

CONCRETE ANALYSIS BY NEUTRON-CAPTURE GAMMA RAYS USING CALIFORNIUM 252

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The feasibility of analyzing concrete and cement by a measurement of the neutron-capture or prompt gamma rays was investigated; a 100- μ g californium-252 source was used to supply the neutrons. A lithium drifted germanium crystal detected the capture gamma rays emitted. The capture gamma rays from cement, sand, and 3 coarse aggregates—quartzite gravel, limestone, and diabase—were studied. Concrete blocks made from these materials were then tested. The capture gamma ray response of the calcium, silicon, and iron in the concrete blocks was in accord with the elements identified in the mix materials. The principal spectral lines used were the 6.42 MeV line of calcium, the 4.93 MeV line of silicon, and the doublet of iron at about 7.64 MeV. The aluminum line at 7.72 MeV was observed in some cases but at a lower intensity with the limited electronic equipment available. This nuclear spectroscopic technique offers a possible method of determining the components of sizable concrete samples in a nondestructive, *in situ* manner. In addition, the neutron-capture gamma ray technique might find application in control of the concrete and cement processes and furnish needed information on production operations and inventories.

•FROM THE point of view of the geochemical analyst, concrete may be considered as rock relocated and reformed at the convenience of the engineer. Because the amounts of concrete are large and the industrial value is high, monitoring the processes and the finished concrete is important. Nuclear techniques provide another avenue for determining the composition. Efforts to analyze concrete by measurements of the delayed gamma rays from the decay of neutron-activated products have been reported (1, 10). However, the gamma rays promptly emitted on neutron capture are also distinctive of an element but, because of detection difficulties, have been given less attention by analysts. Nevertheless, experiments to sense the gross constituents of rocks by capture gamma rays were reported some years ago (2).

More recently, the prompt gamma rays from the capture of neutrons from a nuclear reactor (3) and from californium-252 (4) have given information on the mineral values in geologic materials, e.g., gold, copper, nickel, titanium, and manganese. Consequently, this investigation on the capture gamma ray method has been extended to concrete made of typical aggregates. The results indicate that major constituents of formed concrete and its process components can be determined on sizable samples in a non-destructive manner with almost immediate results. The technique of using nuclear spectroscopy is described. The experiments performed suggest possible applications to the concrete industry.

PREVIOUS NUCLEAR STUDIES WITH CONCRETE

Density and moisture measurements by nuclear techniques have been investigated for concrete materials, such as aggregates (5, 6, 7, 8, 9). The density measurements have been made by transmission and backscattering, such as with the 0.66 MeV gamma rays from cesium-137. Moisture determinations have been made with neutron sources, such as radium and beryllium, and depend on the scattering and slowing of fast neutrons by hydrogen.

Element analysis has been made of concrete materials by neutron activation methods, primarily silicon and calcium (1, 10, 16). The fast neutron activation of silicon-28 gives aluminum-28 that decays with a 2.3-min half-life yielding a 1.78-MeV gamma ray for measurement. The thermal neutron capture by calcium-48, which makes up 0.18 percent of natural calcium, gives calcium-49 that decays with an 8.8-min half-life yielding both 3.09 and 4.05 MeV photons. A portable positive ion accelerator (operating from a station wagon) provided 14.3 MeV neutrons for use with concrete pavement (1).

The possibility of concrete additives, such as manganese, that are easily activated has been considered as an analytical aid; tests were also done toward monitoring with the natural radioactivity in concrete materials (1). Unfortunately, neither of these schemes has proved particularly promising.

NUCLEAR SPECTROSCOPIC TECHNIQUES

Element identification by neutron absorption can be accomplished by the measurement of neutron-capture gamma rays or decay gamma rays. When a nucleus captures a neutron, gamma rays are always emitted immediately (prompt gamma radiation) in about 10^{-14} sec. In addition, the product nucleus may be radioactive and will emit additional gamma rays (delayed gamma radiation) while it undergoes decay to a stable state. The gamma energy spectrum of the emitted radiation, either prompt or delayed, is a characteristic signature of the isotopes of an element. In general, the delayed gamma spectrum of a single element is simpler in structure than the neutron-capture or prompt gamma spectrum. In addition, delayed gamma rays are usually less than 2 MeV, whereas capture gamma rays range as high as 10 MeV.

Most analytical applications of gamma ray spectroscopy in the past have been made with delayed or decay gamma rays. Quite often, interferences are reduced by the use of chemical separation to assist delayed gamma activation analysis. With bulk materials, the penetration of low-energy gamma rays may limit the volume of the analytical sample. Smaller sized samples may lead to lower sensitivity for highly disseminated elements of low concentration, and, if the minerals are nonuniformly distributed, the analyses of small samples may not be statistically significant. In addition, the natural background from the thorium, uranium, and potassium decay may complicate the delayed gamma radiation measurements.

Many elements do not respond readily to decay gamma ray analysis because the nuclides formed are not radioactive or do not have convenient half-lives, e.g., silicon, calcium, aluminum, and iron. In contrast, all elements yield neutron-capture gamma rays. Consequently, the potential application of this approach may be greater. Of course, in either decay or capture gamma ray methods, the neutron absorption rate must be high enough to yield a measurable spectra of the element of interest. For these reasons and as a result of the original studies with germanium detectors on pure elements and mixtures by El Kady (3) and Wiggins (4), our preliminary experiments with concrete materials have been made with the prompt gamma radiation techniques. Also, it was recognized that the generally higher energies of capture gamma rays may have advantages over the lower energies of decay gamma rays. These more energetic photons suffer less interaction in reaching the detector, and this may, as indicated above, allow the examination of larger samples of disseminated materials (and presumably with enhanced sensitivity). Moreover, the interference from any decay gamma rays is lessened by measurements of only the high-energy capture gamma rays.

GAMMA RAY DETECTORS

For any spectroscopic method, an appropriate radiation detector is needed; and, until recently, generally poor resolution of detectors made measurement of high-energy gamma rays difficult. With lithium drifted germanium, Ge(Li), detectors, the resolution is good enough so that it is now possible to identify readily high-energy capture gamma rays. In addition, the pair production (positron and electron) produced in the detector by the high-energy gamma rays can aid element identification. The annihilation of the positron produces two 0.51-MeV photons, one or both of which may escape from the Ge(Li) crystal; thus, for high-energy gamma rays, 3 spectral peaks result for each fundamental or full energy peak. These are designated the full peak, E_γ , the single escape peak, $E_\gamma - 0.51$ MeV, and the double escape peak, $E_\gamma - 1.02$ MeV. The spectrum of iron as the oxide is shown in Figure 1 and illustrates the escape effect of the annihilation photons. (Energies on all curves are in MeV.) The dominant full-energy peak (f) of iron at 7.64 MeV is actually 2 closely spaced peaks (a doublet). The larger single-escape peak (s) at 7.13 MeV and the very large double-escape peak (d) at 6.62 MeV are also doublets. This spectrum shows the greater efficiencies of a larger Ge(Li) crystal (about 50 cm³ active volume) for the high-energy escape peaks compared to the full peak. The multiplicity of peaks may complicate a spectrum, but, because a high-energy gamma ray is revealed 3 times, this trio of peaks assists in identifying an element that may be masked by the prompt gamma rays of other constituents.

ELEMENT SENSITIVITY

As indicated, the number of gamma rays produced in a nuclide depends on the neutron absorption that is reflected by the ratio of σ/A , where σ is the microscopic absorption cross section in barns (10^{-24} cm²) and A is the mass in atomic mass units. Moreover, the gamma ray yields are equally important, and this is expressed by the number of gamma rays I of a particular energy emitted per 100 neutrons absorbed. The values of I are well known and have been measured and compiled (13, 14). The combination $I\sigma/A$, then, gives a sensitivity index that has proved useful in predictions of applications of the technique.

The sensitivities, $I\sigma/A$, for the high energies of interest have been tabulated (17). Lately, another tabulation for the low-energy capture gamma rays has been made (18). Spectral contrast is very important, for a high absorber with many gamma ray energies, e.g., cadmium, may give a bland spectrum, while iron, as shown in Figure 2, with a few prominent lines yields the better spectrum (17). (Iron proved useful in calibration.) The sensitivities and past experimental work (3, 4) show that for the higher energies Ti, Cr, Mn, Fe, Co, Ni, Cu, S, Cl, Mg, and Au respond well to analysis by capture gamma rays.

In addition, a number of elements that have a relatively low sensitivity but are often found in high concentration in materials of industrial interest appear suitable for this technique. Table 1 gives elements in concrete and the sensitivity values of the most interesting prompt photon lines for analytical studies. If the sensitivities and the concentrations of elements in concrete are considered, iron is expected to be most easily identified in basaltic concretes and calcium in those with limestone aggregates. For equivalent concentration, silicon should respond about as well as calcium. Aluminum, magnesium, and sulfur should give a detectable but lower signal.

NEUTRON SOURCES

Neutrons for analyzing bulk samples can be obtained from a nuclear reactor, an accelerator, or an isotopic source. Using a reactor requires transporting the sample to that facility, and most of the analysis with decay radiations has been made with reactors. Reactors with their safety, licensing, and scheduling problems may prove inconvenient for some applications. Accelerators, which are more likely to serve single users, offer portability and, as mentioned, have been applied to concrete analysis using a decay gamma ray method (1). Isotopic sources that depend on the alpha-neutron re-

Figure 1. Iron oxide partial-capture gamma ray spectrum, 10 min and 243 grams of Fe₂O₃.

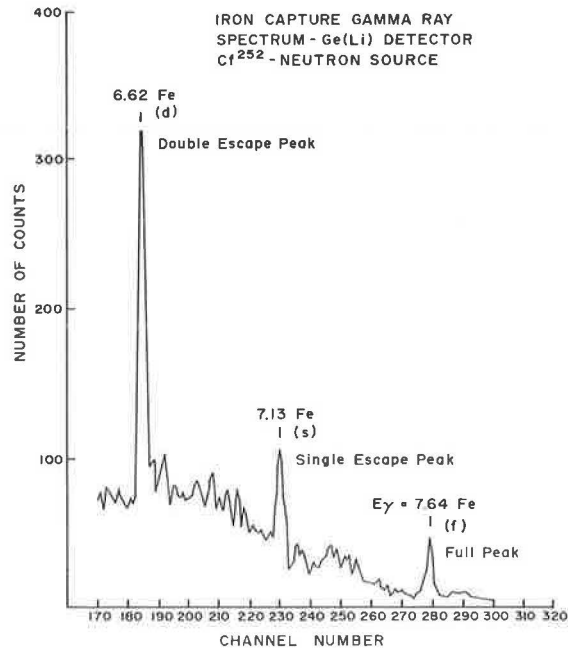


Figure 2. High energy neutron-capture gamma ray spectrum of iron, 100 min and 243 grams of Fe₂O₃.

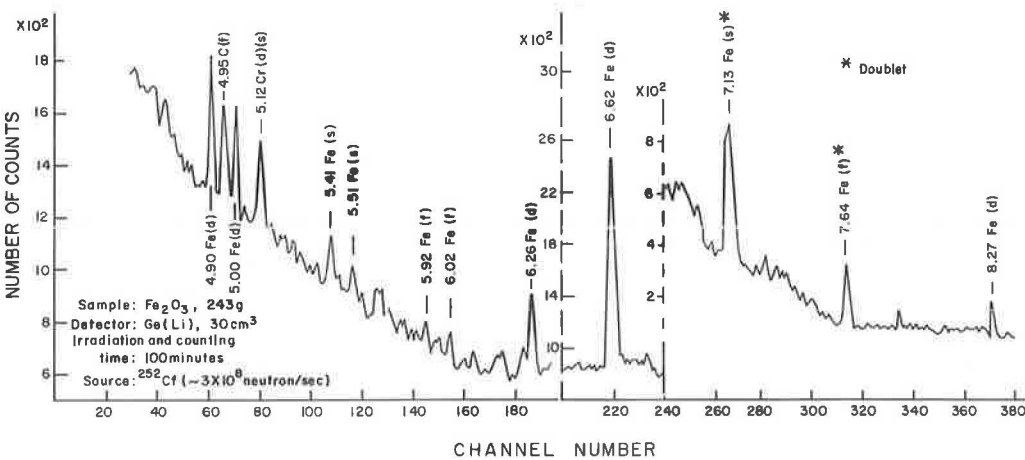


Table 1. Capture gamma ray sensitivities of elements in concrete.

Element	Cross Section (barn)	Atomic Mass Unit	Energy (MeV)	I ^a	Iσ/A
Mg	0.063	24.312	3.916	40.82	0.10
			3.054	10.83	0.028
Al	0.235	26.981	7.724	20.10	0.175
			4.934	70.55	0.402
			3.539	79.58	0.453
S	0.512	32.064	5.420	42.44	0.678
			3.221	19.46	0.311
Ca	0.430	40.08	6.420	28.09	0.301
			4.419	10.79	0.116
Fe	2.62	55.847	7.646 ^b	22.14	1.04
			7.632 ^b	27.19	1.27
			6.018	8.08	0.379
			5.921	8.29	0.389

^aNumber of photons emitted per 100 neutrons absorbed.

^bThese Fe peaks are not resolved in Figures 1 and 2.

action, such as radium, polonium, or plutonium with beryllium, have been used for analytical studies, but the neutron yield is low; most applications with isotopic sources have been with decay radiation, but some work has been reported on the use of prompt gamma rays (19).

Recently, californium-252, a synthetic isotope that undergoes spontaneous fission with a half-life of 2.62 years, has provided a neutron source that can compete in yield with an accelerator and even a low-power reactor (20). For experiments such as those described in this research, the physical size of the californium source is small, and the cost, safety, and licensing problems are much less than those with a reactor. Also, because it is a sealed source, there is essentially no maintenance problem. The low cost and portability indicate that installations of californium-252 as a part of processing control arrangement or for field use should be feasible (15).

CALIFORNIUM SOURCE

The californium-252 source used for this work was approximately 100 μg , yielding about 3×10^8 neutrons/sec. The neutron energy distribution is similar to that from uranium-235 fission in nuclear reactors, but the average neutron energy for the californium-252 source is slightly higher. The associated gamma ray energies from fission and the fission products of californium-252 are generally lower than those of interest in this work. The californium was in the form of the oxide, Cf_2O_3 , and was doubly encapsulated. The inner capsule was made of an alloy of 90 percent platinum and 10 percent rhodium. The Cf_2O_3 was held between 2 small, type 316 stainless steel frits within the capsule. The outer capsule was made of zircalloy-2. (A source encapsulated with stainless steel, type 304L, was used in early experiments but not for the measurements reported here.) These later construction materials were incorporated by the fabricator, the Savannah River Laboratory, to reduce the capture gamma ray background (11, 12). The source outside dimensions were 1.33 by 0.37 in. in diameter; it was held in the end of a 3-ft phenolic (paper base) rod $1\frac{1}{2}$ -in. in diameter.

IRRADIATION ASSEMBLY

The irradiations were performed with this source in a plastic (polymethylmethacrylate) tank 26 by 22 by 26 in. high filled with water (Fig. 3). Cardboard boxes of paraffin were outside the tank and provided 10 in. of additional shielding. The californium source was located within the tank at one side near the bottom. The samples were placed in a given position outside of the tank near the source, as shown. Samples of the cement and granulated aggregate were irradiated in plastic dishes 6 in. in diameter and $\frac{3}{4}$ in. thick, and the concrete was in blocks, 3 by 4 by 16 in. The $\text{Ge}(\text{Li})$ crystal (cooled with liquid nitrogen and shielded with lead, boron as Boral, and cadmium) was positioned on the side of the tank opposite the sample. This provided considerable moderator (water and paraffin) and absorber to lower the outside gamma ray background and to protect the $\text{Ge}(\text{Li})$ crystal from neutron damage. A 400-channel analyzer, with a field effect transistor (FET) preamplifier, a power supply, an amplifier, a biased amplifier, and a plotter served the detector system.

AGGREGATES, CEMENT, AND CONCRETE MIXTURES

Aggregates in common use but of diverse composition were chosen to demonstrate the procedure: quartzite sand from Branchville, Maryland; quartzite gravel from White Marsh, Maryland; limestone from State College, Pennsylvania; and basaltic rock, a diabase, from Chantilly, Virginia. The cement was a blend of type 1 brands. The composition of these raw materials is given in Table 2.

The concrete mixtures contained 5 or 7 sacks of cement/ yd^3 and between 5 and 7 gal of water/sack of cement. The weight of sand aggregate was about 2 to 3 times the cement, and the coarse aggregate (gravel, limestone, or diabase) weight was about 20 to 50 percent more than the fine aggregates. These proportions were chosen more for experimental convenience rather than for quality of concrete. From this compounding, the compositions given in Table 3 on a weight basis for the 7 concrete batches were obtained by calculation.

RESULTS

The constituents that make up concrete, i.e., cement, sand, and coarse aggregates, were irradiated in the experimental arrangement shown in Figure 3. Figure 4 shows the prompt photon spectrum from the cement. The calcium peaks are at 5.91 MeV (single escape, s) and at 5.40 MeV (double escape, d). Silicon is observed at 4.93 MeV (full, f) and 4.42 MeV (s). Lines at 5.63 MeV (d) and 5.12 MeV (s) are from neutron-proton reaction in oxygen to produce the short-lived nitrogen-16. This oxygen was primarily in the water around the neutron source and, hence, appears in all spectra. The spectrum of the diabase rock sample (not shown) reveals mainly the relatively strong 6.62 MeV (d) iron line; also the less intense calcium and silicon lines from the feldspar minerals of the diabase are apparent. The response of the sand and gravel shows primarily the 4.93 MeV (f) and the 4.42 MeV (s) silicon peaks. Figure 5 shows the gamma ray response of the limestone aggregate. The 5.40 MeV (d) calcium line is the most prominent.

The neutron-capture gamma ray spectrum of a concrete block with a coarse gravel aggregate (batch 2) is shown in Figure 6. A relatively small amount of iron is indicated by the 6.62 MeV (d) line. Calcium in the cement is noted by the 5.40 MeV (d) peak, and the silicon lines from both sand and gravel at 4.93 (f) and 4.42 MeV (s) are clear. Figure 7 shows the response of a concrete block with a diabase aggregate (batch 5). The significant 6.62 MeV (d) iron peak and the calcium and silicon are readily apparent. Figure 8 shows the prompt gamma ray spectrum of the limestone aggregate concrete (batch 3) block. The relatively large calcium line from the aggregate and cement is at 5.40 MeV (d), and the silicon peaks at 4.93 (f) and 4.42 MeV (s) are from the sand. The weak peak at 6.62 MeV (d) is expected because of the small amount of iron present.

In general, the intensities of the prompt gamma ray peaks of the spectra reflect the relative composition given in Table 2. The sensitivity curves, shown in Figure 9 for Fe_2O_3 , SiO_2 , and CaO for the concrete blocks tested, are in general agreement with the percentages given in Table 3. The experimental results indicate that iron, calcium, and silicon can be determined in finished concrete and its feed materials with this kind of instrumentation and that iron is the easiest to determine. This is in accord with the sensitivities, I_γ/A , and earlier experiments with pure materials and geologic samples. The aluminum is revealed by the 7.72 MeV line in the spectrum of the high aluminum materials, but at a lower intensity. Other studies lead us to believe that, with more recent electronic equipment, aluminum as well as sulfur can be seen. In addition, from these other studies, we are confident of being able to refine the accuracy limits.

It is realized that elemental content of concrete, in itself, may not be so important as other features, such as cement and water content. However, with a knowledge of the feed material composition and the composition of the finished concrete, this other important analytical information can be deduced.

SUGGESTED APPLICATION

The experiments were performed to show the feasibility of using the capture gamma ray technique in processing plants for concrete materials and in the field for the formed product. Of course, the technique could also be applied in a laboratory near the concrete operation. In either case, an analytical assembly of a shielded californium source and Ge(Li) detector with electronics would be needed. Analytical assemblies (15) have been proposed by the authors for process control, e.g., iron and copper; these could be adapted for concrete. Obtaining the scan of the entire spectrum would not be required; instead only certain energy lines need be counted. This information could be used in turn to control the operation, such as material flow in a process system. For field work, portability is required, and perhaps a mobile laboratory would be useful. Electrical power and liquid nitrogen would be required (a 30-liter supply of nitrogen has been sufficient for the detector for 2 weeks).

For formed concrete, an analytical assembly could be used on roads, foundations, and other reinforced concrete structures (reinforcing iron should be readily identifiable,

Figure 3. Experimental arrangement.

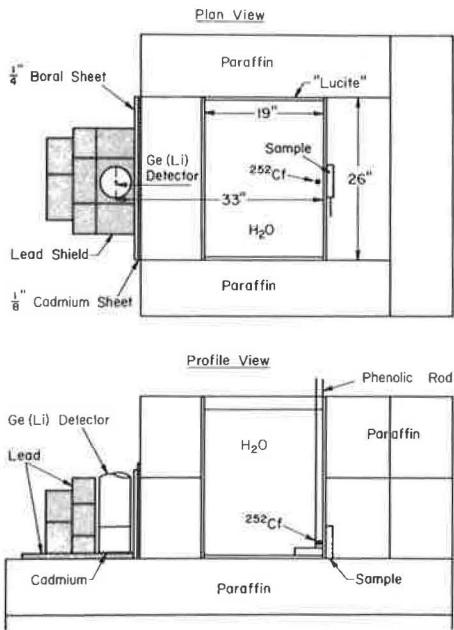


Table 2. Percentage composition of concrete aggregates.

Compound	Cement	Sand	Gravel	Limestone	Diabase
SiO ₂	21	97	98	4	53
CaO	63	—	—	52	9
Al ₂ O ₃	5	2	1	—	17
Fe ₂ O ₃	3	0.8	0.5	—	14
MgO	3	—	—	1	4
SO ₃	3	—	—	—	0.3
CO ₂	—	—	—	42	— ^a

^aAlso some Na₂O, TiO₂, and MnO₂.

Table 3. Composition of concrete batches.

Compound	Gravel Batch 1	Gravel Batch 2	Lime- stone Batch 3	Lime- stone Batch 4	Diabase Batch 5	Diabase and Gravel Batch 6	Gravel Batch 7
Content (percent)							
CaO	7.5	11	30	34	14	13	11
SiO ₂	81	78	40	36	60	68	77
Al ₂ O ₃	1.8	1.9	1.3	1.4	9	6	2
Fe ₂ O ₃	0.9	1	0.6	0.7	7	4	1
MgO	0.4	0.5	0.7	0.9	2	1	0.5
SO ₃	0.4	0.5	0.4	0.5	0.5	0.5	0.5
CO ₂	—	—	18	18	—	—	—
H ₂ O	8	8	9	9	7	7	8
Density (lb/ft ³)							
—	144	144	147	147	147	145	139

Note: Batches 1 and 3 were with 5 sacks of cement per cubic yard concrete, and the others were with 7 sacks.

Figure 4. Neutron-capture gamma ray spectrum of cement (380.5 grams).

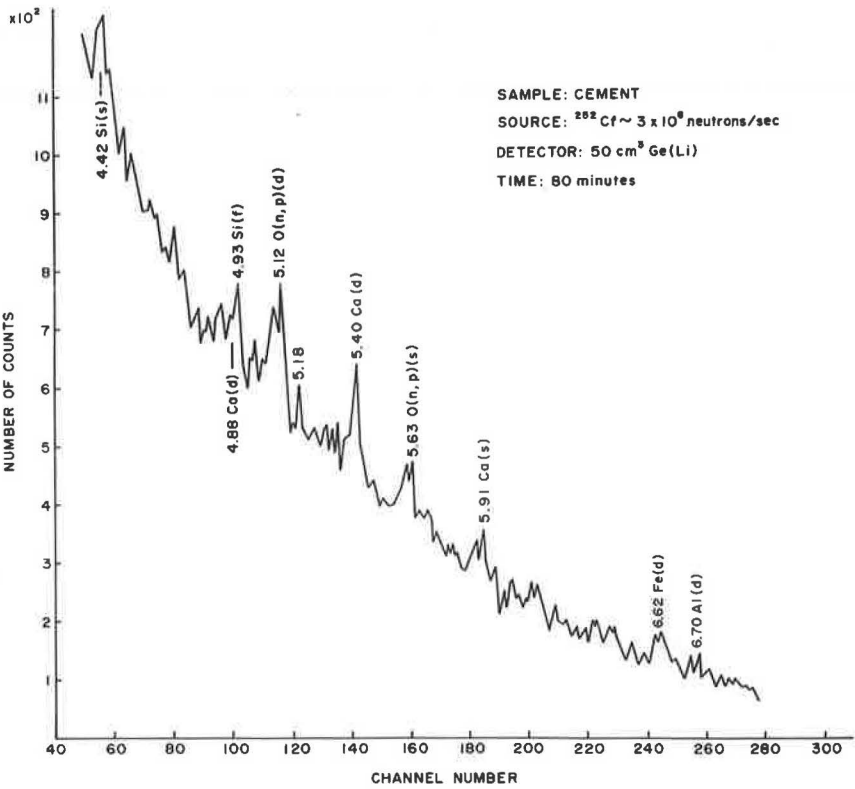


Figure 5. Neutron-capture gamma ray spectrum of limestone (519 grams).

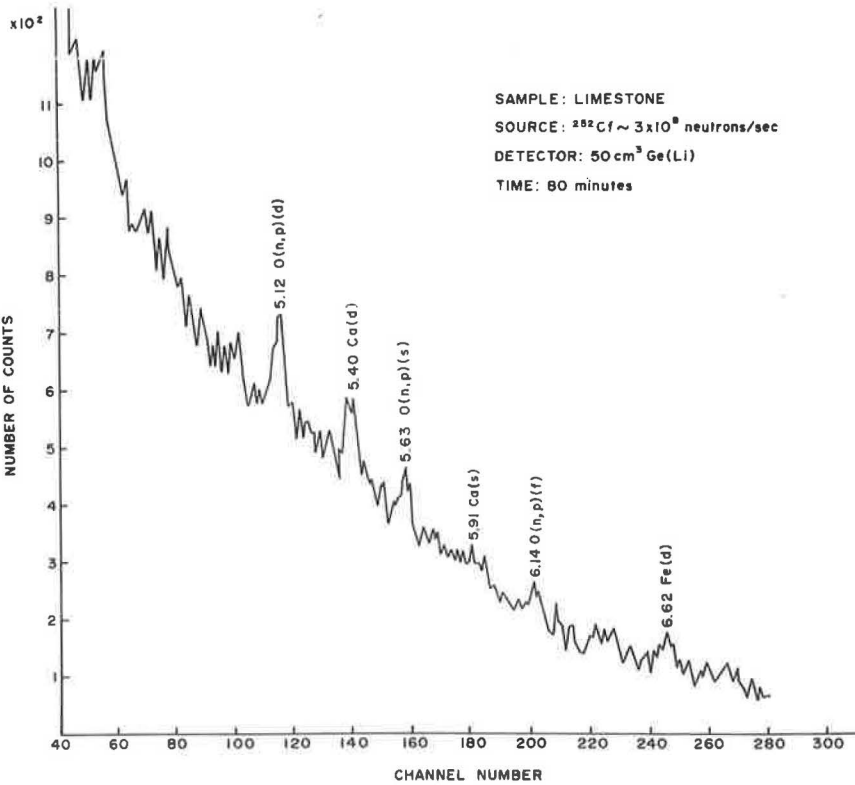


Figure 6. Neutron-capture gamma ray spectrum of concrete with gravel aggregate.

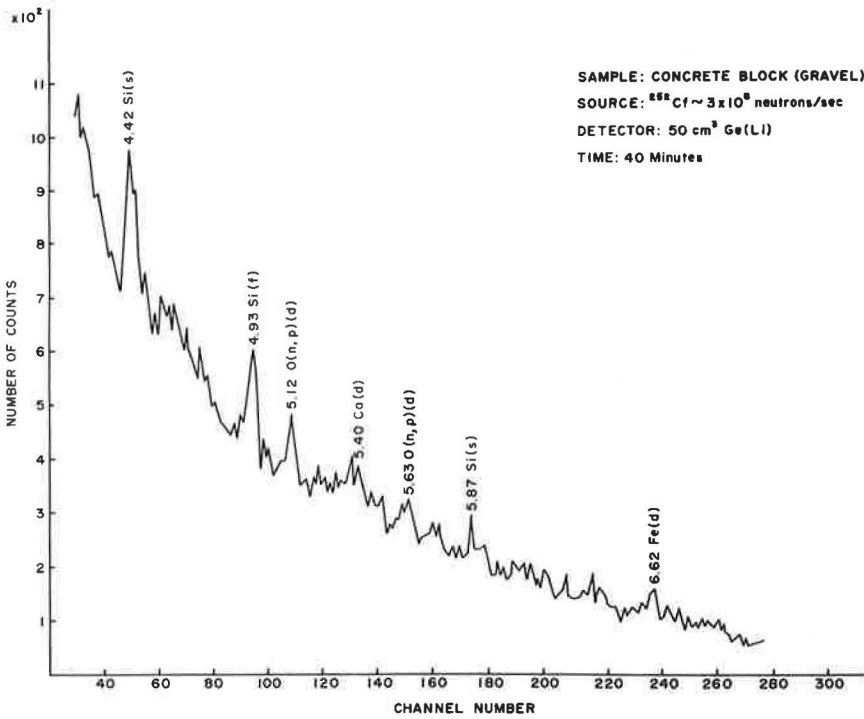


Figure 7. Neutron-capture gamma ray spectrum of concrete with diabase aggregate.

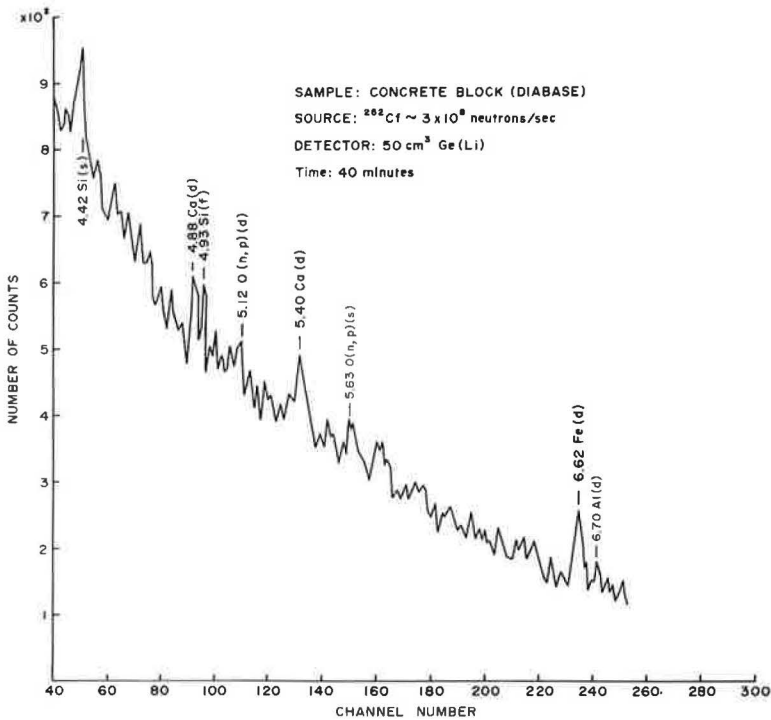


Figure 8. Neutron-capture gamma ray spectrum of concrete with limestone aggregate.

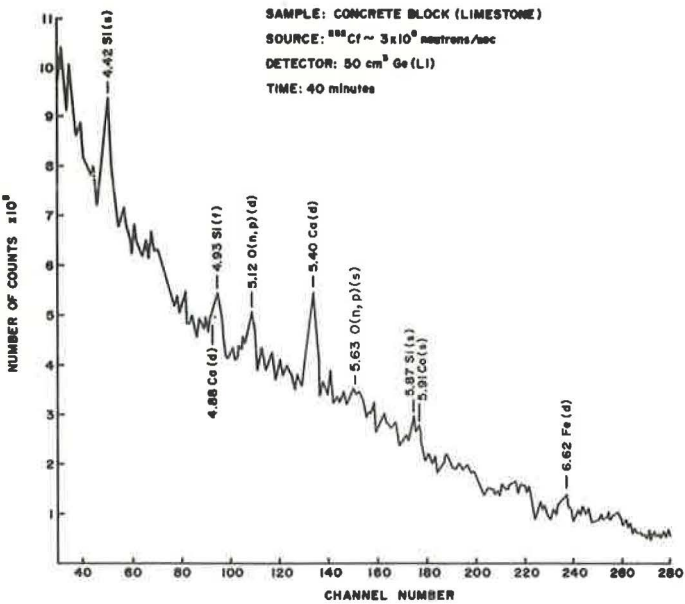
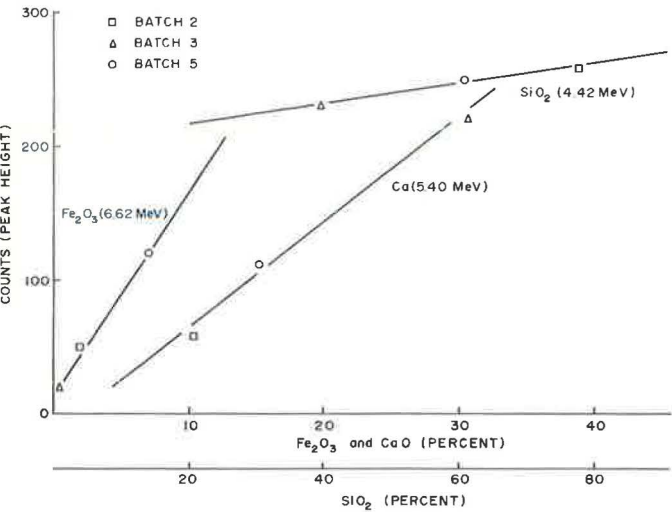


Figure 9. Sensitivity curves for concrete block.



although concrete inspection procedures should, presumably, have already revealed this). As indicated, concrete with the more basic rock aggregates, e.g., traprock of Washington, Oregon, Connecticut, New Jersey, and northern Virginia, should be particularly suitable because of the high iron content.

Monitoring the concrete-mixing process may be an application if the installation is large. The dry raw materials in bins or on conveyors and the wet mixture could be analyzed. However, with the smaller batch plants, the amounts of materials are easily weighed, and the component compositions are generally uniform and are well known; consequently, nuclear analysis does not appear to offer advantages for small operations. Use of the technique for the aggregate preparation processes should be possible; but, as with the smaller plants, there does not appear to be the incentive for such specialized control.

Monitoring the cement process itself by nuclear spectroscopy might be attractive. Here the tonnage is high, and the slow kiln process with sizable inventories necessitates careful control of the streams to ensure a satisfactory cement. The process streams may be in bins and on belts or in water or in air. Monitoring all these streams, as well as the waste streams, to meet environmental restrictions, e.g., for calcium, should be possible by this nuclear method. Checking the gypsum addition by sulfur analysis should also be possible. Concrete involving slag from steel plants might be analyzed for the slag iron and manganese.

In summary, this nuclear spectroscopic scheme appears to offer a new way to determine the components of concrete in situ with almost immediate information. The signals obtained allow process control and the possibility of integrated response and, hence, can yield information on inventories and production.

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