
Draft for Development

Assessment of the composition of fresh concrete

Projet à développer

Estimation de la composition du béton frais

Entwurf zur weiteren Ausarbeitung

Beurteilung der Zusammensetzung von Frischbeton

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This publication is not to be regarded as a British Standard

It is being issued in the Draft for Development series of publications and is of a provisional nature because it is considered that further experience is required in the application of these recommendations before they are put forward as a British Standard. It should be applied on this provisional basis, so that information and experience of its practical application may be obtained. A review of this Draft for Development will be carried out not later than two years after its publication.

Notification of the start of the review period, with a request for the submission of comments from users of this Draft for Development will be made in an announcement in the appropriate issue of *BSI News*. Observations which it is felt should receive attention before the official call for comments will be welcomed.

According to the replies received, the responsible BSI Committee will judge whether the Draft for Development can be converted into a British Standard or what other action should be taken.

Foreword

This Draft for Development has been prepared under the direction of the Cement, Gypsum, Aggregates and Quarry Products Standards Committee.

Over many years there has been an awareness of the benefits that would be derived from the use of a reliable method of analysis of fresh concrete. This has led to the development of several methods of analysis which provide an assessment of the composition of fresh concrete.

Initially, the primary purpose in producing these recommendations was solely to provide full descriptions of some of the available analysis methods to try to ensure uniform application of the procedures. Subsequently, it was felt that there was a risk that the analysis results might be used for compliance purposes without full account being taken of the natural dispersion of the test results, due to the inherent variabilities of production, sampling and testing. As a result of this possibility this Draft for Development comprise four main sections:

Section two. Sampling

Section three. Analysis methods

Section four. Assessment of the accuracy and precision of the analysis methods

Section five. Procedure for assessing compliance

Section two describes the methods of sampling and sample reduction to be used. Section three sets out five methods of analysis for the determination of cement and aggregate content, and two methods of determining water content one of which is included in clause 6. Because all methods are not of equal suitability under all possible conditions, it is essential that the user selects a method suited to his particular concrete and site conditions. To assist in this selection, the description of each method includes a statement of the test principle and indicates the applicability of the method.

Section four deals with the assessment of the accuracy and precision of the analysis methods. It describes how to carry out laboratory tests to assess the accuracy and repeatability of an analysis method, and tests under full scale production conditions to measure the precision of the test methods, and to assess the effect on precision of calibration and fines corrections. This section deals only with repeatability as it is considered that the very large quantity of data required to assess reproducibility would make the procedure unreasonably time consuming.

Section five deals with the procedures for assessing compliance showing how the estimates of accuracy and precision obtained in section four are applied to the test result obtained in section three. Appendix A discusses purchaser's risks. At this stage of the development of the methods it is considered that full account should be taken of all the factors that may have a bearing on the accuracy and precision of the determination and thus the procedures are necessarily complex. It is hoped that use of the methods and procedures for assessment will provide information that will enable future documents to be less complex.

Attention is drawn especially to the need to assess the size of the inherent variabilities of the test and where possible to check that these values do not change significantly during the period of the use of a method at any particular site or location. Users' attention is also drawn to the requirement, in most methods, for calibration tests using samples of raw materials. A test result should not be considered valid for compliance purposes without current data on variability and calibrations.

Draft for Development

Assessment of the composition of fresh concrete

Section one. General

1. Scope

This Draft for Development gives recommendations for the assessment of the composition of fresh concrete. It describes the methods to be used for sampling and sample reduction, five methods of analysing fresh concrete, a method solely for the determination of the water content by drying, methods for the assessment of the accuracy and precision of the five methods of analysis, procedures by which the test results can be used for compliance purposes, and the form of reports to be used.

NOTE 1. The methods do not provide a means of distinguishing between cement, pulverized fuel ash, ground granulated blast-furnace slag and similar materials.

NOTE 2. The titles of the publications referred to in this Draft for Development are listed on the inside back cover.

2. Definitions

For the purposes of this Draft for Development, the definitions in BS 2787 together with the following apply.

2.1 coarse aggregate. Aggregate retained on a 5 mm test sieve complying with the requirements of BS 410.

2.2 fine aggregate. Aggregate passing a 5 mm test sieve complying with the requirements of BS 410.

2.3 fines. Aggregate passing the finest mesh test sieve complying with the requirements of BS 410 used in the particular method of analysis.

2.4 grit. Aggregate passing a 600 μm test sieve complying with the requirements of BS 410 but retained on a 150 μm test sieve complying with the requirements of BS 410.

NOTE. The definition of grit is required only for the purpose of the constant volume (RAM) method of analysis.

2.5 batch. The quantity of concrete mixed in one cycle of operations of a batch mixer, or the quantity of concrete conveyed ready mixed in a vehicle, or the quantity discharged during 1 min from a continuous mixer.

2.6 concrete sample. A sample of concrete of mass equal to that specified in table 1.

2.7 test sample. The portion of the concrete sample that is tested.

2.8 local sample. A sample of either the fine or the coarse aggregate or the cement which is representative of the bulk quantity of these materials that could be used to make a single concrete batch.

2.9 Accuracy and precision

2.9.1 accuracy. The difference between the test results and the true value of the quantity being measured.

2.9.2 precision. The closeness of agreement between test results.

NOTE to 2.9. The terms 'accuracy' and 'precision' refer to different aspects of the variability of analysis results. If a method is used to analyse several samples with the same mix proportions, it may produce cement contents whose average is close to the true content but which are scattered widely about the average; in this case the method would be said to have good accuracy but poor precision. Another method, in the same circumstances, may produce cement contents whose average differs substantially from the true cement content but which are scattered very little about the average; in this case the method would be said to have poor accuracy but good precision.

2.10 bias. The accuracy of a method of analysis measured as the difference between the average of a number of test results and the true value of the quantity being measured, the latter always being subtracted from the former.

2.11 repeatability. The value below which the absolute difference between two test results may be expected to lie with a probability of 95 %, when the two test results are obtained from two test samples obtained from the same sample by the sample reduction procedure described in 3.6, and when the two test results are obtained under the same conditions (same operator, same apparatus, same laboratory, and within a short period of time).

NOTE. Usually the term 'repeatability' is used to refer to the size of the differences that may be expected to occur between tests obtained on identical samples under identical conditions. With concrete, identical samples do not occur in practice, so it is considered more appropriate to define repeatability in such a way as to include the variations that arise in preparing the two test samples from a sample. Repeatability defined in this way can be applied directly to test results obtained on site to assess the performance of a method of analysis and its operator.

Section two. Sampling

3. Sampling and sample reduction

3.1 General. For the test results to be of real value, it is important that the necessary attention be paid to the procedure by which the initial bulk sample is taken from the concrete to be examined. Similarly, the sample reduction process, which is required to provide a test sample of the appropriate size, can be a source of variability if the specified procedure is not followed carefully.

3.2 Samples for analysis. Obtain the test samples for analysis and water content from a sample taken for that and no other purpose.

NOTE. Where it is required that the constituents, found by analysis, are to be expressed in kg/m^3 , it will be necessary, at the same time, to take a sample for the determination of the mass per cubic metre of the fully compacted fresh concrete.

3.3 Apparatus. The following apparatus is required.

3.3.1 A *scoop*, of galvanized iron or other suitable material, that when just filled will contain about 4 kg of concrete.

3.3.2 *Sample containers* of appropriate size and suitable material, e.g. 15 L* plastics buckets.

3.3.3 A *sampling tray or plate* of non-absorbent material and of a suitable size, e.g. a tray 1.2 m $\times$ 1.2 m $\times$ 50 mm deep made from 1.6 mm minimum thickness of steel sheet galvanized.

3.3.4 A *square-mouthed shovel*, size 2, complying with BS 3388.

3.4 Sampling from bulk quantities of concrete. Using the principles described in BS 1881 : Part 101, take, with the scoop, the number of increments of concrete necessary to provide a sample of the appropriate mass given in table 1. Ensure that the scoop contains about 4 kg when each increment is taken.

3.5 Certificate of sampling. Each sample should be accompanied by a certificate of sampling, completed by the person responsible for supervizing the sampling, confirming that the sample was taken in accordance with these recommendations.

The certificate should also provide the following information:

- (a) identification of the sample;
- (b) date and time of mixing;
- (c) time of sampling;
- (d) location of sampled batch after placing.

3.6 Sample reduction to provide the test samples

3.6.1 General. The following description of the sample reduction procedure refers to concrete of low workability, but the general approach should be adopted also when subsampling all concretes. For more workable concretes, the terms 'heap' and 'cone' should not be applied rigidly.

3.6.2 Coning. Place the concrete sample in a heap on one side of the tray or plate. Then, shovel-by-shovel, transfer the concrete to the opposite side of the tray to form a cone; do this by depositing the second and each subsequent shovelful of concrete on the apex of the cone being formed so that the portions which slide down the sides do not displace the centre of the cone to any marked extent. Cone the concrete three times.

3.6.3 Quartering. Flatten the third cone by repeated vertical insertions of the shovel across the apex of the cone, lifting the shovel clear after each insertion. Ensure that the flattened heap is reasonably uniform in thickness and diameter. Quarter along two diameters, intersecting at right angles, using the shovel. Combine opposite quarters to form two separate subsamples.

3.6.4 Test samples for analysis. Treat each subsample separately to obtain two test samples, i.e. one from each subsample.

*'L' is now used as the symbol for litre.

Reduce the subsamples by further coning and quartering, and putting aside (see 3.6.5) two opposite quarters until the two remaining quarters are of the required mass to provide a test sample (see table 1).

Table 1. Sample and test sample masses

Analysis method	Sample mass	Test sample mass
	kg	kg
Buoyancy	32 ± 4	$4 \pm 0.5^*$
Chemical	20 ± 4	2.5 ± 0.5
Constant volume	32 ± 4	8 ± 1
Physical	32 ± 4	4 ± 0.5
Pressure filter	24 ± 4	3 ± 0.5

* 8 ± 1 kg when 40 mm aggregate present in the concrete.

3.6.5 Test samples for water content. When a direct determination of water content is required, retain the concrete that would be put aside after the final quartering to provide the test sample for analysis (see 3.6.4). Ensure, as far as possible, that no water is lost through evaporation or any other cause; proceed as described in clause 10 or, if the chemical (GLC) method of analysis is being used, as described in clause 6.

NOTE. A further sample of the concrete is required, when the chemical method is being used, for a chloride 'blank' determination.

Section three. Analysis methods

4. General

Each of the five analysis methods described in this section has been developed to a stage where the test results have been found to be of value in the estimation of the composition of concrete mixes; this does not mean that the methods are of equal suitability or accuracy over the whole range of concretes that may be met. In practice, the suitability of an analysis method may depend on the physical or chemical properties of the cement and/or the aggregate, but, provided an appropriate analysis method has been selected and the specified procedure followed with the required care, it should be possible to derive useful data about the concrete analysed.

Since the test results generally depend on calibration tests, the raw materials used in these tests need to be truly representative of the materials actually incorporated in the concrete to be analysed. It could be that the raw materials are extremely uniform over an appreciable period of time but this should not be assumed without evidence to show that this is the case. It is because of this uncertainty about the uniformity of the raw materials that no blanket recommendation is given about the frequency of calibration. Any test result should be considered suspect if the calibration data are not relevant to the actual test sample.

Because of the ever present possibility of either operator error or apparatus malfunction, the tests should be carried out in duplicate.

The analysis should start as soon as possible after the addition of water to the cement. Provided the aggregate can be washed free of cement during the normal analysis procedure, the accuracy of the test should not suffer.

5. Buoyancy (BS 1881) method

5.1 Test principle and applicability of the method

5.1.1 Principle. A test sample obtained in accordance with 3.6.4 is weighed in air and in water, and then washed over a nest of sieves to separate the cement and fines passing a 150 μm test sieve complying with the requirements of BS 410 from the aggregate. The washed aggregate is weighed in water and the proportions of cement, coarse and fine aggregates and water are then calculated. The addition of a chemical substance to disperse air is used when air-entrained concrete is to be analysed.

5.1.2 Samples of raw materials required. Samples of coarse and fine aggregates, typical of those used in the concrete to be analysed, are required for the determination of the proportion of the aggregate that passes a 150 μm test sieve complying with the requirements of BS 410, and for the determination of their relative densities. Where Portland cement is not used, a sample of the cement, typical of that used in the concrete to be analysed, is required for the determination of its density. For ordinary Portland cement, the density is assumed to be 3130 kg/m^3 .

5.1.3 Applicability of the method. The method is suitable for the determination of cement content, coarse and fine aggregate contents, and the water content of all concretes. In the analysis, cement and water contents are determined by difference and their accuracies will be affected by any errors in carrying out the test procedure.

The test results are sensitive to any variation between the aggregate samples obtained for the calibration tests and the actual aggregates used in the concrete to be analysed.

5.2 Reagent. The following reagent is required for air-entrained concrete only.

Tri-n-butyl phosphate, laboratory reagent grade.

5.3 Apparatus. The following apparatus is required (see figure 1).

5.3.1 Balance of suitable type to suspend the containers described in 5.3.3, capable of weighing 5 kg to an accuracy of ± 0.5 g.

5.3.2 Two counterpoises for the balance, one to obtain equilibrium when an empty container is weighed in air and the other when one of the empty containers is weighed in water.

5.3.3 Bucket shaped containers made of corrosion resisting metal, each 200 ± 5 mm diameter at the top, and 180 ± 5 mm deep, having smooth sloping sides and a rounded bottom to prevent the trapping of air during immersion.

NOTE. It is advantageous that eight containers are available and that each container is of the same mass in air correct to ± 0.25 g, and each is clearly marked with the difference between its loss in mass when immersed in water and the mass of the second counterpoise.

5.3.4 Immersion apparatus. A main tank of approximately 280 mm diameter and approximately 300 mm deep, fitted with an overflow spout such that the lip of a

container hung from the balance is completely immersed when the main tank is filled to overflowing.

A tap of 6 mm bore is fitted to the main tank and connected by a flexible pipe to a supply of water, e.g. a subsidiary tank as shown in figure 1.

This connection enables the bulk of the water in the main tank to be dropped to below the lip of a container suspended from the balance; alternatively, a suitable bailing vessel should be provided for lowering the water level.

A baffle plate is fitted inside the main tank, such that any flow of water into the main tank does not disturb the water and, therefore, the contents of the container.

5.3.5 Two nesting sieves, approximately 450 mm diameter by approximately 300 mm deep, the upper sieve being fitted with perforated plate of 5 mm aperture size, and the lower sieve being fitted with woven wire cloth of 150 μm aperture size; both meshes complying with the requirements of BS 410.

5.3.6 Means of transferring the washed aggregate from the sieves to the containers, e.g. a funnel 500 mm diameter at the top, 150 mm diameter at the bottom and 260 mm deep, supported in a suitable frame (see figure 1).

5.3.7 Hose fitted with a nozzle that, when fed with water at normal mains pressure, gives a fine spray strong enough to move the particles of fine aggregate over the surface of the 150 μm sieve.

5.3.8 Metal stirring rod of sufficient length and strength, e.g. 450 mm long by 16 mm diameter, for the operator to be able to stir a concrete sample in its container.

5.4 Calibration tests

5.4.1 General. Determine the density of the cement (see 5.4.2), if Portland cement is not used, and the relative densities of the coarse and fine aggregates (see 5.4.3). Determine also a grading correction factor to allow for that fraction of the coarse and fine aggregate, used in the concrete, that passes a 150 μm sieve (see 5.4.4).

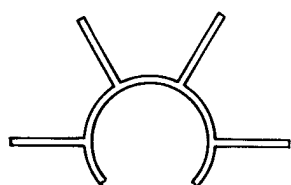
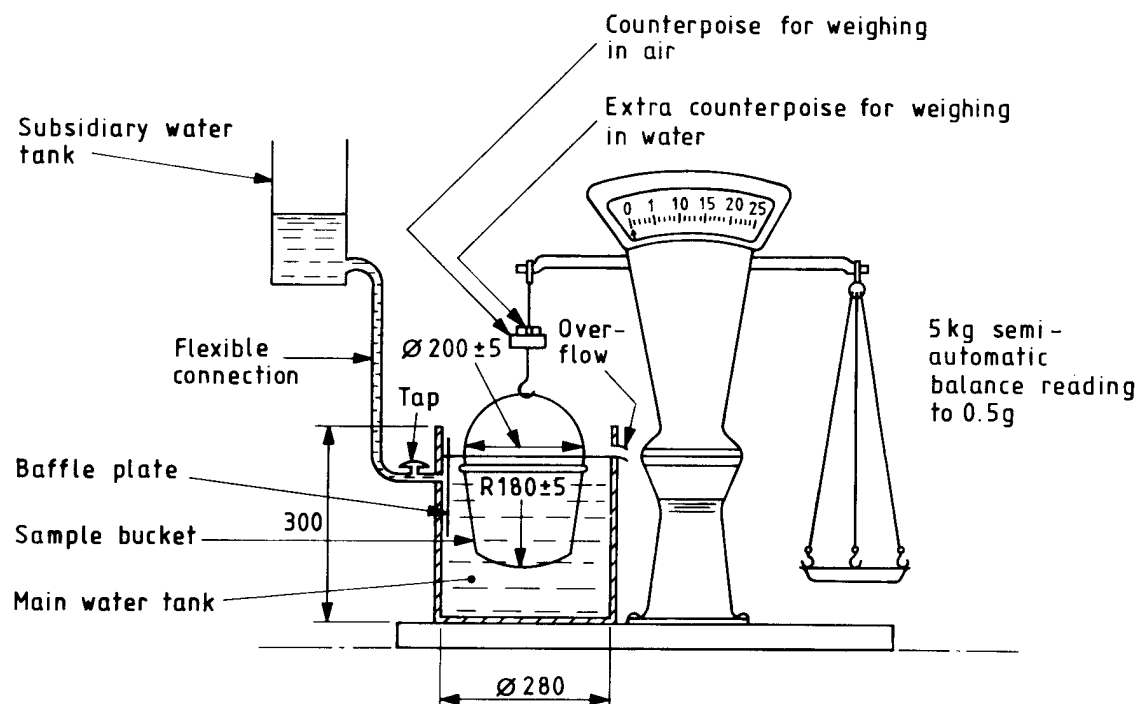
NOTE. The accuracy of the analysis results depends to a great extent on the reliability of the estimates obtained from the above determinations. It cannot be assumed that the results of the calibration tests will remain constant.

5.4.2 Density of the cement. Assume that the density of the cement, if Portland cement is used, is 3130 kg/m^3 ; if other cements are used, determine their densities by the method described in BS 4550 : Section 3.2.

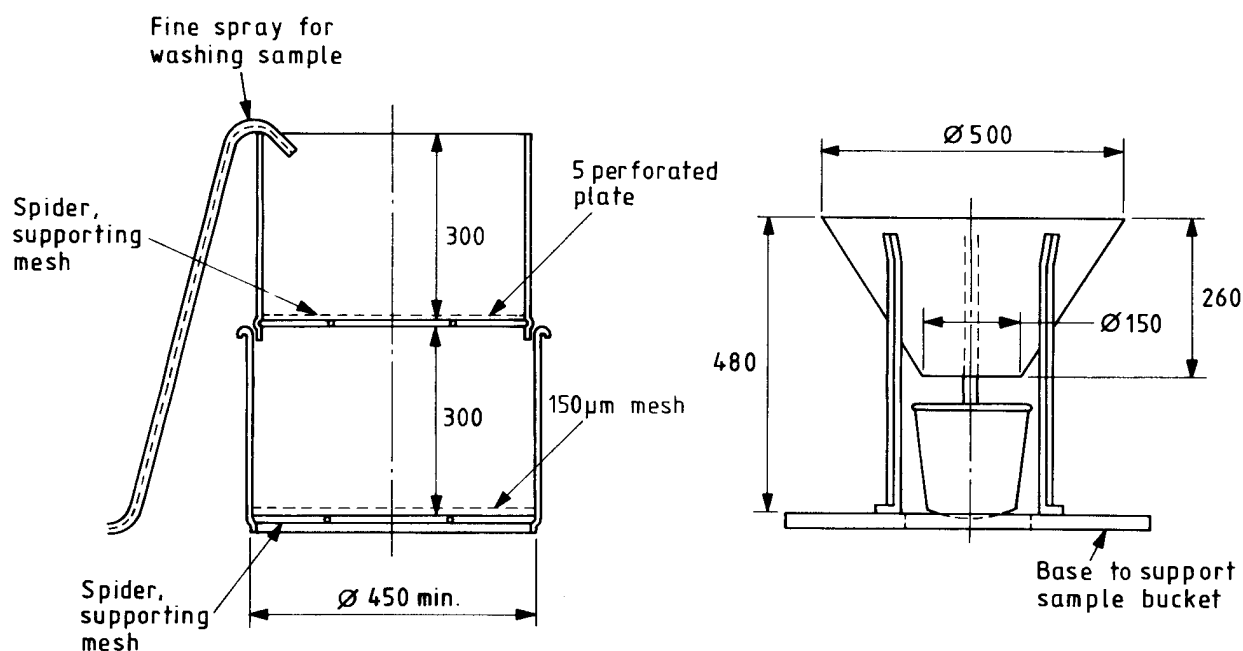
5.4.3 Determination of the relative densities of the aggregates

5.4.3.1 Aggregate samples. Obtain bulk samples, from each of the stockpiles of the coarse and fine aggregates to be used in the concrete to be analysed, by the appropriate method specified in BS 812 : Part 1.

5.4.3.2 Subsamples of aggregates. Assuming a concrete test sample mass of 4 kg, calculate from the expected mix proportions the saturated surface dry (SSD) mass of each individual size and type of aggregate in such a sample. Then reduce each bulk sample of aggregate by the methods described in BS 812 : Part 1 to provide a subsample containing aggregate of between one and two times the appropriate calculated SSD mass.



Plan of spider to support mesh



All dimensions are in millimetres.

Figure 1. Apparatus for buoyancy methods

Take subsamples of the coarse aggregate fractions, making any necessary allowance for the free water in the aggregate, to provide a composite coarse aggregate sample of the mass previously calculated for a 4 kg sample of concrete. Then, separately, repeat this operation for the fine aggregate.

5.4.3.3 Procedure. Determine the relative densities of the coarse and fine aggregates under conditions identical with those to be applied when the concrete is to be analysed.

Do not permit any differences in the water temperature, at the time of making any weighings under water, to exceed 2 °C.

NOTE 1. The water temperature needs to be maintained within the same range when carrying out the analysis.

Treat the samples in pairs, one of coarse aggregate and one of fine aggregate, and carry out the following procedure on each of the pairs.

Place a sample of the fine aggregate in one of the clean containers. Support the 5.0 mm sieve on the funnel, with the container and fine aggregate beneath the funnel. Totally transfer the paired sample of coarse aggregate to the sieve. Then wash the coarse aggregate under a spray of water for 1 min to 2 min to ensure the removal of particles finer than the 5.0 mm sieve, collecting this fraction in the container with the fine aggregate.

Totally transfer the washed coarse aggregate to a clean container using the funnel, then fill the container to within 25 mm of the lip. Stir the aggregate until any entrapped air is removed, i.e. for about 1 min, and hang the container, in the water tank, from the balance (see figure 1). Steadily raise the water level in the main tank, which is initially below the lip of the container, until the container is filled with water and water starts to flow from the overflow spout. Weigh the coarse aggregate in water.

Follow the same balancing and weighing procedures with the container holding the fine aggregate.

NOTE 2. During all weighings, limit the maximum movement of the container to 5 mm to restrict the error caused by variations in its displacement.

If the aggregates have been oven dried, time will be required for water to be absorbed by the aggregates before a steady mass in water is obtained. In such a case, stir the aggregates, and weigh after 10 min immersion to ensure that it does not differ by more than 0.5 g from the previous weighing. If necessary, stir and reweigh the container and contents at 10 min intervals until the above condition is met. Record the time taken to obtain this condition.

Record the final mass in water as B_a for the coarse aggregate, and B_s for the fine aggregate.

Carefully drain the water from each container, if necessary filtering the water from the fine aggregate through a filter paper and returning any retained material. Dry the coarse and fine aggregates separately to the saturated surface dry condition (see BS 812 : Part 2).

Transfer the coarse aggregate onto a dry cloth and surface dry it. Continue the process with a second dry cloth when the first will remove no further moisture. On the second cloth, spread the aggregate out not more than one stone

deep and leave it until the aggregate just becomes surface dry.

NOTE 3. A source of heat, e.g. a fan heater of low wattage, may be used to assist drying provided no internal water is removed.

Expose the fine aggregate to a gentle current of warm air to evaporate the surface moisture; stir the aggregate at frequent intervals to ensure uniform drying. Continue these processes until the fine aggregate just attains a free-running condition (see BS 812 : Part 1 for further guidance).

Record the mass in air of the SSD aggregates as A_a for the coarse aggregate and A_s for the fine aggregate. Retain the fine aggregate for the determination of the grading correction factor (5.4.4).

5.4.3.4 Calculation of relative densities. Calculate the relative densities as follows:

$$\text{relative density of coarse aggregate} = \frac{A_a}{A_a - B_a}$$

$$\text{relative density of fine aggregate} = \frac{A_s}{A_s - B_s}$$

where A_a , A_s , B_a , B_s are as defined in 5.4.3.3.

5.4.3.5 Duplicate determination. Repeat the operations, starting with the subsamples (see 5.4.3.2), three more times; then calculate the average values of the relative densities of the coarse and fine aggregates.

5.4.4 Determination of grading correction factor

5.4.4.1 Test samples. Use the same test samples, that had previously been used to determine the relative density of the fine aggregates, for the determination of the grading correction factor.

5.4.4.2 Procedure. Totally transfer a fine aggregate sample to the 150 µm sieve. Wash the fine aggregate under the spray until the water passing through the sieve is clear, i.e. about 10 min. Wash the aggregate retained on the sieve into a clean container through the funnel. Cover the aggregate with water to within 25 mm of the lip of the container and stir to remove entrapped air. Immerse the container in the water tank and determine the mass in water of the fine aggregate D_s as in 5.4.3.3.

Calculate the grading correction factor C_s from the equation:

$$C_s = \frac{B_s}{D_s}$$

where B_s is as defined in 5.4.3.3.

Repeat this operation three more times and calculate the average of the four estimates to provide the value to be used in the calculation of the mass of the constituents.

5.5 Analysis of concrete

5.5.1 Sampling. Take a sample by the appropriate method described in clause 3.

5.5.2 Subsample for analysis. Reduce the bulk concrete sample, using the procedure described in 3.6, to provide samples of 8 ± 1 kg for concrete with aggregate of 40 mm down and 4 ± 0.5 kg for concrete with aggregate of 20 mm down.

Divide the 8 kg samples to provide two samples of approximately 4 kg, each to be treated separately.

NOTE. It will be necessary to add together the appropriate mass of each constituent to calculate its total mass, for the purpose of 5.5.4.

5.5.3 Procedure. Place a test sample, obtained as in 5.5.2, in a clean, dry container and determine its mass in air W .

Fill the container with water at the required temperature (see 5.4.3.3) to within 25 mm of the lip. At this stage, add 10 ml of tri-*n*-butyl phosphate if air-entrained concrete is being analysed. Stir the contents of the container until as much entrapped air, and for air-entrained concrete as much entrained air as possible, is removed, i.e. for 1 min to 2 min. Hang the container in the water tank, from the balance (see figure 1), taking all necessary care to prevent the container lip from falling below the water level in the tank. Then carefully fill the container with water to the lip and allow the contents to settle for 5 min. Adjust the water level in the tank, without disturbing the material in the container, until the water just overflows from the spout. Do not add more water to the tank and, when overflow has ceased, determine the sample mass in water w .

Change the water in the tank, if it becomes contaminated, to prevent a change in its density.

Totally transfer the sample to the nest of sieves, 5 mm over the 150 μm , and wash the concrete until it is free from cement, i.e. for at least 2 min. Transfer the clean coarse aggregate from the 5 mm sieve to a clean container through the funnel. Fill the container with water to within 25 mm of the lip and stir the contents to remove entrapped air. Immerse the container in the water and determine the mass in water of the coarse aggregate w_a .

Wash the fine aggregate on the 150 μm sieve for a further 10 min or until it is free of cement, and the washing water is clear. Determine the mass in water of the fine aggregate w_s by the same procedure that was used for the coarse aggregate.

Repeat the above procedure with the second test sample.

5.5.4 Calculation of mass of each constituent. Calculate the mass of each constituent, for each test sample, as follows:

(a) the mass of coarse aggregate W_a in the sample is given by the equation:

$$W_a = w_a \times F_a;$$

(b) the mass of fine aggregate W_s in the sample is given by the equation:

$$W_s = w_s \times F_s \times C_s;$$

(c) the mass of cement W_c in the sample is given by the equation:

$$W_c = [w - w_a - (w_s \times C_s)] \times F_c;$$

(d) the mass of water W_w in the sample is given by the equation:

$$W_w = W - (W_a + W_s + W_c).$$

where

$$F_a = \frac{\text{relative density}}{\text{relative density} - 1} \text{ for the coarse aggregate;}$$

$$F_s = \frac{\text{relative density}}{\text{relative density} - 1} \text{ for the fine aggregate;}$$

$$F_c = \frac{\text{density}}{\text{density} - 1000} \text{ for the cement;}$$

C_s is the grading correction factor as found in 5.4.4;

W is the mass of concrete sample in air;

w is the mass of concrete sample in water;

w_a is the mass of coarse aggregate in water;

w_s is the mass of fine aggregate in water.

NOTE. As the mass of water calculated above will reflect any errors in the determinations of W_a , W_s , and W_c , for a higher degree of accuracy it may be advisable to estimate the mass of water by the method described in clause 10.

5.6 Mass of constituents of the concrete

5.6.1 Determination of mass per cubic metre of fresh concrete. On a separate sample, taken at the same time and by the same procedure as the sample for analysis, determine the mass per cubic metre of the fully compacted fresh concrete using the method described in BS 1881.

5.6.2 Calculation of mass of each constituent per cubic metre of concrete. The mass of each constituent per cubic metre of concrete (in kg/m^3) is equal to:

$$\frac{\text{mass of constituent}}{\text{mass of test sample}} \times \text{mass per cubic metre of fresh concrete}.$$

5.7 Repeatability. If the difference between the two masses of cement per cubic metre determined on the duplicate test samples exceeds 20 kg/m^3 , discard the test results.

If the difference between the two masses of water per cubic metre determined on the duplicate test samples exceeds 0.5 %, discard the test results.

5.8 Report. The data to be included in a test report are given in clause 11.

6. Chemical (GLC) method

6.1 Test principle and applicability of the method

6.1.1 Principle. A concrete test sample is weighed and then washed over a nest of sieves to separate the material finer than a 300 μm test sieve complying with the requirements of BS 410, i.e. cement and fines. A subsample of the cement and fines, which is held in suspension in the washing water, is treated with nitric acid and the concentration of calcium determined using a flame photometer. The water content of the concrete is determined by estimating the dilution of a standard sodium chloride solution caused by the free water in the sample. A siphon can is used to determine the coarse aggregate content, and the fine aggregate content is determined by difference; the latter determination will be affected by any errors in the analysis.

6.1.2 Samples of raw materials required. Samples of the cement and the coarse aggregate, typical of those used in the concrete to be analysed, are required; the cement for calibration of the flame photometer, and the coarse aggregate for the calibration of the siphon can.

6.1.3 Applicability of the method. This method of analysis was developed primarily for the determination of the cement and water contents of concrete samples and it is well suited for this purpose. The test for cement content is not suitable when the aggregate contains

calcareous material finer than material passing a 300 μm test sieve complying with the requirements of BS 410.

6.2 Reagents. Use reagents of the specified analytical reagent quality. Use water complying with the requirements of BS 3978 and from the same source throughout a series of analyses.

6.2.1 Reagents required for the determination of cement content

6.2.1.1 Calcium carbonate, analytical reagent grade.

6.2.1.2 Nitric acid solution, approximately 1.6N. Dilute 100 mL of nitric acid (70 % (m/m), $\rho = 1.42 \text{ g/mL}$) with distilled water and make up to 1000 mL.

CAUTION. Nitric acid is harmful to the skin and if taken internally.

6.2.2 Reagents required for the determination of water content

6.2.2.1 Nonyl alcohol, general purpose reagent grade.

6.2.2.2 Silver nitrate, standard volumetric solution, 0.5N*. Dissolve 84.9 g of analytical grade silver nitrate in distilled water and make up to 1000 mL.

CAUTION. Silver nitrate is harmful to the skin and if taken internally.

6.2.2.3 Sodium chloride, standard volumetric solution, 0.5N*. Dissolve 29.25 g of analytical grade sodium chloride in distilled water and make up to 1000 mL.

6.2.2.4 Potassium thiocyanate, standard volumetric solution, 0.05N*. Dissolve 4.85 g of analytical grade potassium thiocyanate (anhydrous) in distilled water and make up to 1000 mL.

6.2.2.5 Nitric acid solution, approximately 8N. Dilute 500 mL of nitric acid (70 % (m/m), $\rho = 1.42 \text{ g/mL}$) with distilled water and make up to 1000 mL.

6.2.2.6 Iron (III) ammonium sulphate, 100 g/L solution. Dissolve 100 g of general purpose reagent grade iron (III) ammonium sulphate in distilled water and make up to 1000 mL.

6.2.2.7 Potassium chromate, 500 g/L solution. Dissolve 50 g of general purpose reagent grade potassium chromate in distilled water and make up to 1000 mL.

6.2.2.8 Iron (III) ammonium sulphate solution, saturated.

6.3 Apparatus

6.3.1 Apparatus required for the determination of cement content

6.3.1.1 Washing machine, single tube with submerged impeller and recirculating pump approximate capacity 40 L.

6.3.1.2 Sieves, of 300 mm diameter; one fitted with perforated plate of 5.0 mm aperture size, and the other fitted with woven-wire cloth of 300 μm aperture size, both meshes complying with the requirements of BS 410.

6.3.1.3 Balance capable of weighing up to 5 kg to an accuracy of $\pm 1.0 \text{ g}$.

6.3.1.4 Balance capable of weighing up to at least 160 g to an accuracy of $\pm 0.001 \text{ g}$.

6.3.1.5 Sampling tower, see figure 2.

6.3.1.6 High-speed stirrer, electrically driven with a no-load speed of 7000 r/min and a stainless steel cup of 500 mL capacity.

6.3.1.7 Flame photometer.

6.3.1.8 Gas supply, natural or bottled.

6.3.1.9 Source of compressed air.

6.3.1.10 Container, 5 L capacity, for dilute nitric acid.

6.3.1.11 Graduated cylinder, 10 mL capacity, complying with the requirements of BS 604.

6.3.1.12 Graduated cylinder, 250 mL capacity, complying with the requirements of BS 604.

6.3.1.13 Two volumetric flasks, 1 L capacity, complying with the requirements of class B of BS 1792.

6.3.1.14 Twelve volumetric flasks, 100 mL capacity, complying with the requirements of class B of BS 1792.

6.3.1.15 Glass beaker, 250 mL capacity.

6.3.1.16 Two watchglasses, of 75 mm diameter.

6.3.1.17 Burette, 50 mL capacity, complying with the requirements of class B of BS 846.

6.3.1.18 Well ventilated oven, controlled at a temperature of $105 \pm 5^\circ\text{C}$.

6.3.1.19 Steel drying pans, of 300 mm diameter.

6.3.1.20 Drainage, including a settling tank, suitable to cope with large volumes of waste water containing cement in suspension.

6.3.2 Apparatus required for the determination of water content. The following apparatus additional to that given in 6.3.1 is required for the determination of the water content.

6.3.2.1 Mechanical shaker, suitable for agitating water and concrete samples together in bottles (see 6.3.2.2).

6.3.2.2 Two metal or plastics bottles, 2.5 L to 5 L capacity, fitted with effective closures.

6.3.2.3 Dispenser, 2.5 mL capacity.

6.3.2.4 Dispenser, 2.0 mL capacity.

6.3.2.5 Dispenser, 5.0 mL capacity.

6.3.2.6 Dispenser, 10.0 mL capacity.

6.3.2.7 Two pipettes, 50 mL capacity, complying with the requirements of class B of BS 1583.

6.3.2.8 Two burettes, 50 mL capacity, complying with the requirements of class B of BS 846.

6.3.2.9 Four conical flasks, 500 mL capacity.

6.3.2.10 Three graduated cylinders, two of 500 mL capacity and one of 2 L capacity, complying with the requirements of BS 604.

6.3.2.11 Standard flask, 200 mL capacity, complying with the requirements of class B of BS 1792.

6.3.3 Apparatus required for the determination of the coarse aggregate content. The following apparatus additional to that given in 6.3.1 is required for the determination of the coarse aggregate content.

6.3.3.1 Siphon can, manufactured to the approximate dimensions shown in figure 3.

NOTE. The siphon can described in BS 812 : Part 2 is suitable; if this can is used, the same outlet should be used throughout.

*The concentrations of solutions in 6.2.2.2, 6.2.2.3 and 6.2.2.4 are adjusted in accordance with the procedure described in 6.4.3.

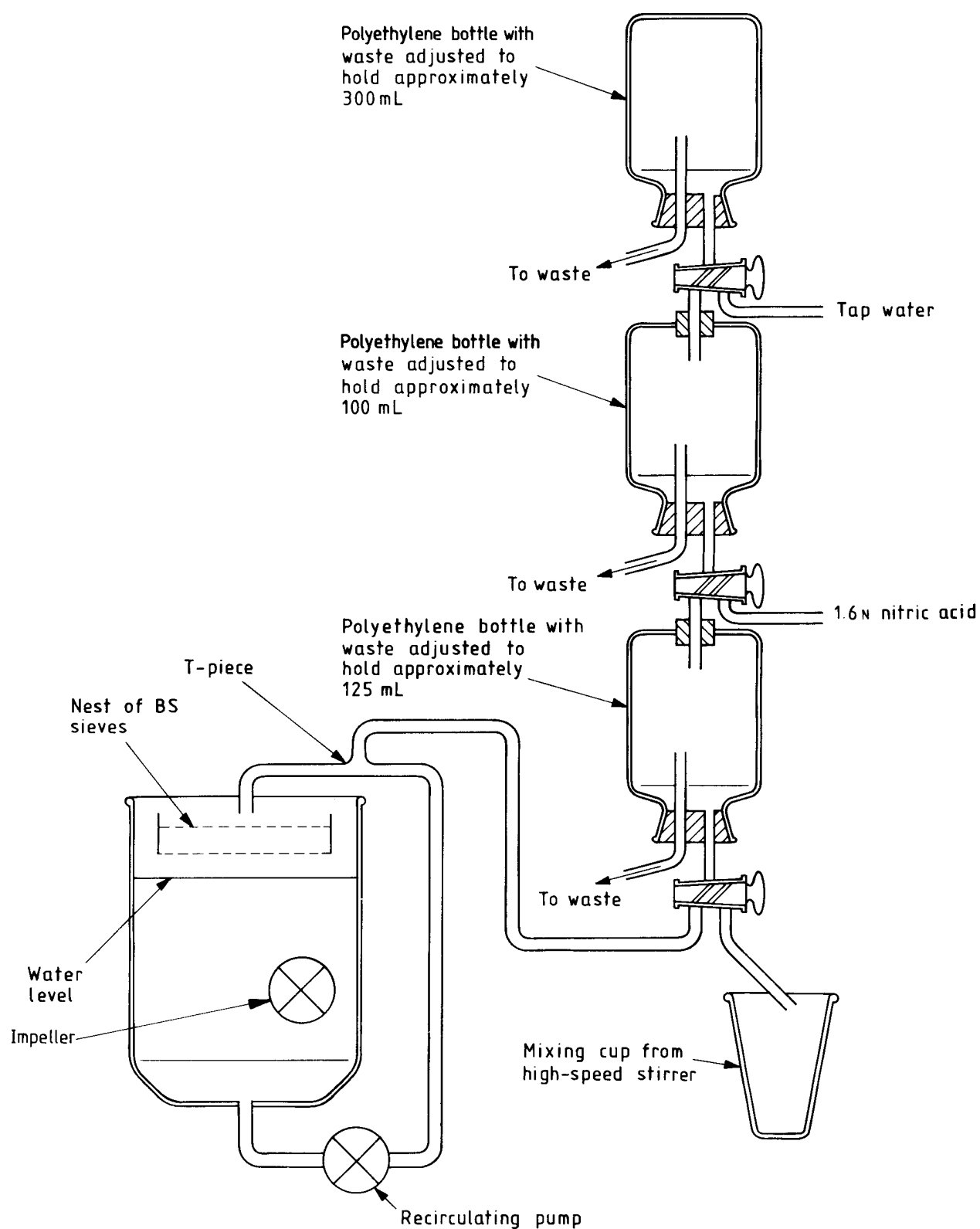
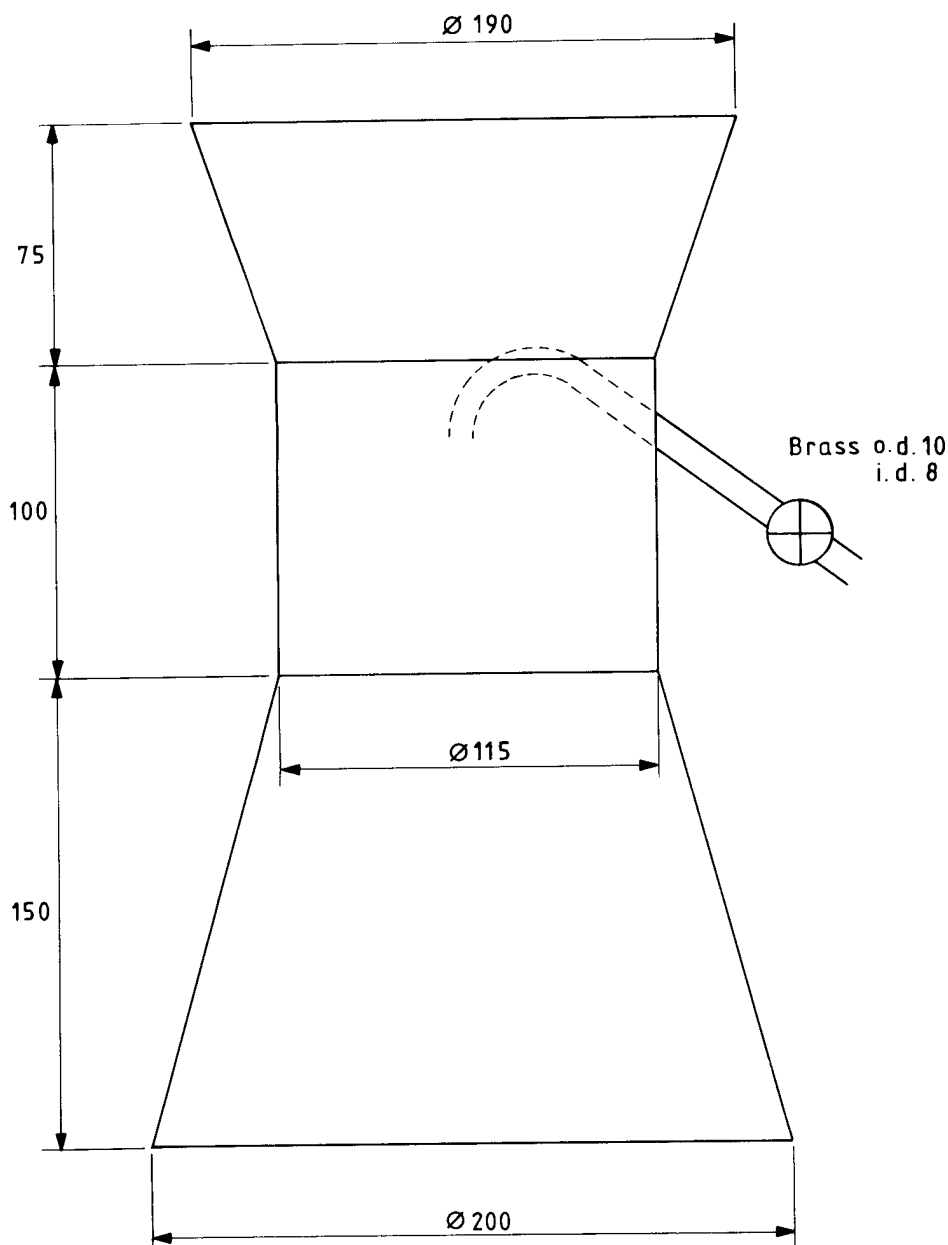


Figure 2. General assembly diagram including sampling tower



All dimensions are in millimetres.

Figure 3. Suitable dimensions for a siphon can

6.3.3.2 Flat, metal stirrer suitable for stirring the aggregate in the siphon can.

6.4 Calibration tests

6.4.1 Determination of the volume of the washing machine tank. Determine the volume of the washing machine tank, up to the fixed mark, by using graduated cylinders of appropriate sizes. Record the volume V .

6.4.2 Assembly and determination of the volumes of the

component parts of the sampling tower. Assemble the sampling tower as shown in figure 2, and adjust the overflow from each component such that the volumes discharged from the bottles are approximately as indicated.

Check the discharge volumes of each of the bottles and record as v_1 for the bottom bottle, v_2 for the middle bottle, and v_3 for the top bottle.

6.4.3 Adjustment of the concentrations of the standard volumetric solutions

6.4.3.1 General. Whenever fresh solutions of silver nitrate, sodium chloride or potassium thiocyanate are prepared, adjust the concentrations so that their respective concentration ratios are 1:1:0.1.

6.4.3.2 Titrations. To adjust the ratios, carry out the following titrations.

(a) *Silver nitrate and sodium chloride.* Pipette 25 mL of sodium chloride into a 250 mL conical flask and add 1 mL of potassium chromate, 50 g/L solution.

Fill a burette to the zero mark with silver nitrate solution and add to the sodium chloride until a permanent reddish colouration is obtained.

Record the volume x mL of silver nitrate required for this titration.

(b) *Silver nitrate and potassium thiocyanate.* Measure from a burette x mL of silver nitrate solution, i.e. the volume required for the titration measured in (a), into a 500 mL conical flask and add 3 mL of iron (III) ammonium sulphate, 100 g/L solution, and 3 mL of approximately 8N nitric acid solution.

Fill the 200 mL standard flask (see 6.3.2.11) to the mark with potassium thiocyanate solution and add to the silver nitrate solution in the conical flask. Rinse out the standard flask with distilled water and add the washings to the contents of the conical flask.

Fill a 50 mL burette to the zero mark with potassium thiocyanate solution and add to the silver nitrate solution until a permanent reddish colouration is obtained.

Record the volume y mL of potassium thiocyanate required for the titration.

6.4.3.3 Comparison of concentrations. Compare the ratio $25:x:y$ with $1:1:10$ to deduce which solutions, if any, have to be diluted to give the required ratio of concentrations:

- (a) if $25 > x$ and $y/10$, then dilute the silver nitrate and potassium thiocyanate solutions;
- (b) if $x > 25$ and $y/10$, then dilute the sodium chloride and potassium thiocyanate solutions;
- (c) if $y/10 > 25$ and x , then dilute the sodium chloride and silver nitrate solutions.

If any adjustments are made to the concentrations of the solutions, repeat the titrations described in 6.4.3.2 and compare the ratios $25:x:y$ with $1:1:10$.

6.4.4 Determination of the cement calibration curve

6.4.4.1 General. Before the analysis of the concrete is carried out, construct a calibration curve based on the deflections obtained when the flame photometer is being offered different concentrations of cement in nitric acid, the cement being typical of that to be used in the concrete to be analysed.

6.4.4.2 Sample of cement. Take a sample of cement by the method specified in BS 4550 : Part 1. Reduce this sample to approximately 1 kg.

6.4.4.3 Procedure. Weigh a representative subsample of 1.250 g of the cement into a 250 mL glass beaker. Add approximately 50 mL of water and stir to disperse

the cement; then wash the contents of the beaker into the mixing cup of the high-speed stirrer. Add 20 mL of 8N nitric acid solution and stir, using the electric stirrer, for 3 min. Transfer the contents of the mixing cup to a 1 L volumetric flask, dilute with water to the 1 L mark and mix thoroughly.

Accurately dilute portions of this solution with water to obtain the concentrations given in table 2.

Table 2. Concentrations of cement solutions for flame photometer calibrations

Volume of solution	Volume of tap water	Concentration
mL	mL	mg/mL
0	100	0.00
8	92	0.10
20	80	0.25
28	72	0.35
40	60	0.50
48	52	0.60
60	40	0.75
72	28	0.90
80	20	1.00
88	12	1.10
100	0	1.25

Within 2 h of the addition of the acid to the cement, offer each of the above solutions to the flame photometer.

Light the gas jet of the flame photometer and allow the machine to warm up for a period of 10 min to 15 min.

Offer a sample of tap water to the flame photometer and adjust the galvanometer deflection to zero. Flush the inlet tube with distilled water and offer the solution containing 1.25 mg/mL of cement to the inlet tube. Adjust the sensitivity of the photometer to give a full scale deflection (i.e. 100). From this point, until the standard calcium has been calibrated (see 6.4.4.4), ensure that the sensitivity setting remains undisturbed.

Thoroughly flush the inlet tube with distilled water and check the zero with tap water, then offer each of the remaining solutions of cement to the photometer and record the scale deflections. Always flush and check the zero between offering the different solutions.

Plot a calibration curve of the concentrations in mg/mL of cement against galvanometer scale readings as shown in figure 4.

6.4.4.4 Calibration of the flame photometer using a standard calcium solution

6.4.4.4.1 General. Because the cement solution will deteriorate after 2 h, prepare a standard calcium solution, equivalent to a solution containing 1.250 mg/mL of cement, which is then used to check that the scale deflection remains at 100 for this solution.

6.4.4.4.2 Preparation of the standard calcium solution. Weigh on a watchglass approximately 5 g of analytical reagent grade calcium carbonate, and dry in an oven at $105 \pm 5^\circ\text{C}$ for 60 ± 10 min.

Mix in a beaker a subsample of 1.605 ± 0.005 g of calcium carbonate and 10 mL of tap water. Carefully add 20 mL

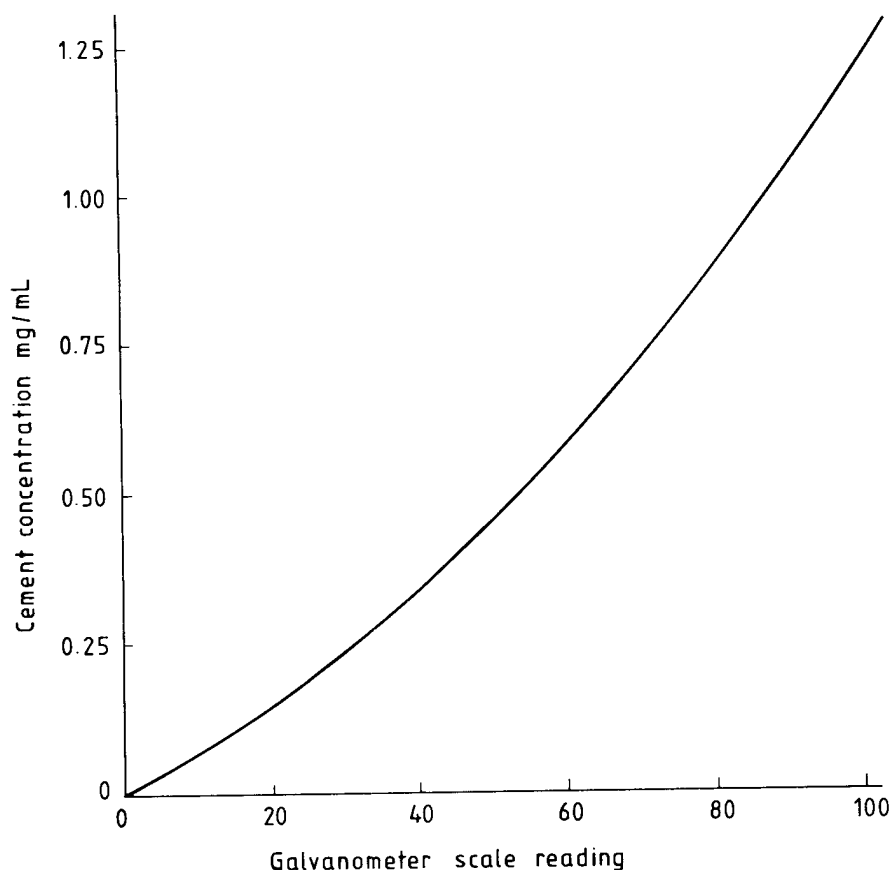


Figure 4. Typical cement calibration curve

of approximately 8N nitric acid solution to the mixture and cover the beaker with a watchglass to prevent any loss of solution.

After the calcium carbonate has dissolved, transfer the solution to a 1 L volumetric flask and dilute to the mark.

6.4.4.4.3 Standardization of the calcium solution.

Successively dilute the calcium solution, offering each diluted solution to the flame photometer until a scale reading of 100 is obtained.

Use this standardized calcium solution to set the galvanometer scale deflection to 100 before each cement determination is carried out.

6.4.5 Calibration of the siphon can to be used for the determination of the coarse aggregate content

6.4.5.1 Sample of coarse aggregate. Take a sample of the coarse aggregate, typical of the material to be used in the concrete, by the appropriate method specified in BS 812 : Part 1.

6.4.5.2 Procedure. Close the outlet tube of the siphon can and fill it with water to above the level of the tube within the can. Open the tube and allow the siphon to operate. Close the tube, after siphoning has been completed, then weigh the siphon can and contents.

Empty the can, then repeat the above procedure twice more.

Calculate an average mass for the can plus water and record as S_w .

Refill the can with water to above the level of the tube within the can. Carefully add a weighed sample of approximately 2 kg of the coarse aggregate in the saturated surface dry condition (see BS 812 for further guidance), and stir or agitate the aggregate to remove any entrapped air. Open the siphon tube and, after siphoning is completed, reclose it. Weigh the can and contents.

Empty the can, then repeat the procedure twice more, with further samples of coarse aggregate.

Calculate an average mass for the can plus contents and record as S_a .

Complete the calibration chart as shown in figure 5.

6.5 Analysis of concrete

6.5.1 Sampling and sample reduction. Using the procedure described in 3.6, take a sample of concrete and reduce it to give duplicate test samples for cement content of 2.5 ± 0.5 kg.

NOTE. The concrete set aside as described in 4.6.5 is required for the determination of the water content of the concrete.

6.5.2 Determination of cement content. Determine the cement content of the duplicate test samples as follows.

Fill the washing machine to the mark with V mL of water and support the two sieves in position over the washing machine tub.

Charge the appropriate bottles of the sampling tower with water and approximately 1.6N nitric acid solution.