SLOVENSKI STANDARD
SIST-TS CEN/TS 16375:2013

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Sredstva za apnjenje - Določevanje količine preostalih fino mletih karbonatov v tleh - Volumetrijska metoda

Liming materials - Determination of the amount of residual finely ground carbonate in soils - Volumetric method

Calcium-/Magnesium-Bodenverbesserungsmittel - Bestimmung der Menge feingemahlener Carbonatrückstände in Böden - Volumetrisches Verfahren

Amendements minéraux basiques - Détermination de la teneur en carbonate résiduel finement broyé dans les sols - Méthode volumétrique

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ICS:

65.080 Gnojila Fertilizers

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TECHNICAL SPECIFICATION

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CEN/TS 16375

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ICS 65.080

English Version

Liming materials - Determination of the amount of residual finely ground carbonate in soils - Volumetric method

Amendements minéraux basiques - Détermination de la teneur en carbonate résiduel finement broyé dans les sols - Méthode volumétrique

Calcium-/Magnesium-Bodenverbesserungsmittel - Bestimmung der Menge feingemahlener Carbonatrückstände in Böden - Volumetrisches Verfahren

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Foreword

This document (CEN/TS 16375:2013) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

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1 Scope

This Technical Specification specifies a method for the determination of low contents (as < 5 g $CaCO_3$ per kilogram) of carbonate in soil samples. It applies to any type of carbonate liming material, such as limestone, chalk, and dolomite.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

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):

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

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

The volume of the carbon dioxide produced is measured with a measuring burette, and is compared with the volume of gas produced by increasing amounts of calcium carbonate added to test portions. To avoid making corrections for differences in temperature and pressure, all determinations are carried out under the same conditions, with a very strict control of ambient conditions and determination timing, e.g. air-conditioned room and water bath, short time during which the variation of atmospheric pressure is supposed to be constant.

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 EN ISO 3696).

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

Dilute 340 ml of 37 % hydrochloric acid in water (4.1) and then fill up to 1 000 ml with water (4.1).

4.3 Sulfuric acid, (H_2SO_4) solution 95 % to 97 %.

4.4 Sodium sulfate, (Na_2SO_4), powder, purity higher than 99 %.

4.5 Internal liquid of the calcimeter, 50 g/l of sodium sulfate (4.4) and 50 ml/l of sulfuric acid (4.3).

4.6 Calcium carbonate, ($CaCO_3$), powder, purity higher than 99 %.

5 Apparatus

5.1 Apparatus for the volumetric measurement of produced gas, inspired of Bernard calcimeter according to Figure 1.

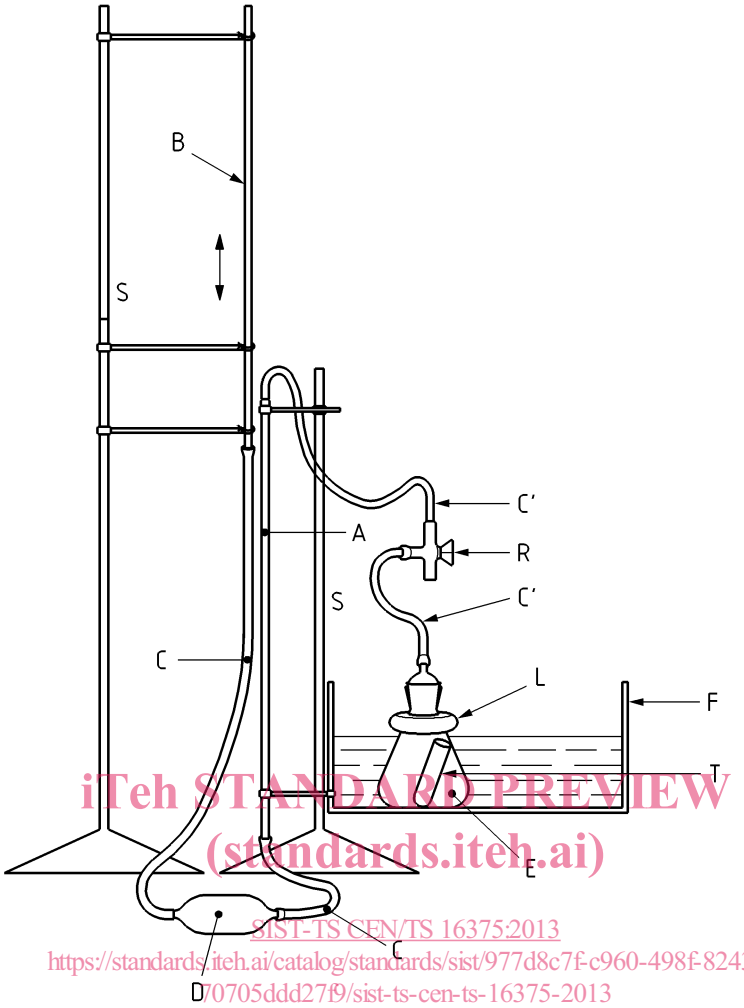
The apparatus is composed of a glass tube of 20 ml graduated every 0,02 ml. The total length shall not exceed 80 cm. Another non graduated glass tube with the same length and the same diameter is connected to the first glass tube with a transparent flexible pipe of about 1 m. A small volume of reserve of 50 ml can be inserted right at the inferior end of the graduated tube. The higher end of the graduated tube is connected by a standard flexible pipe to the Erlenmeyer flask (5.2) of attack.

The graduated tube is fixed vertically on a stand.

The non-graduated tube is held on a second stand, parallel to the first, but so that it is vertically mobile. Mark the tube to approximately 2 cm of its lower end.

The two tubes shall only be 1 mm or 2 mm away from each other.

The whole apparatus shall be dipped several hours in a surface-active cleaning liquid to eliminate any trace on the interior surface of the tubes which could hinder circulation and modify the levelling of the internal liquid. Rinse with demineralised water after treatment.



Key

A glass tube of 20 ml graduated every 0,02 ml
B non-graduated tube
C flexible pipe
C' standard flexible pipe
D bulb of 50 ml
E 200 ml Erlenmeyer flask with finely ground, gas tight glass stopper
F water bath
L metallic ring
R 3 ways tap
S stand
T reaction tube (with hydrochloric acid)

Figure 1 — Calcimeter

5.2 200 ml Erlenmeyer flask, with finely ground, gas tight glass stopper.

Erlenmeyer flasks of attack, with a capacity of 200 ml, are closed by a finely ground glass cup. The upper part of the cap is connected by 200 mm of standard flexible pipe to a 3 ways tap of polytetrafluoroethylene, PTFE), which is also connected to the flexible pipe coming from the graduated tube.

The waterproof of the glass cap is ensured by a thin silicone-grease layer.

5.3 Water bath, the volume of water shall be of at least 5 l and maintain at room temperature.

5.4 Metallic ring.

The Erlenmeyer flask of attack (5.2) is ballasted with a metallic ring and is kept in the water bath.

5.5 Thermo stated room.

The whole device is placed in a thermo stated room of a great thermal inertia or fit with a device of cold-hot regulation which ensures an uninterrupted constant temperature to ± 1 °C. Check the room temperature changes close to the device for 1 h.

The analysis may only start when the water bath temperature is equal to the room temperature.

Absence of important atmospheric pressure variation during the analysis shall be checked to the regional weather services.

5.6 Glass tubes, capacity of 20 ml.

Each tube shall have a length such to allow that:

— it can be vertically introduced into the Erlenmeyer flask using a finger,

— it can be maintained in sub-vertical position in the Erlenmeyer flask without its contents pours out,

— its contents can be completely emptied in the Erlenmeyer flask by inclining the Erlenmeyer flask at an angle lower than 90°.

5.7 Thermometer, with an accuracy of 0,2 °C.

5.8 Analytical balance, with an accuracy of 0,01 mg.

5.9 Stop watch.

6 Procedure

6.1 Preparation of the apparatus

6.1.1 Fill the apparatus with the internal liquid (4.5). Use a small funnel connected by a flexible pipe at the higher end of the non-graduated tube. Eliminate air bubbles.

6.1.2 Fill the tube until the liquid level match with the first higher graduation (position zero) of the graduated tube and the mark traced in the lower part of the non-graduated tube.

6.1.3 Make sure that the two meniscuses' positions are on the same horizontal level (the apparatus is not connected to the Erlenmeyer flask at this point).

6.1.4 Make the non-graduated tube slide downwards. The volume of liquid then goes down the graduated tube. Check, in several points of volume, the horizontal coincidence of the meniscuses.

6.1.5 Reset the meniscuses to position zero.

6.1.6 Lay the water bath close to the device.

6.1.7 Let the whole device stabilize thermically for 0,5 h.

6.1.8 Check that the water bath temperature is equal to the room temperature.