

EUROPEAN STANDARD

EN 196

NORME EUROPEENNE

Part 7

EUROPAISCHE NORM

December 1989

UDC 666.94:691.54:620.11

Key words: Cements, tests, sampling, samples, packing

English version

Methods of testing cement; Methods of taking and
preparing samples of cement.

Méthodes d'essais des ciments; Méthodes de prélèvement et d'échantillonnage du ciment. Prüfverfahren für Zement; Verfahren für die Probenahme and Probenauswahl von Zement.

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European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

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Ref. No. EN 196-7:1989 E

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Brief History

This European Standard was drawn up by the Technical Committee CEN/TC 51 'Cement' the Secretariat of which is held by IBN.

In accordance with the Common CEN/CENELEC Rules, the following countries are bound to implement this European Standard:

Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom

Foreword

The standard EN 196 on methods of testing cement consists of the following Parts:

- Part 1: Determination of strength
- Part 2: Chemical analysis of cement
- Part 3: Determination of setting time and soundness
- Part 4: Quantitative determination of constituents
- Part 5: Pozzolanicity test for pozzolanic cements
- Part 6: Determination of fineness
- Part 7: Methods of taking and preparing samples of cement
- Part 21: Determination of the chloride, carbon dioxide and alkali content of cement

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1 Object and field of application

This European Standard describes only the equipment to be used, the methods to be followed and the provisions to be complied with for taking samples of cement representative of given lots for testing to assess the quality of products prior to, during or after delivery.

The provisions of this standard are only applicable when samples of cement are requested for verifying the compliance of

either:

- a) current production of a cement at any time with a standard (for example to meet the needs of the manufacturers auto-control or again in response to the requirements of a certification procedure);
- or b) a delivery or a lot with a standard, the provisions of a contract or the specification in an order.

The standard is applicable to the taking of samples of all types of cements¹⁾ defined by ENV197²⁾ whether they are:

- c) contained in silos;
- d) contained in bags, canisters, drums or any other packages;
- e) transported in bulk in road vehicles, railway wagons, ships etc.

2 References

EN 196-1: 1987 Methods of testing cement - Determination of strength

EN 196-2: 1987 Methods of testing cement - Chemical analysis of cement

1) The requirements of this standard can also, by agreement between the parties, be followed for acceptance inspections for all non-standardized hydraulic binders.

2) At present at the draft stage.

- EN 196-3: 1987 Methods of testing cement - Determination of setting time and soundness
- ENV 196-4 Methods of testing cement - Quantitative determination of constituents
- EN 196-5: 1987 Methods of testing cement - Pozzolanicity test for pozzolanic cements
- EN 196-6 Methods of testing cement - Determination of fineness
- EN 196-21 Methods of testing cement - Determination of the chloride, carbon dioxide and alkali content of cement
- ENV 197 Cement - Composition, specifications and conformity criteria²⁾

3 Definitions

For the purpose of this European Standard, the following definitions apply.

3.1 order: Quantity of cement covered by a single requisition placed with the same manufacturer (or distribution depot). It may consist of one or more *consignments* spread over a period of time.

3.2 consignment: Quantity of cement delivered at a given time by a manufacturer (or distribution depot). It may consist of one or more *lots*.

3.3 lot: Quantity of cement produced under conditions presumed uniform. After specified tests (mainly by ENV197²⁾ this quantity is regarded as a whole 'complying' or 'not complying' with standards or contract requirements.

3.4 increment: Quantity of cement taken in a single operation of the sampling equipment used.

2) At present at the draft stage.

3.5 sample (general term): Quantity of cement taken at random, or in accordance with a sampling plan, from a larger quantity (silo, stock of bags, wagons, trucks, etc.) or from a fixed lot, relating to the intended tests. A sample may consist of one or more *increments*.

3.6 spot sample: *Sample* taken at the same time and from one and the same place, relating to the intended tests. It can be obtained by combining one or more immediately consecutive *increments*.

3.7 composite sample: Homogeneous mixture of *spot samples* taken:

- a) at different points or
- b) at different times

from a larger mass of the same cement, obtained by thoroughly mixing the combined spot samples and, if necessary, reducing the size of the resulting mixture.

3.8 laboratory sample: *Sample* prepared by thoroughly mixing and if necessary reducing from a larger sample (spot or *composite sample*) and intended for use by the laboratories undertaking the tests. These laboratories are generally those of the producer, those designated in the order or in the certification regulations.

3.9 sample for retest: *Sample* which is to be kept for possible subsequent tests in the event of the results from tests carried out on the laboratory samples being in doubt or disputed. The sample for retest is generally the remainder of a *laboratory sample* after a first series of tests.

3.10 retained sample: *Sample* taken systematically from regular deliveries (for example for large works), if necessary in the presence of all the parties concerned, to be retained for possible testing in the event of doubt or dispute or subsequent problems.

NOTE. Definitions 3.1, 3.2, 3.3 and 3.10 only have meaning in the case of checks made when taking delivery of a supply.

4 General

4.1 The aim of the sampling operations is to obtain from a large quantity of cement (contained in a silo, a stock of bags in a warehouse, truck etc., or in a defined lot) one or more smaller quantities, considered by the parties concerned to represent the quantity of cement of which the quality is to be assessed.

4.2 The equipment used, methods followed, and precautions taken may vary according to the nature of the installation and the circumstances in which the samples are taken.

4.3 In the case of the checks carried out within the context of a certification procedure the measures other than those described below to ensure that the samples are representative are within the jurisdiction of the certification body (see clause 7).

4.4 In the case of taking delivery of a supply, the sampling operations shall, in principle, take place in the presence of the manufacturer (or vendor) and the customer (or purchaser) or their respective representatives. The absence of one of them shall not, however, be a barrier to the operations but if this occurs it shall be mentioned in the sampling report (see clause 10).

The sample is normally taken before or during delivery. However, if necessary, it may be taken after delivery but with a maximum delay of 24 h. In this latter case the results of the checks shall be interpreted with care.

Indeed, for various reasons, the cement to be tested may no longer be representative of the product at the actual moment of its delivery. For this reason the time of sampling shall be carefully recorded in the sampling report (see clause 10). When samples are taken after delivery, the requirements in the first paragraph above remain applicable.

5 Sampling equipment

On account of the diversity of industrial installations and circumstances in which the samples have to be taken, it is not possible to describe one type of reference equipment with which other types of equipment can be compared through a series of tests before being able to be used. Hence, this standard only gives, by way of example in annex A, a simplified illustration of the devices normally used and which have been found satisfactory. This equipment is either portable (ladle, tube, screw sampler etc.) or permanently installed (screw extractor or other equipment fixed permanently onto the container).

The equipment shall be chosen and used in accordance with the following rules.

The equipment shall be

- a) approved by all the parties;
- b) of non-corrodible material, which is not liable to react with the cement;
- c) maintained always in working order and in a clean state. Care shall be taken to ensure that it is carefully cleaned after each sampling operation.³⁾ Care shall also be taken to ensure that it is not contaminated by lubricants from other equipments used.

Permanent equipment shall be installed at locations where there are no hard deposits and where the flow of cement is homogeneous (no segregation) at the time it is being used. In particular, when it is used it shall not be operated during fluidization under air pressure. If fluidization is necessary, no samples shall be taken until the cement has settled for at least half a minute after stopping the air supply.

6 Procedures for taking samples and precautions to be taken depending on the procedures adopted

6.1 General

The most suitable equipment for the circumstances shall be used to take a sample, observing the following precautions as appropriate.

³⁾ However, cleaning is not necessary between the taking of successive increments from the same cement which are intended for making up the sample.

6.2 Sampling from bags, drums and containers of comparable size

When the cement is packed in bags, drums or other small containers, the sample indicated in the second paragraph of 8.1 shall comprise either a single bag or a single container chosen at random from a sufficiently large stock.

6.3 Sampling from large containers

Samples shall be taken while the container is being filled or emptied taking the precautions:

- a) not to operate in dusty or polluting atmospheres;
- b) to take the number of samples necessary to obtain the quantity prescribed in the second paragraph of 8.1;
- c) to transfer the collected cement into a clean, dry and airtight container before proceeding with the operations described in clause 8.

6.4 Sampling from bulk transport (after loading or before unloading)⁴⁾

For all equipment used, care shall be taken not to take material from the top or bottom layers of the mass of cement. The thickness of the layer to be considered is at least 15 cm.

Take the same precautions as those described in 6.3a), b) and c).

6.5 Sampling while loading into bulk transport or silos

Taking samples may be carried out only if suitable equipment is available and if there is access to a homogeneous phase of cement. Take the same precautions as those described in 6.3a), b) and c).

⁴⁾ The equipment described in this standard (see Annex A) does not allow for sample(s) to be taken during unloading.

6.6 Sampling from silos

In the absence of a suitable fixed sampling device (see the last paragraph of clause 5) an appropriate quantity of cement shall be discarded when discharge commences to remove the hard deposits or unwanted mixtures of different cements in the distribution system. This quantity is left to be determined by the manufacturer's representative present at the sampling operation. The necessary quantity prescribed in the second paragraph of 8.1 shall then be collected in a clean and dry container.

6.7 Sampling from bag filling machines

The quantity taken shall comprise a bag of cement obtained during the normal operation of the machine. It can equally comprise the equivalent quantity (see the second paragraph of 8.1) sampled by a mechanical device fitted in the supply hopper directly above the discharge spouts. If the machine is used for packing different cements, it shall be completely purged of all the cement previously packed until the cement from which samples are to be taken arrives. Depending on the type of packing machine, the quantity so removed may be considerable. The manufacturer's representative present at the sampling operation shall determine this quantity.

7 Frequency at which samples are taken and choice of sample type

The frequency of taking samples and the type of sample (spot sample or composite sample) depends on the provisions contained:

- in the agreements between producer and customer;
- in the national, European (ENV 197²⁾ for example) or even international standards;
- in the certification procedures.

2) At present at the draft stage

8 Size and preparation of samples

8.1 Size of sample to be taken for verification of compliance

Each laboratory sample (or sample for retest or retained sample) shall be of such a size that all the tests specified in the contract or required by standards indicated in the contract or the certification procedure can be carried out twice. Thus, unless specified to the contrary, the mass of these samples shall be at least 5 kg (or more, if necessary, to fill completely the container indicated in 9.2).

The total quantity (spot sample or composite sample) to be taken for verifying compliance shall be greater than or at least equal to that required for supplying to all the laboratories concerned the samples indicated in the first paragraph above. This quantity shall be taken by the equipment specified in clause 5 and in accordance with the procedures stated in clause 6. In general taking a sample of 40 kg to 50 kg will suffice.

8.2 Homogenization

8.2.1 General requirements

As soon as the quantity of 40 kg to 50 kg indicated in the second paragraph of 8.1 has been collected, it shall be carefully homogenized (preferably in a laboratory) with clean dry implements not liable to react with the cement.

The homogenization shall preferably be achieved using a mixing machine, the efficiency of which shall have been previously demonstrated (see 8.2.2). Whatever procedure is chosen, it shall be carried out as quickly as possible to minimize the exposure of the cement to the air.

In the absence of a homogenizer, the following procedure is to be adopted:

the quantity of cement to be distributed between the laboratories (see the second paragraph of 8.1) shall be tipped onto a clean dry cloth (or plastics sheet) and shall then be mixed carefully using a shovel.

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This procedure shall only be used if:

- a) the ambient relative humidity is less than 85 %;
- b) all risk of the sample being affected by wind, rain, snow or dust is avoided.

8.2.2 Verification of the efficiency of the homogenizing procedure selected

Take approximately equal quantities of two cements of differing characteristics (i.e. Blaine fineness, by EN 196-6, and additionally colour. Mix these using one of the procedures described in 8.2.1 and note the time between starting and finishing the operation.

Once the operation is judged to be complete, take fifteen 'micro-samples', each of 12 g to 20 g, from points equally distributed within the mass of which the homogeneity is to be verified⁵⁾.

Determine the Blaine fineness three times on each of these micro-samples.

Homogenization shall be considered to be obtained if the analysis of variance shows there to be no significant differences between the fifteen micro-samples. This analysis shall be carried out as described in the literature dealing with statistical control.⁶⁾

In the case of an unsatisfactory result, the mixing shall be repeated, but doubling the time of the mixing operation.

If, after this second trial, there are no significant differences between the fifteen micro-samples, the method with twice the homogenization time shall be considered valid, but, if no noticeable improvement has been obtained in relation to the first trial, the chosen method shall be declared unsuitable. Finally, if there is no decisive improvement, it shall be decided, in view of the time required, whether it is worth repeating the trial with a longer time.

5) To obtain initial information, the colour of these micro-samples can be compared if care has previously been taken to select two cements of distinctly different colour.

6) Each CEN member can give bibliographic references here.

8.2.3 Choice of method

The verification of the efficiency of the homogenizing method (see 8.2.2) is only to be carried out at the time of choosing the method. This choice is left to the initiative of the manufacturer, but he shall be in a position to prove (for example to an expert representing a customer or the certification body) that he has carried out the trial described above by presenting a dossier drawn up on that occasion.

8.3 Division of the quantity necessary for verification of compliance

After homogenization (see 8.2) of the sample made up as specified in the second paragraph of 8.1, it shall be divided into the required number of laboratory or retained samples either by using a sample divider or, after quartering the quantity to be distributed, by extracting with a hand scoop increments of approximately 0,5 kg from each of the quarters and transferring these successively into containers prepared for receiving the laboratory (or retained) samples. This operation shall be continued until the desired mass (stated in the first paragraph of 8.1) is obtained in each container.

The sequence of distributing the contents of the hand scoop from which each laboratory sample is gradually made up shall be as follows:

Distribute successively to each of the laboratory containers X, Y, Z etc.

first a scoopful from A,
then a scoopful from B,
then a scoopful from C,
then a scoopful from D.

This represents one distribution sequence; repeat the same sequence as many times as necessary to reach the quantity prescribed in the first paragraph of 8.1 (see figure 1).

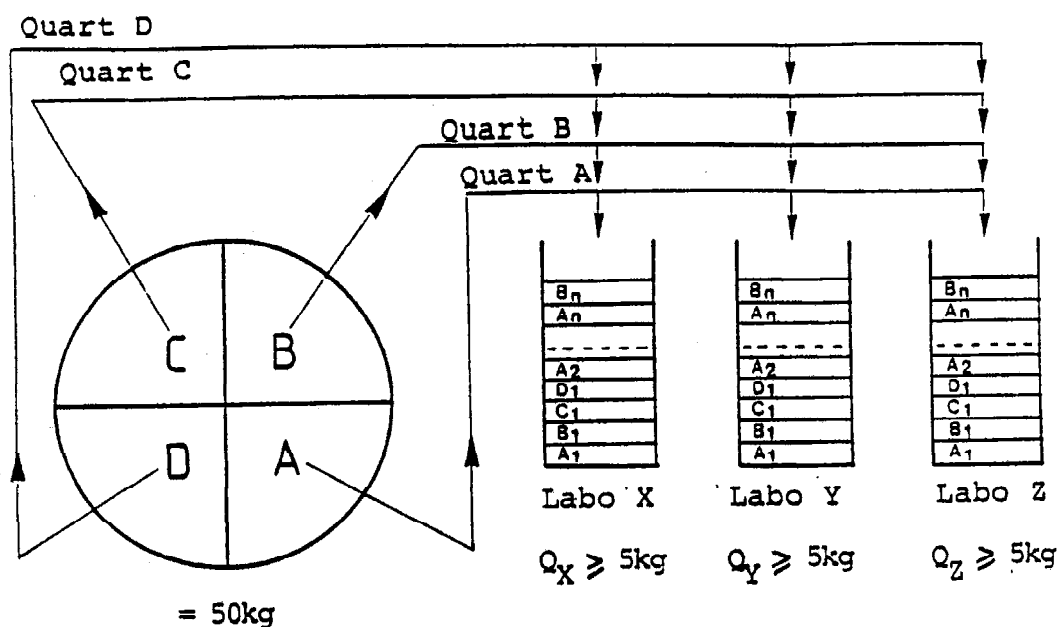


Figure 1 - Make up of laboratory samples

Each laboratory (or retained) sample so prepared shall be packaged as stated in clause 9 and despatched with the minimum delay. It is then up to the receiving laboratory, to store, prepare and treat the sample in a manner appropriate to its subsequent use.

8.4 Foreign bodies in the sample

During the above operations any foreign bodies which may appear shall, in all cases, be noted in the sampling report (clause 10).

7) At this point the work of the laboratory is usually specified in the test standards. (Principally EN 196-1 to EN 196-6 and EN 196-21.)

If they appear to be uniformly distributed in the cement (for example, a powder other than cement, lumps) it is not possible to remove them from the sample. Otherwise, they shall be removed and kept:

a) by one of the two parties (manufacturer, customer) with the agreement of the other in the case of taking delivery of a supply,

or

b) by the agency responsible for inspection in the case of a sampling operation performed in the context of certification regulations.

9 Packaging and storage

9.1 Principle

The packaging of the samples and the method of storage shall always be such as to preserve the properties of the sampled cement. Any conditions likely to affect this shall be indicated.

9.2 Containers

The samples shall be packed, despatched and stored in bags, drums, or solid containers. These shall be made of a material which is inert with respect to cement and non-corrodible. They shall be dry, impervious (to air and moisture) and clean. In this respect they shall not have been previously used for packaging products which are likely to affect the samples.

To prevent aeration the containers shall as far as possible, be completely filled and their closure sealed with adhesive tape⁸⁾.

When made of plastics, containers or bags shall only be used under the following conditions:

a) the period of storage shall be limited to 3 months;

b) the sheet from which they are made shall be at least 100 µm thick;

8) It has to be remembered, however, that the packaging, however air-tight, cannot in the long term prevent a certain amount of aeration, the extent of which may vary depending on the properties of the product.

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c) the plastics material shall under no circumstances cause the cement to become 'air entraining' whether by loss from this material or as a result of surface treatment. On this point, check for the absence of risk by carrying out appropriate tests where needed;

d) provision shall also be made to seal them where necessary, by using suitable tape.

9.3 Storage condition

The samples shall preferably be stored at a temperature of less than 30 °C.

9.4 Identification of samples

In order to identify the samples unambiguously, the containers (bags or drums) shall be clearly and indelibly marked in at least one place. When there is only one mark, this shall be on the body of the container and not on the lid.

Furthermore, a copy of the sampling report described in clause 10 shall be slipped where necessary into a protective envelope placed inside the container.

9.5 Authentication of samples

If necessary, when this is required by the contract of sale or certification regulations, the container shall bear an agreed seal authenticating the sample. This seal shall be fixed so as to prevent the unauthorized opening of the container.

10 Sampling report

10.1 General

A sampling report corresponding to each sample shall be drawn up by the person responsible for the operation. Copies shall be attached to the laboratory (or retained) samples prepared and distributed in accordance with the clauses of the contract or certification regulations.

10.2 Information to be included

10.2.1 *Obligatory information*

The report shall, unless otherwise agreed by the parties, include the following obligatory information:

- a) Name and address of the body responsible for the sampling;
- b) Name and address of the customer (in the case of taking delivery);
- c) Complete standard designation of the cement as specified in ENV 197 ²⁾ , ⁹⁾
- d) Identity of the production works;
- e) Place, date and time of sampling;
- f) Type of sample (spot or composite comprising 'n' spot samples);
- g) Identification mark on the sample container;
- h) Any comments, particularly:
 - the presence of foreign bodies;
 - the circumstances in which the operations took place which are likely to have an effect on the quality of the cement sampled, for example, transport conditions;
 - all information allowing a more precise identification of the cement sampled to be made, for example, the silo number.

10.2.2 *Optional information*

The report shall, unless otherwise agreed by the parties, include the following optional information:

- a) Definition of the lot, the approximate mass of the cement and the type of stock from which the sample has been taken. Example: 3000 t in bags in a warehouse;
- b) The type of container used for the laboratory samples.

2) At present at the draft stage.

9) In the possible case of non-standardized hydraulic binders (see note 1 on page 1) the product should be given an unequivocal mark.

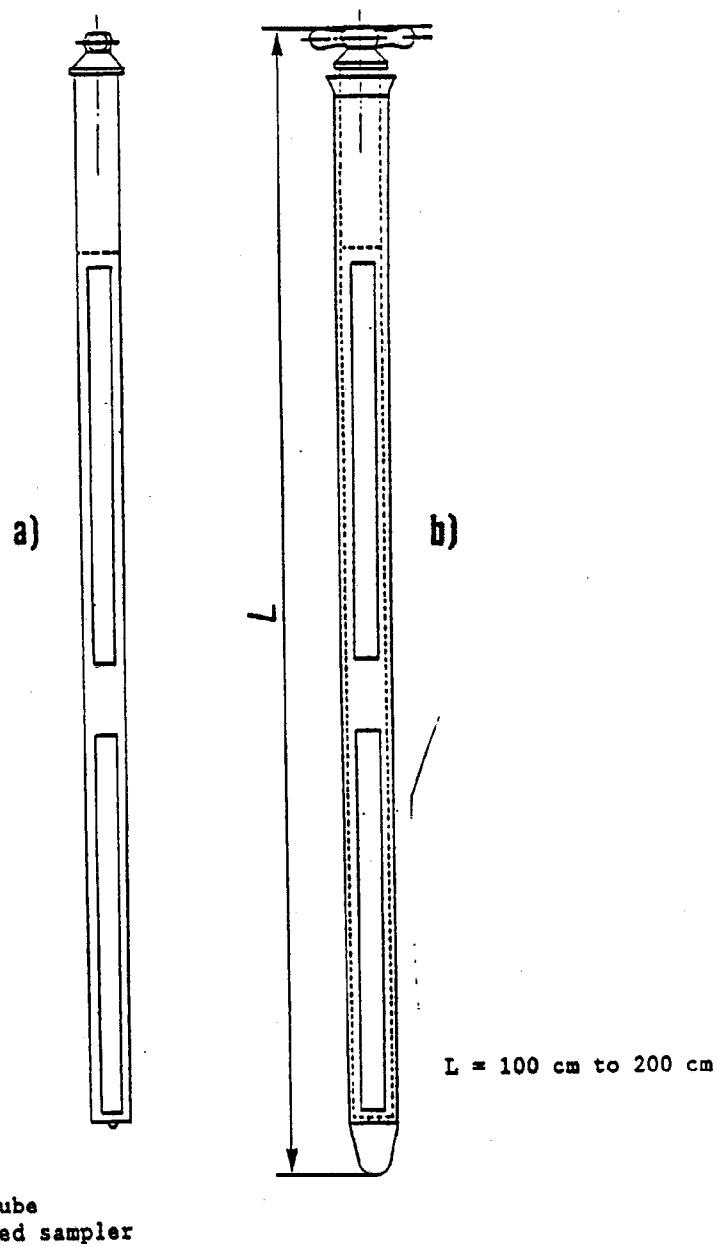
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10.3 Authorization of the report

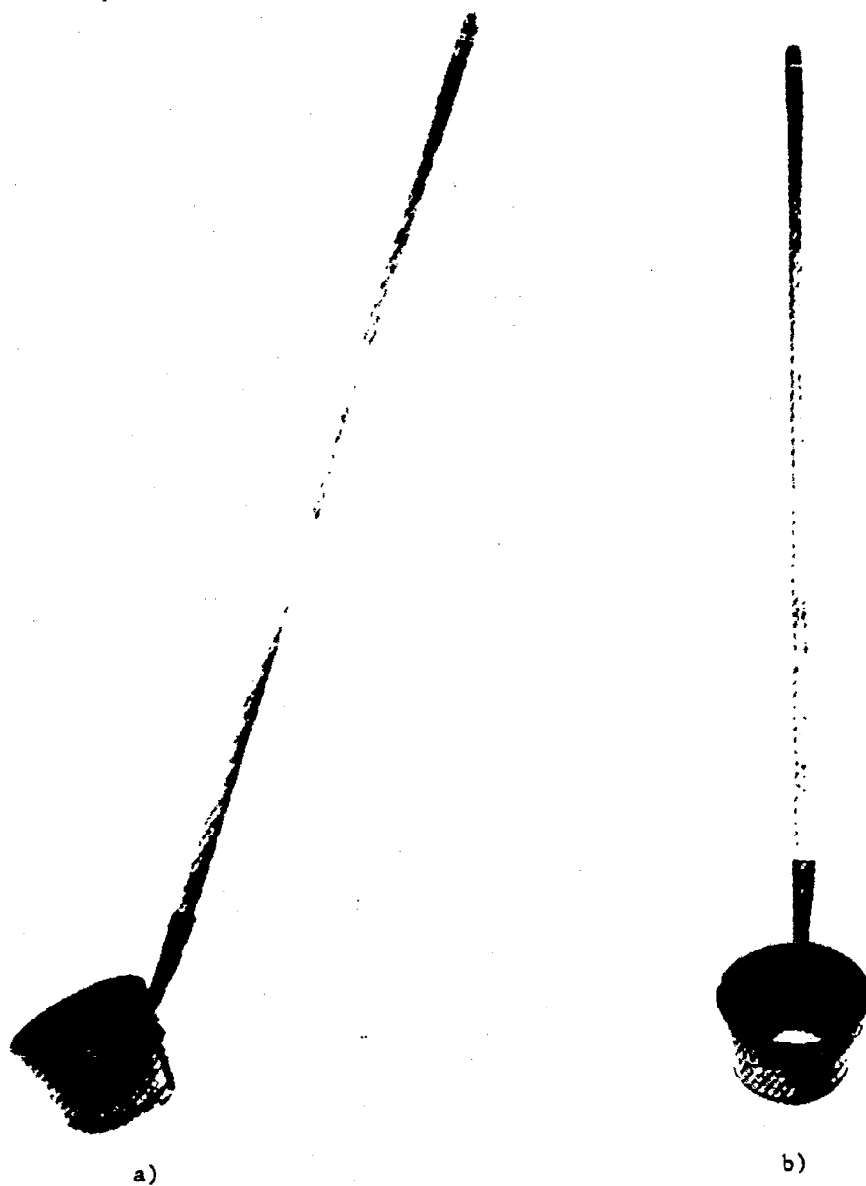
The report and copies shall be signed by the representatives of the parties present at the sampling and/or any other responsible witness approved by the parties.

10.4 Despatch of the report

Copies of the report shall be sent without delay to each of the parties concerned. In addition, one of them shall be enclosed in the container of each laboratory sample and each retained sample as stated in 9.4.

Annex A**Typical examples of sampling equipment normally used****Figure 2. Sampling tube**

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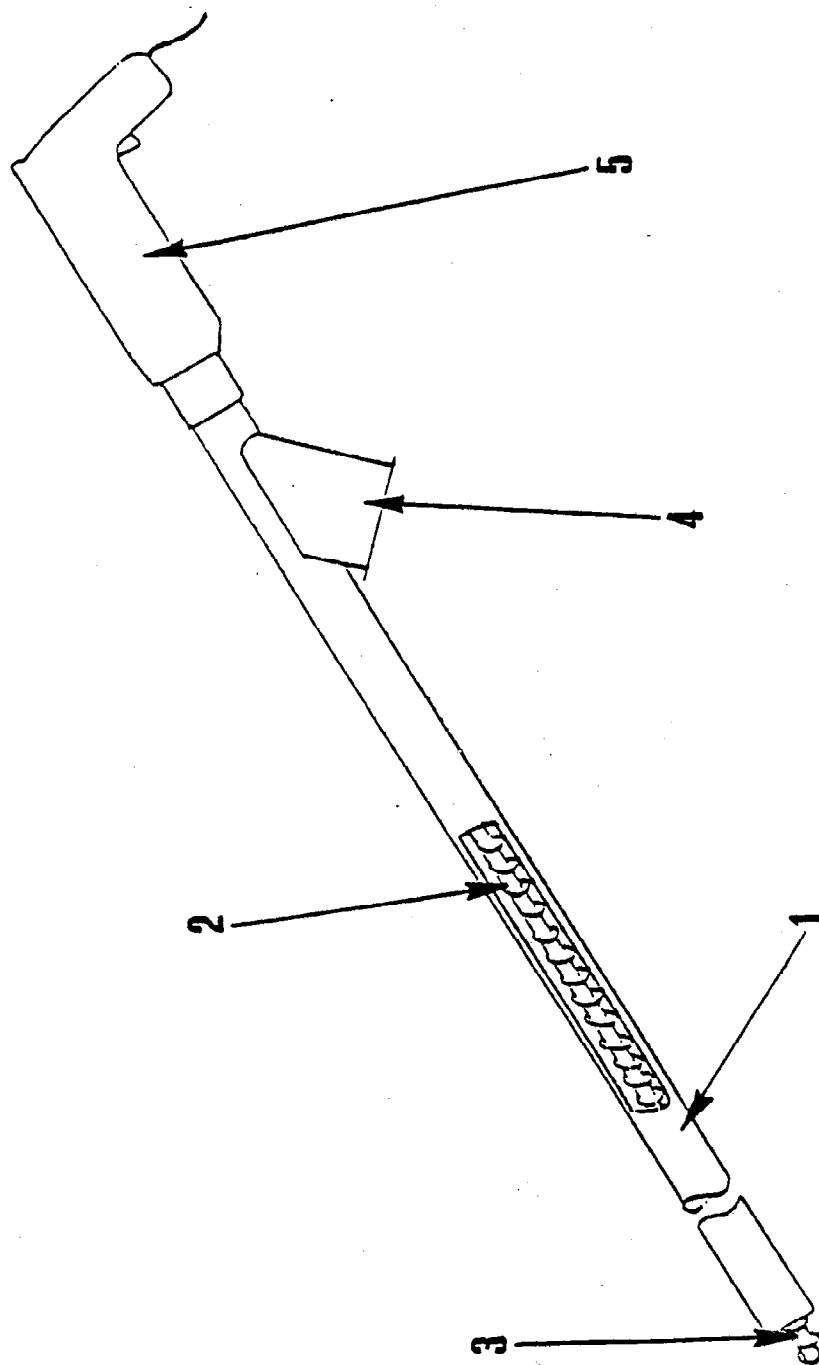


a) side view
b) front view

Approximate size of ladle

Diameter 20 cm
Depth 15 cm
Handle length 180 cm

Figure 3. Typical ladle



1. Tube, diameter approximately 6 cm
2. Spiral flight screw
3. End of flight screw acting as probe at point of entry of cement
4. Discharge chute
5. Electric motor

Figure 4. Typical mechanical screw sampler (overall length approximately 200 cm)

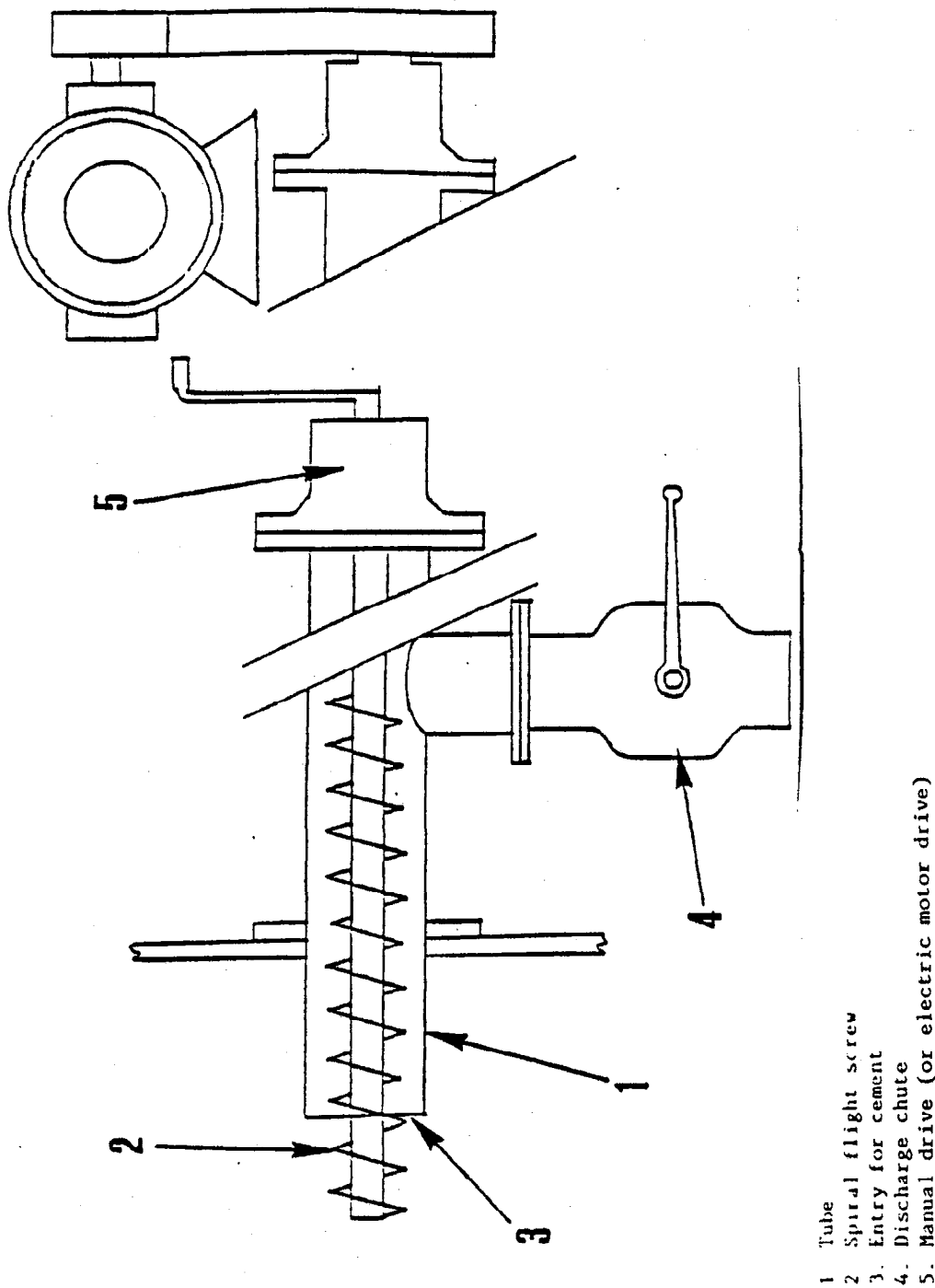
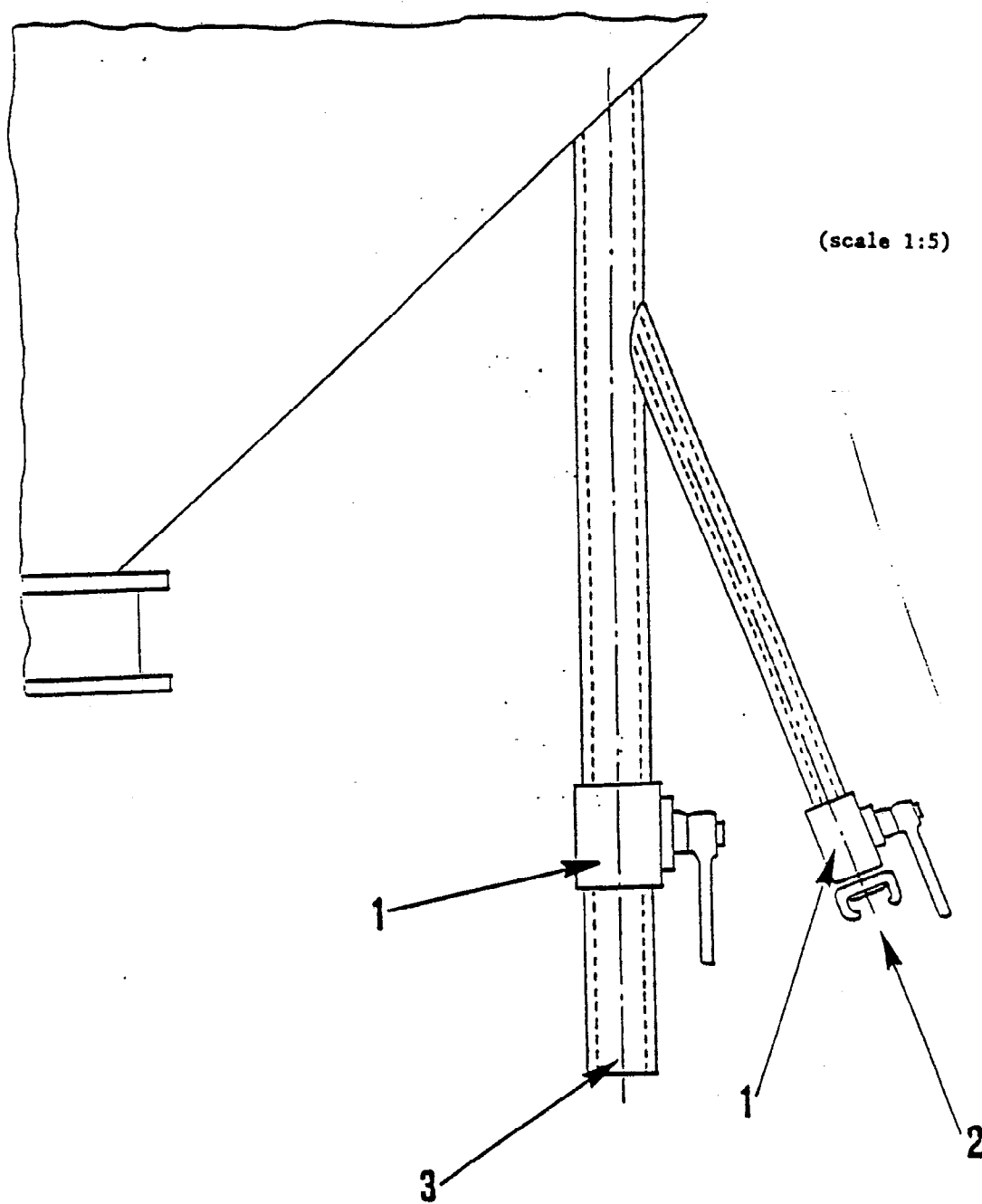


Figure 5. Typical permanently installed mechanical screw sampler



1. $\frac{1}{4}$ turn valve
2. Rubber inlet tube for factory air (3 bar)
3. Discharge chute

Figure 6. Sampling equipment