

Method Validation for Determination of Exchangeable Acidity in Soils

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Abstract

The laboratory analyzed and determined the same type of forest soil samples at three content levels. The relative standard deviations of exchangeable acidity, exchangeable hydrogen and exchangeable aluminum in the soil were 2.26%, 2.31%, 1.72%, 10.06%, and 4.61%, respectively. %, 5.12%, 4.02%, 7.70%, 5.42%, and the actual samples were verified by the experiment, and the relative deviations of the parallel samples of exchangeable acidity, exchangeable hydrogen and exchangeable aluminum of soil samples were 1.30%, 1.03%, and 1.75%, respectively. All of them meet the determination requirements of "Determination of Soil Exchangeable Acidity by Potassium Chloride Extraction - Titration Method" (HJ 649-2013) when the measured value is ≤ 10.0 mmol/kg, and the maximum allowable relative deviation is $\pm 20\%$. It shows that the laboratory has the ability to detect the exchangeable acidity in soil.

Keywords

Exchangeable Acidity; Method Validation; Soils.

1. Introduction

The soil is extracted with a neutral salt solution, and the hydrogen ions and aluminum ions adsorbed on the soil colloid are exchanged to enter the solution, and then titrated with a standard solution of sodium hydroxide. The titration result is called exchangeable acidity [1-3].

2. Main Instruments and Reagent Consumables

Digital bottle-top titrator (16F99488, German Brand); percent balance (BSA2202S-CW, Sartorius); pH meter (B524041976, Mettler Toledo Instruments Co., Ltd.).

Potassium chloride (Sinopharm Group Chemical Reagent Co., Ltd.); Sodium Fluoride (Tianjin Fuchen Chemical Reagent Factory); Sodium Hydroxide (Tianjin Damao Chemical Reagent Factory).

3. Experimental Process

3.1. Sample Pretreatment

Weigh 5.00g of air-dried 2mm soil sample, put it in the funnel that has been covered with filter paper, and rinse it with potassium chloride solution several times in a small amount. . The filtrate was placed in a 250 mL volumetric flask, and the volume was fixed with 1.0 mol/L potassium chloride solution when it was close to the mark.

3.2. Determination of Exchangeable Acidity

Pipette 100 mL of the sample extract into a beaker, boil for 5 min to volatilize carbon dioxide that may exist in the solution, cool to room temperature, use pH as an indicator, and titrate with sodium hydroxide solution to pH 7.80 ± 0.08 , Record the number of milliliters consumed by volume V_1 of sodium hydroxide solution.

For the blank test, titrate 100 mL of the blank sample extract at the same time with the above method, and record the consumption of the volume V of sodium hydroxide solution.

3.3. Determination of Exchangeable Hydrogen

Pipette 100 mL of sample extract into a beaker, add 2.5 mL of sodium fluoride solution, boil for 5 min, remove carbon dioxide, cool to room temperature, use pH as an indicator, and titrate with sodium hydroxide solution to pH 7.80 ± 0.08 , record the number of milliliters of sodium hydroxide solution volume V_2 consumed.

For the blank test, titrate 100 mL of the blank sample extract simultaneously with the above method, and record the number of milliliters of sodium hydroxide solution volume V_0 consumed.

4. Results Analysis

4.1. Method Precision

The laboratory analyzed and measured the same type of forest soil samples with three content levels in Chenggu County, Hanzhong City with task number 2022026, and each sample was measured 6 times in parallel. The data summary table is shown in Table 4, and the detailed process data is shown in the original appendix record sheet. 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.

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

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

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 Sign		Exchangeable Acidity			Exchangeable Hydrogen			Exchangeable Aluminum		
		1	2	3	1	2	3	1	2	3
Results mmol/kg	1	1.60	2.81	6.89	0.55	1.39	2.84	1.05	1.41	4.05
	2	1.61	2.86	7.12	0.59	1.49	2.99	1.02	1.37	4.13
	3	1.68	2.94	6.99	0.63	1.29	2.91	1.05	1.65	4.08
	4	1.59	2.98	6.93	0.47	1.35	3.08	1.12	1.63	3.85
	5	1.66	2.83	7.02	0.59	1.38	2.73	1.07	1.45	4.30
	6	1.61	2.87	7.22	0.61	1.41	2.71	1.00	1.46	4.51
Average Value \bar{x} mmol/kg		1.62	2.88	7.03	0.57	1.39	2.88	1.05	1.49	4.15
Standard Deviation s mmol/kg		0.037	0.066	0.121	0.058	0.064	0.147	0.042	0.115	0.225
Relative Standard Deviation %		2.26	2.31	1.72	10.06	4.61	5.12	4.02	7.70	5.42
Relative standard deviation of standard requirements%		<20								
Whether the method requirements are met		Satisfy								

4.2. Determination of Actual Samples

Table 2. Actual sample test data table

Indicator Name	Sample Name	Detection value	Evaluation	Require	Result Evaluation
Exchangeable Acidity	Actual Sample(mmol/kg)	2.22	The relative standard deviation is 1.30%	When the measured value is less than or equal to 10.0 mmol/kg, the maximum allowable relative deviation is $\pm 20\%$	Qualified parallel sample test
	Parallel(mmol/kg)	2.18			
Exchangeable Hydrogen	Actual Sample(mmol/kg)	1.40	The relative standard deviation is 1.03%		
	Parallel(mmol/kg)	1.38			
Exchangeable Aluminum	Actual Sample(mmol/kg)	0.83	The relative standard deviation is 1.75%		
	Parallel(mmol/kg)	0.81			

According to the requirements of Article 17 of the Supplementary Requirements for the Evaluation of Ecological Environment Monitoring Institutions for the Qualification of Inspection and Testing Institutions: 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 determined according to all the steps of the analysis. The data summary table is shown in Table 2.

5. Conclusion

Precision: The laboratory analyzed and determined the same type of forest soil samples with three content levels in Chenggu County, Hanzhong City, task number 2022026. The relative standard deviations of exchangeable acidity, exchangeable hydrogen, and exchangeable aluminum in the soil were 2.26%, 2.31%, 1.72%, 10.06%, 4.61%, 5.12%, 4.02%, 7.70%, 5.42%, all meet the requirements of "Determination of Soil Exchangeable Acidity by Potassium Chloride Extraction - Titration Method" (HJ 649-2013) When the measured value is less than or equal to 10.0 mmol/kg, the maximum allowable relative deviation is $\pm 20\%$.

Actual sample: The verification experiment was carried out on the actual sample. The relative deviations of the parallel samples of exchangeable acidity, exchangeable hydrogen and exchangeable aluminum of soil samples were 1.30%, 1.03% and 1.75%, respectively, which met the requirements of "Determination of Soil Exchangeable Acidity of Potassium Chloride". Extraction-titration method (HJ 649-2013) when the measured value is less than or equal to 10.0 mmol/kg, the maximum allowable relative deviation is $\pm 20\%$.

It shows that the laboratory has the ability to detect the exchangeable acidity in soil.

References

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