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Method Validation for Determination of Calcium Carbonate in Soil by Neutralization Titration

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Abstract

This paper discusses the verification of the method for the determination of calcium carbonate in soil by neutralization titration method. The laboratory has verified the determination of calcium carbonate in soil by three groups of actual soil samples by neutralization titration method, and the precision is in line with "Determination of Calcium Carbonate in Forest Soil".

Keywords

Calcium Carbonate; Method Validation.

1. INTRODUCTION

Calcium carbonate, also known as limestone and stone powder, is a common chemical substance on the earth. It is an inorganic salt mineral. It is alkaline, insoluble in water, and easily soluble in acid. It naturally exists in aragonite, calcite, chalk, limestone, marble, etc. In limestone and other rocks, insoluble calcium carbonate in the limestone layer and water dissolved in carbon dioxide can be converted into calcium bicarbonate, and water dissolved in calcium bicarbonate can also precipitate calcium carbonate, which is the reason for the formation of karst caves. Calcium is necessary to maintain the normal function of human nerve, muscle, skeletal system, cell membrane and capillary permeability. The main component of animal bones or shells is calcium carbonate. Calcium carbonate used in medicinal and edible additives is prepared from calcite. Since it has the effect of calcium supplementation after purification, sterilization, filtration and refining, it needs to be used in large quantities in the process of preparing penicillin, streptomycin and oxytetracycline. .

2. MAIN INSTRUMENTS AND REAGENT CONSUMABLES

Tall beaker (100 mL), percent balance, digital bottle top titrator

Hydrochloric acid (HCl), sodium hydroxide (NaOH), phenolphthalein

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3. EXPERIMENTAL PROCESS

Weigh 3.0~10.0 g of air-dried soil samples (containing 0.2~0.4 g of calcium carbonate) that pass through a 0.149 mm sieve, put them in a 100 mL high beaker, add 20.00 mL of 0.5 mol/L hydrochloric acid standard solution with a pipette, cover Take a watch glass, stir with a glass rod, and drive off the carbon dioxide produced. After cooling, transfer it into a 100 mL volumetric flask, rinse the soil and beaker with cold water 5 to 6 times, and set the volume.

Draw 50 mL of the clear solution into a 150 mL conical flask, add 2 drops of phenolphthalein indicator, drop the remaining hydrochloric acid with 0.25 mol/L sodium hydroxide standard solution, and titrate to a clear red color, which does not stop in about 1 min. until it fades. Note the number of liters of standard sodium hydroxide solution used.

4. RESULTS

4.1. Verification of Precision

The laboratory analyzed and measured three levels of actual soil samples, and measured them according to all the steps of sample analysis. Each sample was measured 6 times in parallel. The data summary table is shown in Table 1, and the detailed process data is shown in the original record sheet in the attachment. The average, standard deviation, relative standard deviation and other parameters of different samples were calculated respectively. Calculate the standard deviation and relative standard deviation according to formulas 1 and 2, respectively.

$$S=\sqrt{\frac{\sum(Xi-\bar{X})^2}{N-1}} \quad (1)$$

$$RSD（\%）=\frac{S}{\bar{X}}\times 100\% \quad (2)$$

Where: S—standard deviation;

$\bar{X}$- the average value of the measurement results;

X_i—the result of a single measurement;

N—measurement times;

RSD—relative standard deviation.

Table 1. Method precision test data table

Parallel No.		Specimen 1	Specimen 2	Specimen 3
The measurement results (g/kg)	1	27.1	79.5	102.0
	2	26.3	76.5	107.3
	3	27.1	77.9	109.5
	4	26.1	79.2	105.1
	5	27.7	77.3	102.1
	6	25.6	78.6	106.1
Average (g/kg)		26.6	78.2	105.3
Standard deviation s (g/kg)		0.72	1.06	2.70
Relative standard deviation RSD (%)		2.70	1.35	2.56
Absolute deviation (g/kg)		0~1.1	0.1~1.5	0.1~3.7

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4.2. Verification of Actual Samples

According to the supplementary requirements for the evaluation of ecological environment monitoring institutions for the accreditation of inspection and testing institutions, the requirements of Article 17: According to the scope of application of the standard, no less than one actual sample is selected for measurement.

Soil samples were selected for testing, and parallel samples were added as required. All samples were tested in accordance with all the steps of the analysis. The data summary table is shown in Table 2, and the detailed sampling, process data, quality control evaluation, and actual sample testing report, etc., see the original records in the attachment one.

Table 2. Actual sample test data

Parallel No.	Detected Value (g/kg)	Evaluation	Requiremen	Evaluation of Results
I2022001-001S	69.9	Absolute deviation1.0g/kg	Absolute deviation10 ~2 g/kg	Pass
I2022001-002S	68.0			

Conclusion: The verification experiment was carried out on the actual sample, the calcium carbonate content was 10-100 g/kg, and the absolute deviation was 10-2 g/kg, which met the requirements of "Determination of Calcium Carbonate in Forest Soil" (LY/T 1250-1999) 3 Neutralization titration method measurement requirements.

5. CONCLUSION

5.1. Precision

Three levels of actual soil samples were analyzed and determined by neutralization titration. The absolute deviations of calcium carbonate in the soil ranged from 0 to 1.1 g/kg, 0.1 to 1.5 g/kg, and 0.1 to 3.7 g/kg. Determination of Calcium Carbonate in Forest Soil (LY/T 1250-1999) 3 In neutralization titration method, calcium carbonate content>100 g/kg, absolute deviation>10 g/kg, calcium carbonate content 10~100 g/kg, absolute Deviation 10 ~ 2 g/kg, calcium carbonate content <10 g/kg, absolute deviation <2 g/kg determination requirements.

5.2. Actual Samples

The verification experiment is carried out on the actual sample, the calcium carbonate content is 10~100 g/kg, and the absolute deviation is 10~2 g/kg, which meets the determination of "Determination of Calcium Carbonate in Forest Soil" (LY/T 1250-1999) 3 Neutralization titration method Require.

The laboratory verified the determination of calcium carbonate in soil by three groups of actual soil samples through neutralization titration method, and the precision met the requirements of "Determination of Calcium Carbonate in Forest Soils" (LY/T 1250-1999) 3 neutralization titration method. The laboratory has the ability to detect calcium carbonate in soil.

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