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# Soil quality - Determination of nickel - Flame atomic absorption spectrophotometry

土壤质量 镍的测定 火焰原子吸收分光光度法

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# Soil quality - Determination of nickel - Flame atomic absorption spectrophotometry

## 1 Subject content and scope of application

**1.1** This standard specifies the flame atomic absorption spectrophotometric method, for the determination of nickel in soil.

**1.2** The detection limit of this standard (calculated on the basis of weighing 0.5 g of specimen and distilling it to 50 ml) is 5 mg/kg.

**1.3** Interference

**1.3.1** When using the 232.0 nm line as the absorption line, there are three nickel lines with very close wavelength distances, which shall be overcome, by selecting a narrower spectral passband.

**1.3.2** The 232.0 nm line is in the ultraviolet region. The light scattering and molecular absorption, which are caused by salt particles and molecular compounds, are relatively serious, which will affect the measurement. Use background correction to overcome this type of interference. If the concentration permits, the method of diluting the test solution can also be used, to reduce background interference.

## 2 Principle

The method of hydrochloric acid-nitric acid-hydrofluoric acid-perchloric acid full decomposition is used, to completely destroy the mineral lattice of the soil, so that all the elements to be measured in the specimen enter the test solution. Then, the soil digestion solution is sprayed into the air-acetylene flame. At the high temperature of the flame, the nickel compound dissociates into ground state atoms; the ground state atom vapor selectively absorbs the characteristic spectral line 232.0 nm, which is emitted by the magnesium hollow cathode lamp. Under the optimal measurement conditions selected, the absorbance of nickel is measured.

## 3 Reagents

The reagents used in this standard, unless otherwise specified, use analytically pure reagents and deionized water or water of equivalent purity, that meet national standards.

**3.1** Hydrochloric acid (HCl), $\rho = 1.19$ g/ml, superior grade.

# 5 Samples

Mix the collected soil samples (generally not less than 500 g). Divide them into about 100 g, by quartering method. After the divided soil samples are air-dried (naturally air-dried or freeze-dried), remove stones, animal and plant residues and other foreign matter in the soil samples. Use wooden sticks (or agate sticks) to grind and press, to make it pass through a 2 mm nylon sieve (to remove grit more than 2 mm). Mix well. Use an agate mortar to grind the soil samples, which passed through the 2 mm nylon sieve, until all the soil samples pass through the 100 mesh (0.149 mm aperture) nylon sieve. Mix well to prepare for use.

# 6 Analytical procedures

**6.1** Preparation of test solution

Accurately weigh 0.2 ~ 0.5 g (accurate to 0.0002 g) of the specimen, in a 50 ml polytetrafluoroethylene crucible. Add 10 ml of hydrochloric acid (3.1), after wetting it with water. Place it on the electric heating plate in the fume hood, to heat it at low temperature, to decompose the sample initially. When it evaporates to about 3 ml left, take it off and cool slightly. Then add 5 ml of nitric acid (3.2), 5 ml of hydrofluoric acid (3.5), 3 ml of perchloric acid (3.6). After adding the cover, heat it on the electric heating plate at medium temperature for about 1 hour. Then open the cover. Continue heating to remove silicon. In order to achieve a good effect of silicon removal, shake the crucible frequently. When heated to the generation of thick perchloric acid white smoke, cover it to decompose the black organic carbon. After the black organic matter on the crucible wall disappears, open the lid. Drive off the white smoke. Steam until the contents are viscous. Depending on the digestion situation, add another 3 ml of nitric acid (3.2), 3 ml of hydrofluoric acid (3.5), 1 ml of perchloric acid (3.6). Repeat the above digestion process. When the white smoke is exhausted again and the contents are viscous, take it out and cool it slightly. Use water to rinse the inner wall and the crucible lid. Add 1 ml of nitric acid solution (3.3) to warm to dissolve the residue. Then transfer the whole amount into a 50 ml volumetric flask. After cooling, make the volume reach to the mark. Shake well. Prepare for measurement.

Since there are many types of soils and the organic matter contained in it varies greatly, it shall be carefully observed during digestion. The consumption of various acids can be increased or decreased, according to the digestion situation. The soil digestate shall be white or light yellow (for soils with high iron content), with no obvious sediment.

Note: The temperature of the heating plate should not be too high; otherwise, the PTFE crucible will be deformed.

**6.2** Determination

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