

ICS 13.080



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 17141-1997

**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

(English Translation)

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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.

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), $\rho = 1.19 \text{ g/mL}$, guaranteed reagent.

3.2

Nitric acid (HNO₃), $\rho = 1.42 \text{ g/mL}$, guaranteed reagent.

3.6

Perchloric acid (HClO₄), $\rho = 1.68 \text{ g/mL}$, guaranteed reagent.

3.7

Aqueous solution of diammonium hydrogen phosphate [(NH₄)₂HPO₄] (guaranteed reagent), with a weight fraction of 5%.

3.8

Standard lead stock solution, 0.500 mg/mL. Accurately WEIGH 0.5000g (ACCURATE to 0.0002g) of spectral pure metallic lead. PUT it in a 50mL beaker. ADD 20mL of nitric acid solution (3.3). Slightly HEAT the solution for dissolution. After cooling, TRANSFER the solution into a 1,000mL volumetric flask. ADD water to the scale. SHAKE evenly.

3.9

Standard cadmium stock solution, 0.500 mg/mL. Accurately WEIGH 0.5000g (ACCURATE to 0.0002g) of spectral pure metallic cadmium particles. PUT them in a 50mL beaker.

3.10

Standard lead and cadmium working mixture, where lead is 250 µg/L and cadmium is 50 µg/L. Before use, USE nitric acid solution (3.4) to dilute and prepare the standard lead and cadmium stock solution (3.8) (3.9) step by step.

4 Instruments

4.1

General laboratory instruments and the following instruments.

4.2

Graphite furnace atomic absorption spectrophotometer (with background subtraction device).

4.3

Lead hollow cathode lamp.

4.4

Cadmium hollow cathode lamp.

4.7 Instrument parameters

The optimal testing conditions for different types of instruments are different. It is allowed to select by yourselves according to the instrument instruction manual. For the measurement conditions generally adopted in this Standard, SEE Table 1.

5 Samples

Evenly MIX the soil samples (generally not less than 500g) taken. USE the quartering method to divide the samples to 100g or so. CONDUCT air drying (natural air drying or freeze drying) to the divided soil samples. REMOVE the foreign matters in the soil, such as stones, animal and plant residues, etc. USE a wooden stick (or an agate pestle) for milling.

6.1 Test solution preparation

Accurately WEIGH 0.1g to 0.3g (ACCURATE to 0.0002g) of test pieces. PUT them in a 50mL teflon crucible. After wetting by using water, ADD 5mL of hydrochloric acid (3.1). HEAT the test pieces on an electric hot plate in a fume cupboard at low temperature for preliminary decomposition.

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.

7 Result representation

The lead and cadmium contents W [Pb (Cd), mg/kg] in soil sample shall be calculated according to the following equation.

8 Precision and accuracy

Many laboratories use this method to analyze the precision and accuracy of the lead and cadmium in the ESS series of soil samples. SEE Table 2.

Annex A (Normative) Determination of the water content in soil sample

A.1 WEIGH 5g to 10g (ACCURATE to 0.01g) of air-dried soil sample passing through an 100 mesh sieve. PLACE the soil sample in an aluminum box or weighing bottle. DRY in a drying oven at 105°C for 4h to 5h, until the soil sample comes up to constant weight.

A.2 The water content in air-dried soil sample w expressed as percentage shall be calculated according to the following equation.