer.

With proper "donut" adapters, the activation/shield assembly can also be used for activation of soil-cement samples and standard core samples. Adapters must be used to keep radiation intensity at the operator position at a safe level. The detector system for all these samples is the same as that used on soil-cement samples, as shown in Figure 7.

RESULTS

Operation of the in-place concrete analysis system in the lab using carefully prepared standard slabs produced the results shown in Figure 8. The slabs cover the range from 4 to 7 bags of cement per cubic yard of concrete. Their physical size is 20 × 20 × 5 in. (56 × 56 × about 12.5 cm), with some variation in thickness. Designations of A, B, and C groups of samples mean separately mixed batches of each composition. Corrections for variation in thickness (see Figure 1) are applied to the results. Similar results were obtained in the field on a new section of Interstate 10 in which both the calibration slabs and highway surface were examined.

Results on plastic concrete include only laboratory measurements. Figure 2 includes data taken on plastic concrete samples of varying size. Figure 9 shows the results obtained on cylindrical samples using a 140-μg Cf-252 source in the soil-cement activation/shield assembly (Figure 10). The standard deviation (1 σ or 68 percent confidence) for each set of 5 different samples represents a variation of less than 5 percent of the amount of cement measured, i.e., 10.0 ± 0.5 percent cement. The same samples (sealed in polyethylene bags) activated using a 29-μg Cf-252 source gave the results in Figure 11. Even with an increase in activation time, precision and sensitivity are lost using the smaller neutron source.

CONCLUSIONS

For siliceous aggregate and sand samples of concrete, cement content is rapidly and accurately measured by neutron activation analysis. Both in-place and plastic samples

Figure 5. Diagram of detector system used for determination of cement content of in-place concrete.

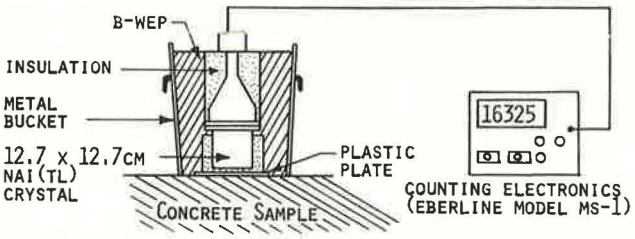


Figure 6. Activation/shield assembly for use with plastic concrete samples.

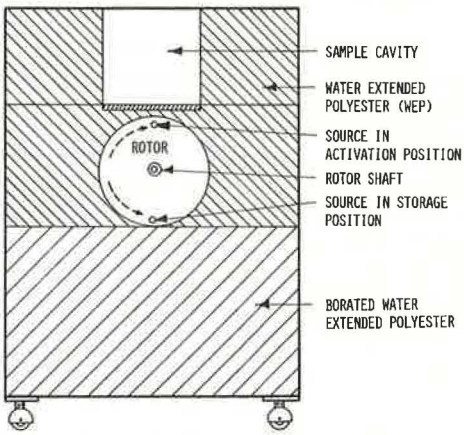


Figure 7. Detector system for determination of cement content in plastic cement, soil-cement mixtures, and standard core samples.

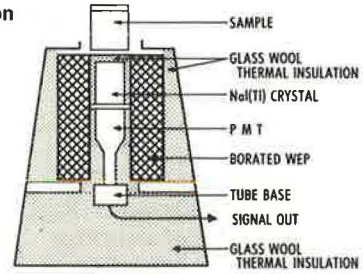


Figure 8. Laboratory analysis of in-place concrete slabs.

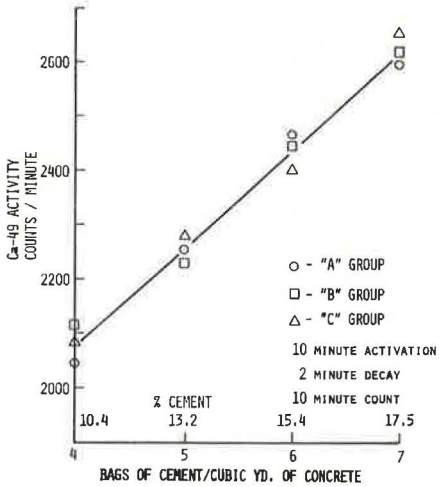


Figure 9. Determination of cement content in plastic concrete samples using soil-cement field system.

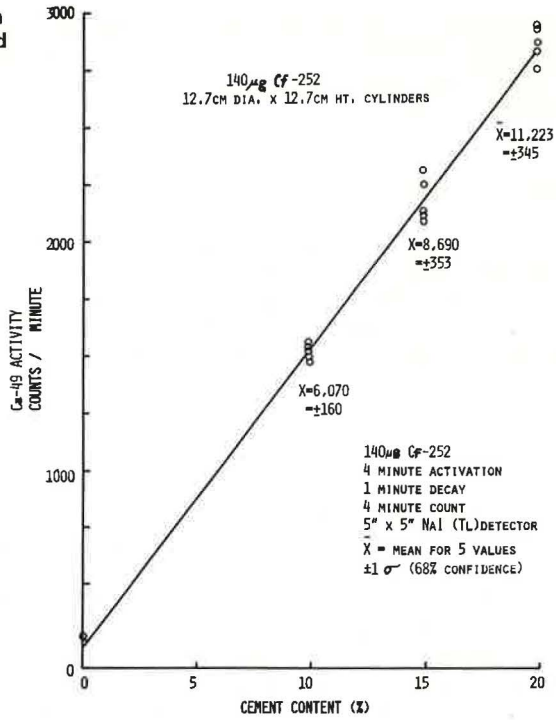


Figure 10. Cross-sectional diagram of field activation/shield assembly for soil-cement samples.

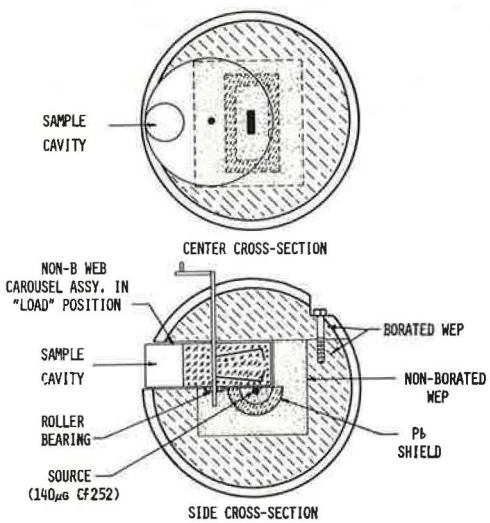
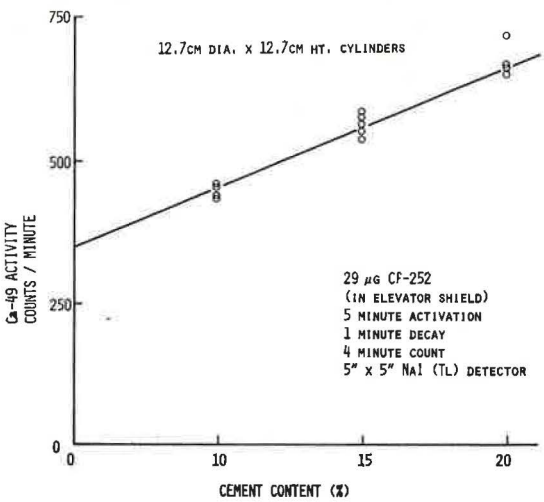


Figure 11. Determination of cement content in concrete samples using a small Cf-252 source in a laboratory activation system.



can be analyzed in field as well as in laboratory environments. Commercially available electronic systems and sources are adequate for use. Activation/shield assemblies must be fabricated by the user or specialty companies since they are not yet commercially available.

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