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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 CaCO₃ 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):

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

$$H_2CO_3 \rightarrow H_2O + CO_2$$
 (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(HCI) = 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, (Na₂SO₄), 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**, (CaCO₃), 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.