

sidered, the inhibitor-containing concrete is free of corrosion. This is not too unusual because strong concrete will aid the formation of a tight passive film on the steel. In weaker concrete, where some signs of corrosion are apparent even when $\text{Ca}(\text{NO}_2)_2$ is present, inhibited concrete still shows less corrosion than the unprotected concrete.

CONCLUSIONS

Accelerated tests for determining the corrosion susceptibility of iron in concrete, such as

1. Open-circuit-potential measurements in concrete,
2. Polarization measurements in concrete, and
3. Similar electrical measurements in limewater,

show that $\text{Ca}(\text{NO}_2)_2$ offers effective corrosion protection.

Induced electrolysis of concrete is not a reliable technique for studying a corrosion inhibitor when electrolysis of water takes place.

Large deck tests confirm the effectiveness of $\text{Ca}(\text{NO}_2)_2$ as a corrosion inhibitor in concrete after 6 months of daily salting. Because this is an accelerated-test procedure, the use of $\text{Ca}(\text{NO}_2)_2$ as an inhibitor should lead to many years of corrosion-free concrete.

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Measurement of Cement Content by Using Nuclear Backscatter-and-Absorption Gauge

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The delays inherent in current methods for the quality control of portland cement concrete allow large volumes to be placed before problems are discovered. This paper discusses an instrument and a test method for obtaining early quality information: The device is a nuclear backscatter-and-absorption gauge that measures the cement content of plastic concrete. The paper includes a description of the device, a summary of two laboratory evaluation studies, and a discussion of the data obtained by using the gauge on five highway construction projects. Laboratory evaluations established the accuracy of the gauge, its field worthiness and dependence on aggregate composition, and its lack of dependence on concrete density, temperature, and other batching variables. The results showed accuracies of $\pm 13 \text{ kg/m}^3$ ($\pm 22 \text{ lb/yd}^3$) for most siliceous aggregate mixes and $\pm 18 \text{ kg/m}^3$ ($\pm 31 \text{ lb/yd}^3$) for calcareous aggregate mixes. In most cases, nuclear-gauge determinations on the field sites agreed with

calculated cement factors established from batch tickets. Discrepancies encountered on two of the field projects are discussed. The major limitations of the gauge are the necessity for recalibration whenever the aggregate source or the ratio of coarse to fine aggregates is changed and its reduced accuracy for calcareous and certain siliceous aggregates.

The need for early-age composition measurements on portland cement concrete is becoming more and more evident. Reliance on compressive-strength tests made 7 or 28 d after placement can allow large quantities of pavement or structural concrete to be placed before defects are discovered. Accelerated strength tests, which

give results after 24 or 48 h, reduce the delays before problems become known, but even these shorter delays may be very costly. Gravimetric control also has shortcomings; it leaves the quality of the final concrete subject to scale errors, to accidental substitution of incorrect materials (e.g., fly ash for cement), and to other batching and mixing problems.

A rapid field test for the cement content of plastic concrete would allow an earlier assessment of the even-

tual quality of the materials. Several methods (1, 2, 3, 4) have been developed recently for cement-content measurements. One, a nuclear backscatter-and-absorption gauge, is the subject of this paper. The paper includes (a) a description of the device, (b) a summary of the results of laboratory evaluations, and (c) a discussion of some of the data obtained by using the gauge at construction sites.

DESCRIPTION OF DEVICE

The principles underlying the operation of the cement-content gauge are similar to those involved in the widely used nuclear gauges for measuring the density of soils and bituminous pavements. However, the cement-content gauge uses a much lower energy source than does the density gauge and, hence, the chemically sensitive, photoelectric absorption process is the dominant attenuating mechanism for the gamma rays.

Figure 1 shows a cement-content gauge probe immersed in a concrete sample. The probe contains a 14-mCi low-energy (60-keV) gamma-ray source (americium-241) and a radiation detector [a 25-mm (1-in) diameter by 25-mm-long sodium iodide scintillation crystal and a photomultiplier tube]. The detector is shielded on the direct line from the source, so that the only path by which the gamma rays can reach the detector is through the sample. The figure shows typical gamma-ray paths; the gamma rays are both scattered and absorbed by a concrete sample. The amount of absorption depends strongly on the chemical composition of the sample, particularly on the quantities of high-atomic-number (high-Z) elements present. Calcium is generally among the highest Z elements present in significant quantities in concrete; it also occurs in fairly constant amounts in portland cements of various types and sources. Thus, as the proportion of cement in concrete is increased, the number of gamma rays absorbed in the concrete is also increased and the fraction of the original gamma rays that will reach the detector is correspondingly reduced.

Figure 2 shows the components that make up the most recent model of the gauge. These include a polymer-impregnated concrete (PIC) test standard, the probe sitting in a sample holder, and an analyzer. The PIC standard is used to periodically determine a standard count, so that a count-ratio procedure can be used to compensate for changes in the electronics with time and temperature. For testing, the analyzer is connected to the probe by a length of coaxial cable; it is a portable single-channel analyzer whose main function is to count the pulses that arrive from the probe. It also provides the high voltage necessary to operate the photomultiplier tube. The probe and sample holder are shown schematically in Figure 3. The sample holder is a slightly modified 0.03-m³ (1-ft³) unit-weight bucket.

One cement-content determination takes less than 15 min to complete, including the time required to fill the sample holder before the test and to empty and clean it afterwards. After establishing the standard count by using the PIC standard, the sample holder is filled with the fresh concrete sample according to the standard procedure for filling unit-weight buckets (AASHTO T121). A sequence of six 20-s counts is then made by placing the probe at the vertical slot locations 25 mm apart in the sample.

The operator calculates the ratio of the observed sample count (the average of the six readings) to the standard count and reads the corresponding cement content from a previously established calibration curve such as that shown in Figure 4. The calibration curves of count ratio as a function of cement factor are constructed

Figure 1. Configuration of probe and concrete sample.

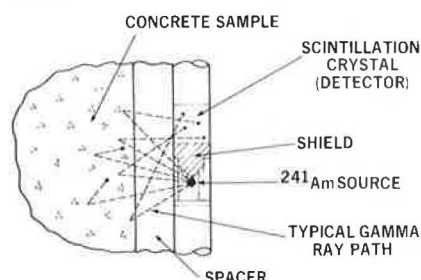


Figure 2. Cement-content gauge: polymer-impregnated concrete test standard, sample holder and probe, and analyzer.



Figure 3. Schematic of sample holder and probe.

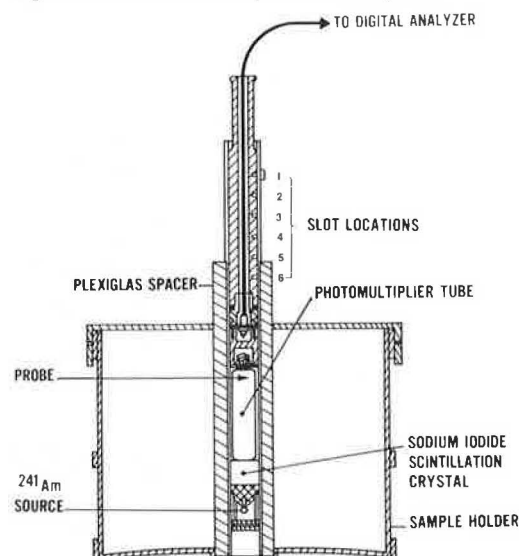


Figure 4. Typical calibration curve (95 percent confidence limits).

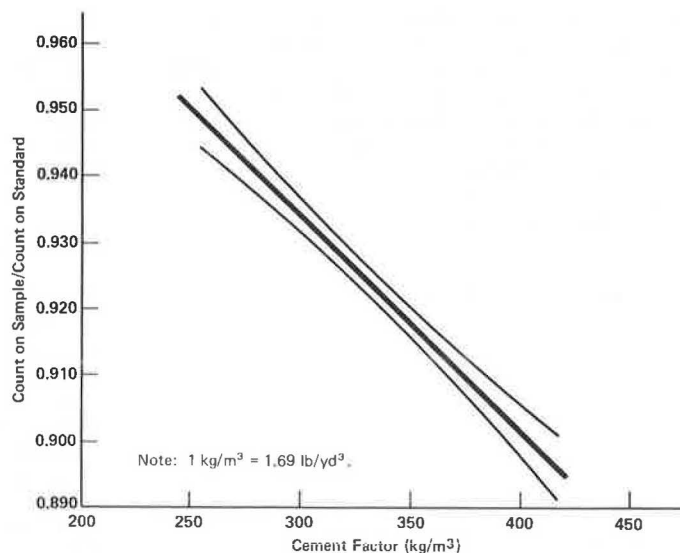
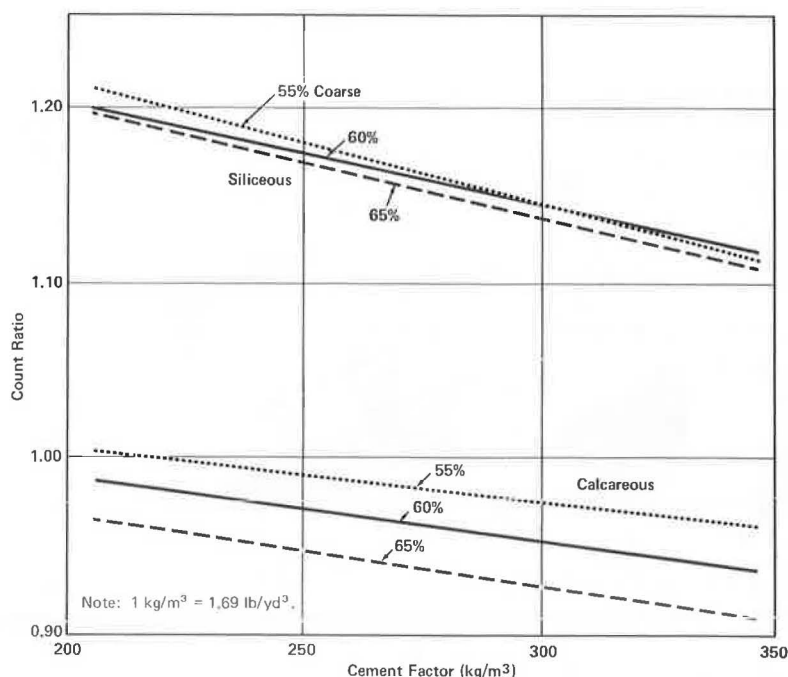


Figure 5. Calibration curves for two aggregates and three ratios of coarse to fine aggregates: state A.



in the laboratory for each concrete job by using small, carefully controlled laboratory batches for which the actual cement factor can be established from the weights of the components.

That the response of the nuclear gauge depends not only on the calcium content but also on all of the elements in the mix (particularly those having a high Z -value) leads to the main limitation on its usefulness; i.e., whenever the aggregate composition is changed significantly, a new calibration curve is required. New calibration curves are required for each distinct aggregate source or combination, for each significant change in the ratio of coarse to fine aggregate when the two sizes are not chemically alike, and (possibly) for within-quarry chemical composition changes in a single aggregate.

Detailed information on the design and operation of the instrument is available in an operating manual (5).

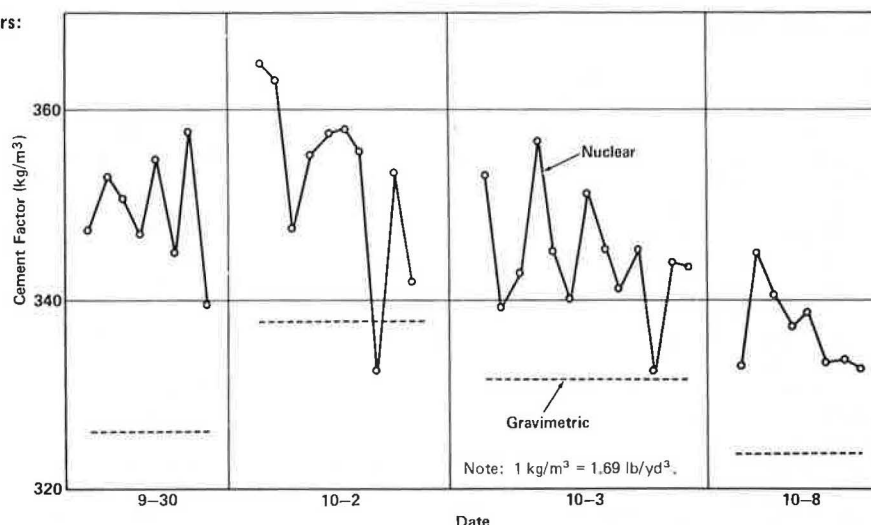
LABORATORY EVALUATIONS

The first of two laboratory evaluations of the cement content gauge was completed at the Federal Highway Administration (FHWA) Fairbank Highway Research Station in 1973 (6).

Two coarse aggregates (25.4-mm maximum size) were used in the main part of the evaluation; one was siliceous (a river gravel) and had very little intrinsic high- Z material and the other was calcareous (a dolomitic limestone) and had more than 20 percent calcium. A siliceous fine aggregate (quartz grains) and a type 1 cement were used throughout. It was anticipated that concretes made with aggregates that had large quantities of calcium and other high- Z elements would have low count rates, reduced sensitivities (changes in count rate per unit change in cement content), and less accurate cement-content determinations.

Tests of mixes made with each of the two aggregates over a range of cement factors [270 to 400 kg/m³ (450

Figure 6. Nuclear and gravimetric cement factors: project B-1.



to 670 lb/yd³)] and densities [2180 to 2400 kg/m³ (136 to 150 lb/ft³)] gave the following results (1 kg/m³ = 1.69 lb/yd³):

Type of Coarse Aggregate	N	Standard Error (kg/m ³)
Siliceous	45	13
Calcareous	90	18

(The standard error is the root-mean-square of the difference between the cement factor as determined by using the nuclear gauge and the actual cement factor of a batch as determined from the weights of the components.)

The effect of varying the proportions of coarse to the fine aggregates when the two components are distinctly different chemically was also noted in this evaluation. Data were obtained by using the calcareous coarse and the siliceous fine aggregates in two different proportions, 65 and 50 percent coarse. When the calibration curve for the 65 percent coarse-aggregate concrete was used to determine the cement content of samples in which this aggregate made up only 50 percent of the total aggregate volume, the resulting errors were found to average about 160 kg/m³ (270 lb/yd³). This shows that a specific calibration curve is required for each ratio of coarse to fine aggregates when the two fractions differ chemically.

Temperature was found to significantly affect the raw gauge counts, but the effect was eliminated when the count-ratio procedure was used with the PIC standard. The gauge geometry is such that its response is relatively independent of the concrete density over the range studied. The response also does not vary with either water or air contents over the usual range of those variables in highway concrete.

A second laboratory evaluation was undertaken in 1976 in state A, and the results were recently published (7). Two coarse aggregates were used, one siliceous (an alluvial gravel) and the other calcareous (a crushed limestone), in combination with a single siliceous fine aggregate and a type 1 cement. The cement factors ranged from 220 to 350 kg/m³ (370 to 590 lb/yd³).

The results of this evaluation are shown below (1 kg/m³ = 1.69 lb/yd³).

Type of Coarse Aggregate	N	Standard Error (kg/m ³)
Siliceous	84	8
Calcareous	84	17

These results also show that there is a considerable loss of accuracy when the aggregate matrix is calcareous; however, it is still possible to make valid measurements on concrete mixes that contain these aggregates (albeit at a lower level of accuracy).

The effect of varying the ratio of coarse to fine aggregates can be seen in Figure 5. Among the siliceous coarse-aggregate samples, the slight differences between the calibration curves suggest that the 60 percent coarse-aggregate curve could be used for samples anywhere in the range of 55 to 65 percent coarse. When this curve was used as the calibration curve for all 84 of the siliceous aggregate samples, regardless of the coarse-aggregate percentage, the standard error was ± 14 kg/m³ (± 23 lb/yd³).

Among the calcareous coarse-aggregate mixes, however, if the 60 percent curve were used for a sample that had 65 percent coarse aggregate, the resulting error in the cement factor would be greater than 70 kg/m³ (120 lb/yd³). This confirms the need for individual calibration curves for different ratios of coarse to fine aggregate. Even when the ratio is held constant, the gauge will be less accurate for calcareous aggregate than for siliceous aggregate mixes because of reduced sensitivity. For the calcareous aggregates, the gauge was approximately 40 percent less sensitive to changes in cement content; i.e., a given change in cement factor of calcareous aggregate mixes produced a 40 percent smaller change in count rate than did the same change in siliceous aggregate mixes.

The state A researchers also reported an overall estimation of the gauge precision: ± 9 kg/m³ (± 15 lb/yd³) for repeated measurements on the same sample. This value varies from material to material and is much higher for calcareous aggregate mixes than for siliceous aggregate mixes.

Thus, the two laboratory evaluations have shown that the nuclear gauge can be used to determine the cement factor of most siliceous aggregate mixes within 13 kg/m³ (22 lb/yd³) of the actual value (65 percent of the time) and of calcareous aggregate mixes within 18 kg/m³ (31 lb/yd³) of the actual value. When the coarse and fine aggregates are very different chemically (e.g., calcareous coarse and siliceous fine), these tolerances will apply only at constant ratios of coarse to fine aggregates. Brief laboratory evaluations by states B and C prior to their field tests yielded standard errors below the limiting values suggested here.

FIELD EXPERIENCE

Field trials of nuclear cement-content gauges were undertaken by states B and C during the 1974 construction season.

In state B, one of the prototype gauges was used on three Interstate projects, two of which involved pavements and the other bridge decks. The results were published in a recent FHWA report (8). All of the aggregates used in these projects were siliceous.

The coarse aggregate used on the first project (project B-1) was a 38.1-mm (1½-in) maximum-size crushed granite, the fine aggregate was a natural sand, and the cement was a type 1. Calibration curves were constructed for this and the other two state B projects in the laboratory. The batching plant was a portable paving plant that had an electronic balance-beam scale with overweight and underweight indicators. Concrete was transported in side dump trucks. Test samples were obtained from the concrete between the spreader and the slip-form paver. Field measurements were obtained on the concrete on 4 different days over a 9-day period.

The results of the 39 test measurements obtained on this project are shown in Figure 6, which also shows the gravimetric cement content for each sample, based on the batch-ticket cement content adjusted by periodic unit-weight measurements. All but one of the 39 nuclear measurements indicated cement factors higher than the gravimetric values; the average difference was 16 kg/m³ (27 lb/yd³).

The data for project B-1 are summarized below (1 kg/m³ = 1.69 lb/yd³):

Project	N	Cement Factor (kg/m ³)		
		Mean Gravimetric	Nuclear	SD of Nuclear
B - 1	39	330	346	9
B - 2	38	329	326	12
B - 3	46	409	403	17

For project B-1, the mean cement factor, as determined by the nuclear gauge, was 346 kg/m³ (583 lb/yd³) with a standard deviation of ±9 kg/m³ (±15 lb/yd³). The design cement factor for this concrete was 320 kg/m³ (540 lb/yd³) but, when adjustments were made for unit weight, the average gravimetric cement factor was 330 kg/m³ (556 lb/yd³). The nuclear data did not indicate any apparent quality-control problems with cement content on this project.

The coarse aggregate on the second pavement project (B-2) was also a 38.1-mm crushed granite, the fine aggregate was a manufactured sand from the same source, and the cement a type 1. The batch plant, transporting vehicles, and sampling procedures duplicated those used on project B-1. Field measurements were made on 3 different days in an 8-day period.

As shown above, the mean cement factor, as determined by the nuclear gauge, was 326 kg/m³ (550 lb/yd³) and the standard deviation was ±12 kg/m³ (±20 lb/yd³). The design cement factor was again 320 kg/m³, but the mean of the gravimetric cement factors was 329 kg/m³ (555 lb/yd³), which is not significantly different from the nuclear-determined values. Again, there were no apparent quality-control problems.

The third project (B-3), several bridge-deck pours, used the same crushed-granite coarse aggregate used in project B-2 and a type 1 cement. The concrete came from a ready-mix plant that had electronic dial scales. The fine aggregate was a blend of a natural sand and the manufactured sand used in project B-2. The blend ratio was initially 80:20 (natural:manufactured) but during the

field testing program was changed to 50:50. The chemical difference between the two fine aggregates necessitated the preparation of a new calibration curve when this ratio change was made. A third calibration curve was prepared later on, when the ratio of the coarse to fine aggregates was changed from 65:35 to 62:38 although, in retrospect, a new calibration curve was not necessary in this case; i.e., there was no significant difference in the calibration curves for these two ratios. Field measurements were made on 7 different days over a 1.5-month period.

For this project, the mean cement factor, as determined by the nuclear gauge, was 403 kg/m³ (679 lb/yd³) and the standard deviation was ±17 kg/m³ (±29 lb/yd³). The design cement factor was 400 kg/m³ (675 lb/yd³), and the mean of the gravimetric cement factors was 409 kg/m³ (690 lb/yd³). The nuclear-gauge results indicate that there were no apparent problems with the quality of the concrete.

To make conclusions about the quality of concrete in the field (in terms of absolute cement-content measurements), the user must rely on the values for the gauge accuracies established in the laboratory where the cement factors of samples are carefully controlled and known. This is also true for other new methods for determining cement content, because there is no reliable standard of comparison in the field. Gravimetric cement factors based on batch-ticket weights with adjustments for unit weight are not good standards for comparison because they are subject to errors in the ticket weights themselves and to a variety of accidents and questionable practices in batching and mixing procedures.

Some of the conclusions that can be drawn about the concrete on the three state B projects from the nuclear-gauge data are very obvious. For example, on all three, the average of the nuclear-determined cement factors exceeded the design cement factor, a good quality sign from the purchaser's viewpoint. As shown by the standard deviations, the variability of the concrete in the ready-mix project (B-3) significantly exceeded the variability of the concrete in the centrally batched projects. (This is not intended as a general conclusion about the relative variability of concrete from the two types of plants, but merely as a statement showing the kind of information the cement-content gauge can provide.) With more data available (e.g., taking several samples from a single batch), mixer evaluations could be undertaken easily with the nuclear gauge.

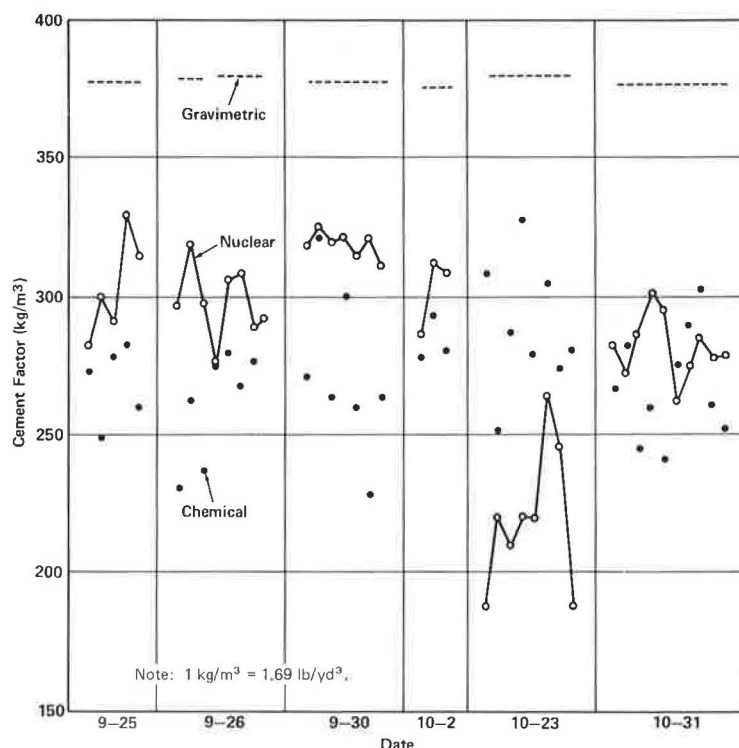
State C used a second prototype gauge on two Interstate projects: The first was an overlay paving project that used a calcareous aggregate concrete, and the second was a bridge deck that used a lightweight aggregate.

On both projects, a chemical method was used to determine the cement content of a portion of the concrete used in each nuclear-gauge measurement. This state uses this method, which is based on SO₃ measurement, for cement-factor determinations on mixes in which the aggregates contain both calcium and silicates because such aggregates make ASTM C85 unreliable. The accuracy of the SO₃ procedure is estimated as ±10 percent.

The coarse aggregate used on the first state C project (C-1) was a 38.1-mm crushed limestone, the fine aggregate was a siliceous sand, and the cement was a type 1. The concrete was supplied from a job-site central-mix plant and transported in side dump trucks to the paving operation. Test samples were taken from the forms after placement by the spreader. Field measurements were made on 6 different days over a 1.5-month period.

The 41 nuclear-gauge-determined, the chemically determined, and the gravimetrically determined cement factors are shown in Figure 7. The chemically determined and the nuclear-gauge-determined cement factors

Figure 7. Nuclear, chemical, and gravimetric cement factors: project C-1.



are well below the gravimetric values. The mean cement factor, as determined by the nuclear gauge, was 283 kg/m^3 (477 lb/yd^3), and the standard deviation was 37 kg/m^3 (63 lb/yd^3). The SO_3 determinations showed a mean cement factor of 291 kg/m^3 (490 lb/yd^3) and a standard deviation of 26 kg/m^3 (43 lb/yd^3). These values contrast with the design cement factor of 362 kg/m^3 (610 lb/yd^3) and the average gravimetric factor of 375 kg/m^3 (632 lb/yd^3).

A number of efforts were made to establish the cause of the difference between the batch-ticket determinations and the results of the two test methods. Such items as the scales and gates at the batch plant were checked and found to be operating satisfactorily. The aggregate stockpiles were resampled, and the nuclear-gauge calibration curve was checked and verified. Flexure test specimens indicated that the concrete met minimum strength requirements. A sizable shift in the ratio of coarse to fine aggregate (63:37 to 58:42) would produce a 90 kg/m^3 (150 lb/yd^3) change in the nuclear readings but not in the SO_3 test results; the possibility of such a change was investigated and ruled out.

Early in 1976, some 18 months after placement, cores were taken from the pavement for further investigation. Three 100-mm (4-in) diameter cores were taken from concrete placed on each of the 4 d when the nuclear gauge had been in used. ASTM C85 was used to establish the cement factors of the cores although, as discussed above, the aggregates used in this concrete are a difficult combination for this test method. The results of this test showed cement factors of 294 to 425 kg/m^3 (496 to 716 lb/yd^3) with a mean value of 346 kg/m^3 (584 lb/yd^3); this was about 30 kg/m^3 (50 lb/yd^3) lower than the gravimetric cement contents, but not as low as the nuclear or chemically determined cement contents. The result of all of these investigations, then, was a stand-off, and the questions remain unresolved. Sizable cement-content deficiencies were indicated by the nuclear and the SO_3 test procedures. At the same time, the ASTM cement-content test showed much smaller deficiencies, and a very thorough examination of the

concrete-plant operations did not locate any cause for the discrepancies.

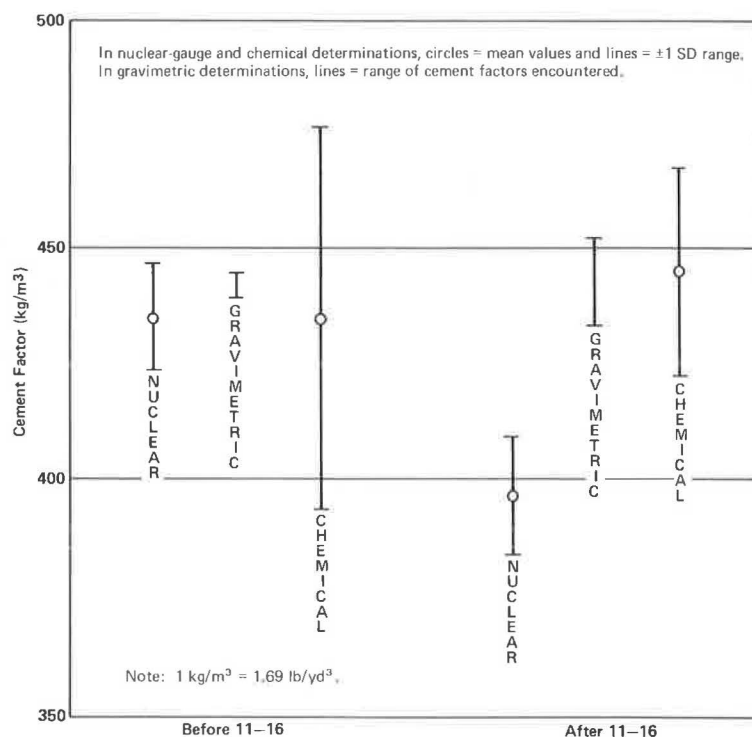
The coarse aggregate used on the second state C project (C-2) was a 38.1-mm lightweight fired slag, the fine aggregate was a siliceous sand, and the cement was a type 2. The lightweight concrete was supplied from a central-mix plant and transported in agitating trucks. The concrete was sampled randomly as it was discharged from the trucks. Cement-factor measurements were made on 9 different days over a 2.5-month period.

A total of 60 cement-factor determinations were made by each of the three methods: nuclear, chemical, and gravimetric. The mean cement factor, as determined by the nuclear gauge, was 411 kg/m^3 (693 lb/yd^3). This was substantially lower than the averages determined by the SO_3 tests [441 kg/m^3 (743 lb/yd^3)] and the gravimetric tests [444 kg/m^3 (748 lb/yd^3)]. However, the nuclear-gauge data also showed that the within-batch SD for three samples from each of the 10 batches was 5 kg/m^3 (8 lb/yd^3); this indicated very good reproducibility of the method with this aggregate.

Further examination of the nuclear results indicated that there was a distinct change (break) in the data midway through the testing. These results are summarized in Figure 8 in which the data are grouped according to these time periods, one the data taken before the change and the other after. For the first 22 samples, i.e., those taken before November 16, 1974, the two test methods were in good agreement with the gravimetric cement factors: The nuclear-gauge-determined values averaged 435 kg/m^3 (733 lb/yd^3). After the break, the average gauge-determined cement factor was 396 kg/m^3 (668 lb/yd^3). The respective SDs of the two groups were 11 and 13 kg/m^3 (19 and 21 lb/yd^3); these values are comparable to those shown elsewhere in this paper for aggregates that work well in the gauge, i.e., noncalcareous aggregates.

Attempts to locate the cause of the shift in nuclear-gauge readings were not successful, although most likely it was an undiscovered change in either the chemical composition of one of the concrete components or in

Figure 8. Distributions of nuclear, chemical, and gravimetric cement factors: project C-2 before and after November 16, 1974.



the nuclear-gauge electronics or geometry. The break did coincide with a large change in air temperature at the construction site, but subsequent laboratory tests, as well as the data developed during the laboratory evaluation in state A, rule temperature change out as the cause. The possibility of a change in the chemical composition of the slag aggregate was rejected when a calibration curve constructed by using aggregates sampled at the end of the project showed no significant difference from the curve constructed before testing began.

The break in the nuclear-gauge data limits any conclusions that can be drawn about the concrete quality on project C-2. However, all of the SD values indicate that the batching and mixing were well controlled.

CONCLUSIONS

1. The nuclear cement-content gauge is suitable for rapid field determinations of the cement content of plastic concrete. Possible applications include mixer studies, troubleshooting, and routine quality control.

2. The gauge measures the cement factors of most siliceous aggregate mixes to within $\pm 13 \text{ kg/m}^3$ ($\pm 22 \text{ lb/yd}^3$) and of calcareous aggregate mixes to within $\pm 18 \text{ kg/m}^3$ ($\pm 31 \text{ lb/yd}^3$) of the true cement content of the sample.

3. The major limitations of the gauge are (a) the necessity for recalibration when the aggregate source is changed or when the ratio of coarse to fine aggregates is changed and (b) its reduced accuracy for calcareous and certain siliceous aggregate concretes.

4. Nuclear-gauge determinations of cement factors agreed with calculated gravimetric cement factors (from batch tickets) on three of the five field projects discussed in this paper and a portion of a fourth. On the fifth project, the nuclear gauge and another cement-content test method showed cement factors that were more than 90 kg/m^3 (150 lb/yd^3) lower than the batch-ticket value.

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