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Soil quality - Determination of lead, cadmium – Graphite furnace atomic absorption spectrophotometry

土壤质量 铅、镉的测定

石墨炉原子吸收分光光度法

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Soil quality - Determination of lead, cadmium – Graphite furnace atomic absorption spectrophotometry

1 Subject content and scope of application

1.1 This Standard specifies the graphite furnace atomic absorption spectrophotometry for determining the lead and cadmium in the soil.

1.2 The detection limit (calculated by digesting 0.5g of test piece to the constant volume of 50mL) of this Standard is as follows: 0.1 mg/kg, for both lead and cadmium.

1.3 USE the Zeeman method, self-absorption method and deuterium lamp method to deduct the background. In the presence of a matrix modifier such as diammonium hydrogen phosphate or ammonium chloride, directly DETERMINE the trace lead and cadmium in test solution. There is no interference.

2 Principle

USE the hydrochloric acid - nitric acid - hydrofluoric acid - perchloric acid full digestion method to completely destroy the mineral lattices in soil so that all the elements to be tested in the test pieces can enter into the test solution. INJECT the test solution into a graphite furnace afterwards. EVAPORATE and REMOVE the coexisting matrix components by a predetermined temperature rising program such as drying, ashing and atomization. In the meantime, DISSOCIATE the lead and cadmium compounds into ground-state atomic vapors at high temperature in the atomization phase. Furthermore, the selective emission of the characteristic spectrum of the hollow cathode lamp is induced. Under the optimal determination condition, DETERMINE the absorbance of the lead and cadmium in test solution by means of background subtraction.

3 Reagents

Unless otherwise specified, the analytical reagents and deionized water or water of equivalent purity, complying with national standards, are used as reagents in this Standard.

3.1 Hydrochloric acid (HCl), $\rho = 1.19$ g/mL, guaranteed reagent.

remove silicon. In order to achieve a good silicon flying effect, it is necessary to frequently SHAKE the crucible. KEEP on heating until there is white and dense perchloric acid smoke coming out of the crucible. COVER the lid so that black organic carbides can fully decompose. WAIT until the black organics on the crucible disappear. OPEN the lid. DRIVE the white smoke. EVAPORATE until the contents become viscous. Based on the digestion condition, it is allowed to add 2mL of nitric acid (3.2), 2mL of hydrofluoric acid (3.5) and 1mL of perchloric acid (3.6) again. REPEAT the above-mentioned digestion process. When the white smoke basically exhausts and the contents become viscous again, REMOVE the crucible for slight cooling. USE water to flush the crucible cover and inner wall. ADD 1mL of nitric acid solution (3.3) to warm the dissolved residuals. TRANSFER the solution into a 25mL volumetric flask. ADD 3mL of aqueous diammonium hydrogen phosphate solution (3.7) afterwards. After cooling, DETERMINE the constant volume. SHAKE evenly for standby measurement.

Since there are various kinds of soil, there are major differences between the contents of organic matters. During digestion, PAY attention to observe. It is allowed to increase and decrease the dosage of various kinds of acid according to the digestion condition. The soil digestion solution shall be white or faint yellow (soil with higher iron content). There shall be no significant precipitates.

Note: The temperature of the electric hot plate shall not be too high. Otherwise, the teflon crucible will be out of shape.

6.2 Determination

ADJUST the instrument to optimal operating conditions according to the instrument instruction manual. DETERMINE the absorbance of the test solution afterwards.

6.3 Blank test

USE water to replace the test pieces. USE the same procedures and reagents as Section (6.1). PREPARE the whole process blank solution. DETERMINE in accordance with the procedure (6.2). PREPARE at least two portions of blank solution in each batch of samples.

6.4 Calibration curve

Respectively TRANSFER 0.00mL, 0.50mL, 1.00mL, 2.00mL, 3.00mL and 5.00mL of standard lead and cadmium working mixture (3.10) into a 25mL volumetric flask. ADD 3.0mL of aqueous diammonium hydrogen phosphate solution (3.7) afterwards. USE nitric acid solution (3.4) to determine the constant volume. The lead contents in this standard solution shall be respectively 0µg/L, 5.0µg/L, 10.0µg/L, 20.0µg/L, 30.0µg/L and 50µg/L. The

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