基于分光光度法测定土壤阳离子交换量的方法优化及开发

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摘要：为准确测定土壤阳离子交换量，在《土壤 阳离子交换量的测定 三氯化六氨合钴浸提-分光光度法》（HJ 889-2017）的基础上，对方法进行优化和开发。采用超声浸提代替传统的振荡浸提，确定了在 40 mL 浸提液中加入 2.8 g 土壤样品，用 1 mol/L 氢氧化钠调节溶液 pH 至 8±0.5，超声浸提 10 min，用滤纸过滤代替离心，1.66 cmol/L$[Co(NH_3)_6]Cl_3$溶液直接作为空白样品的实验条件。验证该实验条件和测试方法。结果表明，该方法在 0～1.49 cmol/L 范围内具有良好的线性，线性系数为 1；方法检出限为 0.7 $cmol^+/kg$，方法定量下限为 2.8 $cmol^+/kg$；10 种土壤标准物质的测定结果均在认证值范围内，相对标准偏差（n=6）在 0.28%～2.68%，具有良好的精密度和准确度。超声浸提-分光光度法测定土壤阳离子交换量准确可靠，操作过程简单，提高了工作效率，可应用于批量土壤阳离子交换量样品的检测。

关键词:土壤；阳离子交换量；超声浸提；分光光度法；三氯化六氨合钴

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Optimization and development of a method for the determination of soil cation exchange based on spectrophotometry

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Abstract： In order to accurately determine the soil cation exchange amount, the method was optimized and developed on the basis of " Soil quality-Determination of cation exchange capacity （CEC）-Hexamminecobalt trichloride solution-Spectrophotometric method " (HJ 889-2017). Using ultrasonic extraction instead of the traditional oscillatory extraction, It was determined that 2.8 g of soil samples were added to 40 mL of leaching solution, the pH of the solution was adjusted to 8±0.5 with 1 mol/L sodium hydroxide, ultrasonic extraction was performed for 10 min, and filter paper was used instead of centrifugation. 1.66 cmol/L$[Co(NH_3)_6]Cl_3$ solution was directly used as the experimental condition of blank sample. Validate the experimental conditions and test methods. The results show that the method has good linearity in the range of 0~1.49 cmol/L, the linear coefficient is 1; the detection limit of the method is 0.7 $cmol^{+}/kg$, and the lower limit of quantification of the method is 2.8 $cmol^{+}/kg$; The determination results of 10 soil reference materials were all within the certified value range, Relative standard deviation (n=6) in 0.28%～2.68%, showing good precision and accuracy. The ultrasonic extraction-spectrophotometry method is accurate and reliable for the determination of soil cation exchange capacity, the operation process is simple, the work efficiency is improved, and it can be applied to the detection of batch soil cation exchange capacity samples.

Keywords：soil; Cation exchange capacity; Ultrasonic extraction; Spectrophotometry; Hexammine cobalt trichloride

阳离子交换量(Cation Exchange Capacty，简称 CEC)，是指土壤胶体所能吸附的各种阳离子的总量，以每千克土壤的厘摩尔数表示($cmol^{+}/kg$)阳离子交换量的大小[1]。阳离子交换量是土壤缓冲性能的主要来源，它可以调节土壤溶液的浓度，保证土壤溶液成分的多样性，保持土壤溶液的生理平衡，防止各种养分被雨水淋溶，测定土壤阳离子交换量不仅可以合理施肥、改良土壤，而且对研究污染物的环境行为具有重要意义[2-5]。

常用的土壤阳离子交换量的测定方法有乙酸铵交换法和三氯化六氨合钴浸提-分光光度法，乙酸铵交换法所需试剂和配置溶液较多、样品预处理步骤繁琐、样品测定周期长，且样品预处理和测定过程全程序对分析人员素质要求较高,容易造成系统误差。三氯化六氨合钴浸提-分光光度法使用化学试剂少，操作步骤简单方便，实验过程安全可靠，检测效率高，但该测定方法存在浸提液用量大，酸性样品测定值低，样品吸光度高于空白样品，吸光度值不稳定，检测精度差等问题[6]。本实验以三氯化六氨合钴浸提-分光光度法为基础，采用超声浸提代替传统振荡浸提，讨论了影响土壤阳离子测定的因素，选择了最佳测定条件，优化了实验流程，缩短了检测时间，提高了检测效率，使测定结果更加准确可靠，以期为土壤阳离子交换量的测定提供新的参考。

1 实验部分

1.1 仪器和试剂

紫外可见分光光度计、电子天平、超声波清洗器、pH 计、Milli-Q 超纯水系统纯水仪。