BRITISH STANDARD

BS EN ISO 10693:2014

Soil quality —

Determination of carbonate content — Volumetric method

ICS 13.080.10

National foreword

This British Standard is the UK implementation of EN ISO 10693:2014. It is identical to ISO 10693:1995. It supersedes BS 7755-3.10:1995 (dual numbered as ISO 10693:1995), which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee EH/4, Soil quality.

A list of organizations represented on this committee can be obtained on request to its secretary.

Additional information. References to labelled parts of the apparatus refer exclusively to Figure 1.

In paragraph 1 of **7.2** it is recommended that the variation in temperature is no more than ± 1 °C and the variation in pressure is no more than ± 0.5 kPa.

In paragraph 2 of **7.2** the phrase “two standards of respectively 0,200 g and 0,400 g” means that one sample of each should be prepared.

Textual errors. In note 2 in clause 3, “Annex A ” should read “Annex B”. In paragraph 4 of **7.2** “tube H” should read “tube E”.

This British Standard, having been prepared under the direction of the Health and Environment Sector Board, was published under the authority of the Standards Board and comes into effect on 15 October 1995

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Amendments/corrigenda issued since publication

Date	Comments
30 June 2014	This corrigendum renumbers BS 7755-3.10:1995 (dual numbered as ISO 10693:1995) as BS EN ISO 10693:2014

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 10693

March 2014

ICS 13.080.10

English Version

Soil quality - Determination of carbonate content - Volumetric method (ISO 10693:1995)

Qualité du sol - Détermination de la teneur en carbonate - Méthode volumétrique (ISO 10693:1995)

Bodenbeschaffenheit - Bestimmung des Carbonatgehaltes - Volumetrisches Verfahren (ISO 10693:1995)

This European Standard was approved by CEN on 13 March 2014.

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Ref. No. EN ISO 10693:2014 E

Foreword

The text of ISO 10693:1995 has been prepared by Technical Committee ISO/TC 190 “Soil quality” of the International Organization for Standardization (ISO) and has been taken over as EN ISO 10693:2014 by Technical Committee CEN/TC 345 “Characterization of soils” the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2014, and conflicting national standards shall be withdrawn at the latest by September 2014.

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Endorsement notice

The text of ISO 10693:1995 has been approved by CEN as EN ISO 10693:2014 without any modification.

Contents

1 Scope

This International Standard specifies a method for the determination of carbonate content in soil samples.

It is applicable to all types of air-dried soil samples.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 11464:1994, *Soil quality — Pretreatment of samples for physico-chemical analyses.*

ISO 11465:1993, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method.*

3 Principle

Hydrochloric acid is added to a soil sample to decompose any carbonates present. The reaction in simplified form reads as follows (Me means metal):

$$MeCO_3 + 2H^+ \rightarrow Me^{2+} + H_2CO_3$$

$$H_2CO_3 \rightarrow H_2O + CO_2 \text{ (gaseous state)}$$

The volume of the carbon dioxide produced is measured by using a Scheibler apparatus (**5.1**), and is compared with the volume of carbon dioxide produced by pure calcium carbonate. To avoid making corrections for differences in temperature and pressure, all determinations are carried out under the same conditions. The determination should be carried out in a temperature-controlled room.

NOTE 1 The carbonate content is expressed as an equivalent concentration of calcium carbonate ($CaCO_3$). In fact all carbonates and bicarbonates present in the sample are measured. Many carbonates appear in the form of calcite and aragonite ($CaCO_3$), dolomite [$CaMg(CO_3)_2$], siderite ($FeCO_3$) and rhodochrosite ($MnCO_3$). In soils in dry (arid) regions, soda ($Na_2CO_3.10\ H_2O$) may be present. When it is known that a certain form of carbonate, other than calcium carbonate, is mainly present in the soil under study, the final concentration of this form can be used.

NOTE 2 Other gases that are produced [e.g. hydrogen sulfide (H_2S) in samples of anaerobic soil containing sulfides] may result in an overestimate of the carbonate content. In these cases the gas produced should be purified and its volume measured in another way (see [1] in Annex A). When sulfides are known to be present in the soil samples, mercury(II) chloride ($HgCl_2$) is added to the hydrochloric acid solution to form insoluble mercury(II) sulfide (HgS).

4 Reagents

Use only reagents of recognized analytical grade.

4.1 *Water*, with a specific electrical conductivity not higher than 0,2 mS/m at 25 °C (conforming to grade 2 of ISO 3696).

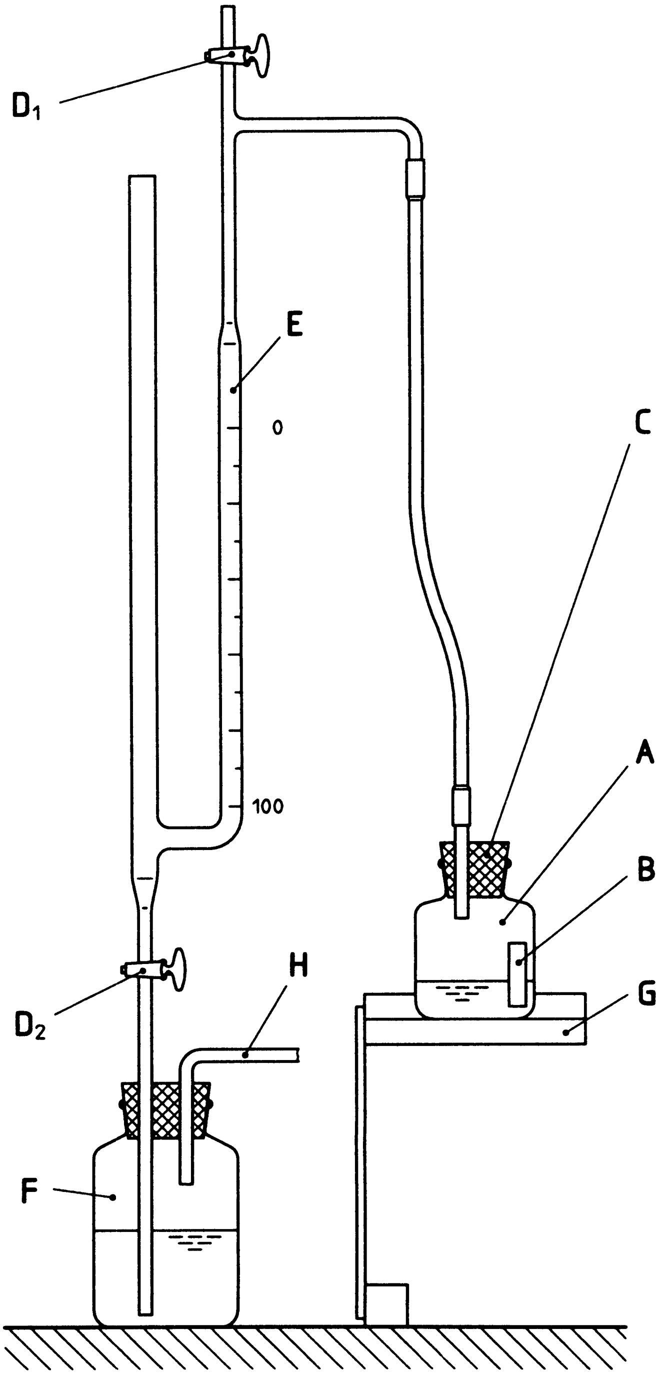
4.2 *Hydrochloric acid*, $c(HCl) = 4$ mol/l.

Dilute 340 ml of concentrated hydrochloric acid ($\rho = 1{,}19$ g/ml) to 1 000 ml with water (**4.1**).

4.3 *Calcium carbonate* ($CaCO_3$), powder.

5 Apparatus and glassware

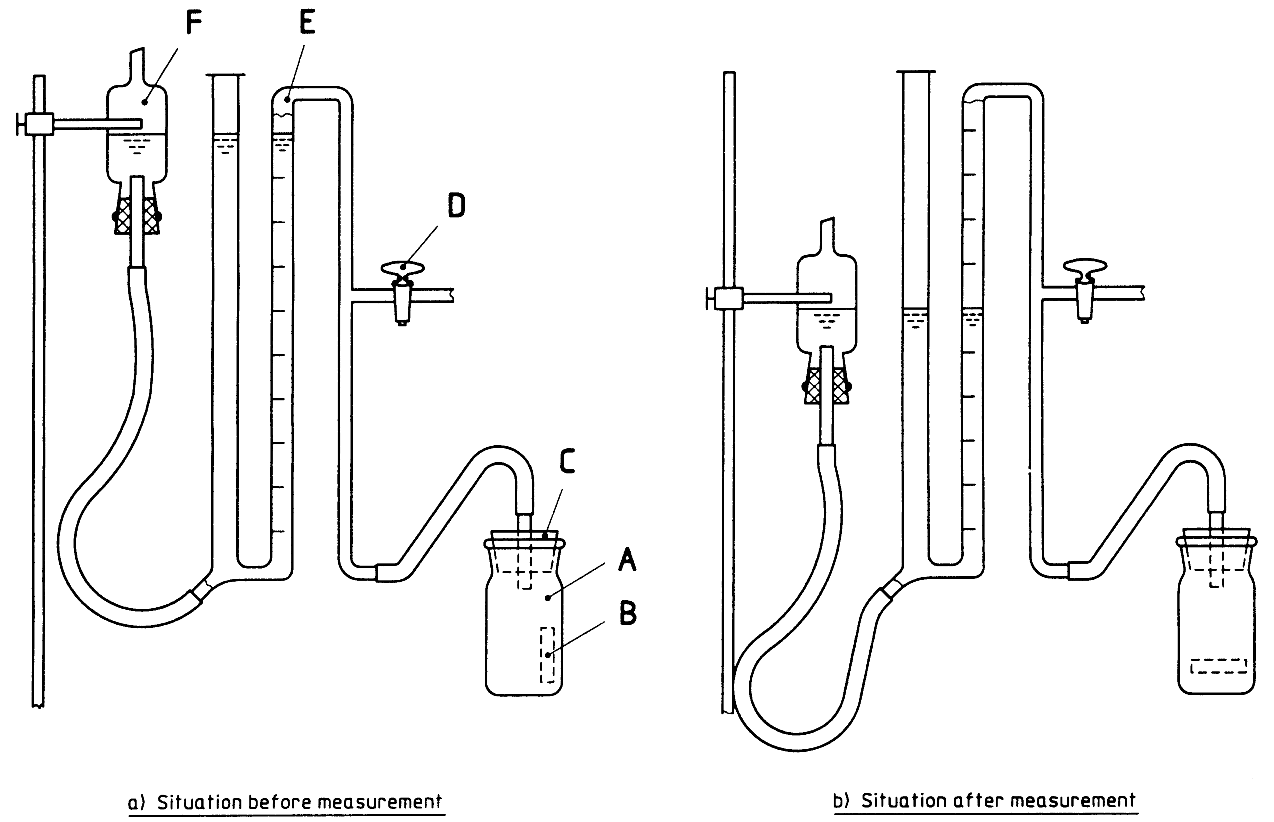
5.1 *Scheibler apparatus*, adapted for carrying out a single sample determination (see Figure 1). An example of a single unit of apparatus, together with an indication of the water level before and after the measurement, is given in Figure 2.



Key

A	Reaction vessel (**5.3**)
B	Plastics cup (**5.4**) with hydrochloric acid (**4.2**)
C	Rubber stopper
D_1	Stop-cock
D_2	Stop-cock
E	U-shaped calibration tube
F	Storage vessel with water
G	Shaking beam
H	Air-inlet tube

Figure 1 — Example of a Scheibler unit for a single sample determination



Key

A	Reaction vessel (**5.3**)
B	Plastics cup (**5.4**) with hydrochloric acid (**4.2**)
C	Rubber stopper
D	Stop-cock
E	U-shaped calibration tube
F	Storage vessel with water

Figure 2 — Example of a single Scheibler unit

NOTE 3 For the determination specified in this International Standard, a Scheibler apparatus with two glass U-shaped calibration tubes each having a volume of 100 ml is used. It is also possible to use tubes with a different volume (i.e. 50 ml or 200 ml). In this case the mass of the test portion used for the measurement has to be adapted.

NOTE 4 The temperature of the water in the system should be equal to the ambient temperature.

NOTE 5 When only a few soil samples have to be analysed, a minimum of five Scheibler units is sufficient. In this case the reaction vessels can be shaken by hand. However, it is then necessary to take into account the temperature variations in the air inside the vessels.

NOTE 6 By lowering the water level in the tube on the left (see Figure 2) with the stop-cock closed, an underpressure is created in the tube on the right. By creating such an underpressure for some time, the unit can be checked for leaks.

5.2 *Analytical balance*, with an accuracy of at least 0,1 mg.

5.3 *Reaction vessels*, of capacity 150 ml and with a wide neck.

5.4 *Plastics cup*, of capacity about 10 ml, which can pass through the neck of the reaction vessel (**5.3**).

5.5 *Tongs*, acid-proof.

5.6 *Watch glass*

6 Laboratory sample

Use the < 2 mm fraction of air-dried soil samples pretreated according to ISO 11464. Use part of the laboratory sample, to determine the water content according to ISO 11465.

7 Procedure

7.1 Preparation

For a preliminary test, add some hydrochloric acid (**4.2**) to a portion of the soil on a watch glass (**5.6**). The carbonate content of the sample can be estimated on the basis of the intensity and time of effervescence; see Table 1. Determine from Table 1 the mass of test portion for the determination according to **7.2**.

Table 1 — Relationship between intensity and time of effervescence, estimated carbonate content of the soil and mass of test portion

Intensity of effervescence	Carbonate content g/kg	Mass of test portion g
None or only limited	< 20	10
Clear, but for a short time	20 to 80	5
Strong, for a long time	80 to 160	2,5
Very Strong, for a long time	> 160	⩽ 1

NOTE 7 If the mass of the test portion is less than 2 g, it should be taken from a representative portion of the laboratory sample that is crushed to a particle size of less than 250 µm (according to ISO 11464).

NOTE 8 Soils with a pH-H_2O less than 6,5 contain carbonates only in exceptional circumstances (e.g. recent liming or the presence of carbonates with a low solubility such as shells).

7.2 Measurement

Determinations of samples, blanks and the calcium carbonate used as standard material, shall be performed simultaneously in a room where temperature and pressure do not vary too much during the measurement.

For each series, include two blank determinations and two standards of respectively 0,200 g and 0,400 g of calcium carbonate (**4.3**).

Weigh a representative test portion of the laboratory sample (see **7.1** and Table 1). Transfer this amount quantitatively into the reaction vessels (**5.3**) and add 20 ml of water (**4.1**). Also weigh the standards of 0,200 g and 0,400 g of calcium carbonate, transfer these amounts quantitatively into the reaction vessels (**5.3**) and add 20 ml of water (**4.1**). For the blank determinations, use reaction vessels containing 20 ml of water.

Place the reaction vessels in front of the Scheibler units (**5.1**, see Figure 1) in such a way that blanks and standards are randomly distributed over the units. Open the stop-cocks D_1 and D_2 and bring the water level in the tube H to 3 ml for the samples and standards, and to 20 ml and 80 ml for the respective blanks. Close stop-cock D_2.

Fill the plastics cup (**5.4**) with 7 ml of hydrochloric acid (**4.2**) and place this, using tongs (**5.5**), in the reaction vessel containing the test portion. Take care that there is no contact between the hydrochloric acid and the soil before the reaction vessel is connected to the Scheibler unit.

Moisten the rubber stoppers of the apparatus with water and connect the reaction vessels. Close stop-cock D_1 and once again check the water level in the tubes. Carefully add the hydrochloric acid from the cup (**5.4**) to the soil by tilting the reaction vessel at an angle. The gas produced will lower the water level in the tube on the right and at the same time will raise the water level in the tube on the left. Shake for 5 min and note the volume when it no longer varies. If it still varies, continue shaking until the volume is stable, but not longer than 1 h. Note the volume. During shaking, take care that the difference between the water levels in both tubes does not exceed 3 ml. This can be controlled by opening the stop-cock D_2. At the end of the shaking period, bring the water level in both tubes to the same height and measure the volume of gas in the calibrated tube with an accuracy of 0,1 ml.

NOTE 9 The changes in volume of the blanks should not exceed 1,0 ml.

NOTE 10 More water should be added to soils with a high organic matter content.

8 Calculation

Calculate the changes in volume of the gas produced by the sample (V_1), the calcium carbonate standards (V_2) and the blanks (V_3) by subtracting the initial value in the calibrated tube from the final value.

Calculate the carbonate content of the sample using the following equation:

$$w(\mathrm{CaCO_3}) = 1\ 000 \times \frac{m_2(V_1 - V_3)}{m_1(V_2 - V_3)} \times \frac{100 + w(\mathrm{H_2O})}{100}$$

where

$w(\mathrm{CaCO_3})$	is the carbonate content, in grams per kilogram, of the oven-dried soil;
m_1	is the mass, in grams, of the test portion;
m_2	is the mean mass, in grams, of the calcium carbonate standards;
V_1	is the volume, in millilitres, of carbon dioxide produced by the reaction of the test portion;
V_2	is the mean volume, in millilitres, of carbon dioxide produced by the calcium carbonate standards;

V_3 is the volume change, in millilitres, in the blank determinations (this value can be negative);

$w(H_2O)$ is the water content, expressed as a percentage by mass, of the dried sample, determined according to ISO 11465.

9 Repeatability

The repeatability of the determination of carbonate content carried out in two separate, consecutive measurements shall satisfy the conditions given in Table 2.

Table 2 — Repeatability

Carbonate content g/kg	**Acceptable variation**
0 to 50	3 g/kg
> 50 to 150	6 % of the value
> 150 to 180	9 g/kg
> 180	5 % of the value

10 Test report

The test report shall contain the following information:

a) a reference to this International Standard;

b) all information necessary for the complete identification of the sample;

c) the results of the determination in whole numbers, in grams per kilogram, calculated on the basis of oven-dried soil expressed as calcium carbonate or as another specified form of carbonate, when it is known to be the main form present;

d) details of any operation not specified in this International Standard or regarded as optional, as well as any factor which may have affected the results.

Annex A (informative)
Results of an interlaboratory trial for the determination of the carbonate content of soils

An interlaboratory trial was organized in 1993 by the Wageningen Agricultural University (Netherlands), to verify the procedure specified in this International Standard.

For this interlaboratory trial, the determination of the carbonate content of five soils was carried out by nine laboratories.

The summary of the results of the interlaboratory trial is presented in Table A.1.

The repeatabilities (*r*) and the reproducibilities (*R*) listed in Table A.1 were calculated according to ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests*.

Table A.1 — Results of the interlaboratory trial for the determination of the carbonate content

Soil No.	Origin	Mean Value g/kg	r	R	n
1	Great Britain	662	31,8	94,8	9
2	France	540	28,4	55,0	9
3	France	158	7,6	18,6	8
4	Netherlands	66	3,8	19,2	9
5	Argentina	1	2,0	3,3	9
n = number of laboratories retained after eliminating outliers.					

Annex B (informative)
Bibliography

[1] ALLISON, L.E. Wet combustion apparatus and procedure for organic and inorganic carbon in soils. *Soil Science Society of America Proc.* **24** (1960), pp. 36-40.

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