DETERMINATION OF NICKEL IN FERTILIZERS

Principle of method

Nickel is extracted from an ammoniacal solution as its dimethylglyoxime complex, and determined spectrophotometrically at 450 or 480 nm.

Reagents

Hydrochloric acid, approx. 6N.: Mix 1 volume of concentrated hydrochloric acid (about 36% w/w; 11N) with 1 volume of water.

Hydrochloric acid, approx. 2N.: Mix 1 volume of concentrated hydrochloric acid (about 36% w/w; 11N) with 5 volumes of water.

Nitric acid, approx. 6N.: Mix 1 volume of concentrated nitric acid (about 70% w/w; 16N) with 2 volumes of water.

Chloroform: Shake 500 mL of chloroform with 50 mL of 10% w/V HCl solution; take the lower chloroform layer into another separating funnel and wash it with water until it is free from acid.

Saturated bromine solution

Ammonia solution, concentrated .: 35% w/w (18 N)

Diluted ammonia solution.: Mix 1 volume of concentrated ammonia solution (about 35% w/w; 18N) with 19 volumes of water.

Diluted hydrochloric acid.: Mix 1 volume of concentrated hydrochloric acid (about 36% w/w; 11N) with 19 volumes of water.

Sodium citrate solution.: 25% w/v solution

Disodium dimethylglyoxime solution.: 0.2% w/V solution in diluted ammonia solution.

Standard nickel solution .: 1.002 g pure metallic nickel is dissolved in concentrated nitric acid (about 70% w/w; 16N) by boiling. 2 mL of concentrated sulphuric acid (about 98% w/w (36 N) is added and the mixture is evaporayed until white fumes of sulfur trioxide are evolved, cooled and diluted the solution to 1 litre at 20^{o}C with distilled water. Dilute 5.0 mL of this solution to 500 mL at 20^{o}C with water; 1 mL ≡ 10 µg of nickel, Ni.

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Instrumentation

Temperature controlled oven
Silica basins; Silica watch glasses
Hot plate
VIS Spectrophotometer

Sample Preparation

The sample must be ground finely to pass a sieve of 16 mesh (1 mm^2 holes). Moisture determination should be carried out on the sample "as received" and again on the sample after grinding.

Organic fertilizers: 5 g of dried sample is ashed (in a silica basin with silica clock glass cover) overnight at about 450^{o}C. The residue is cooled and treated with 10 mL diluted HCl (6N) and evaporate to dryness on a water bath. The soluble salts is extracted from the residue by two successive 10 mL portions of boiling diluted HCl (2N) decanting the solution through the same filter paper into a 50-mL volumetric flask. 5 mL diluted HCl (6N) and 5 mL of dliluted HNO3 (6N) are added to the residue and evaporated to dryness on a hot plate. The soluble parts are taken into solution with 10 mL boiling HCl(2N), filtered through the former paper into the volumetric flask. The solution is diluted to the mark with distilled water.

Inorganic fertilizers: 5 g sample is extracted directly by diluted HCl (6N) and subsequently is extracted repeately with boiling diluted HCl (2N). The extracts are filtered through the same paper into a 50-mL volumetric flask. If excess acid is required to effect solution, the dilution of the final solution should be more than 50 mL to get a analytical solution 1 N acidity with respect to HCl.

Determination of nickel

A suitable aliquot of the acid solution from the preparation of the sample is measured into a beaker (containing not more than 100 µ g of Ni) and diluted to 100 mL with water and 10 mL of sodium citrate solution is added. The solution is made alkaline (litmus paper) with ammonia solution with slight excess.

The solution is transferred to a 250 mL separating funnel. 10 mL of disodium dimethylglyoxime solution is added. The solution is shaken for 1 minute and left to stand for 10 minutes. The solution is shaken for 1 minute after adding 10 mL of chloroform. The chloroform phase is separated. The extraction is repeated twice with 10 mL of chloroform portions. The chloroform extracts are combined.

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The chloroform extract is shaken vigorously after adding 15 mL of diluted HCl. After the separation of aqueous layer, the washing of the chloroform is repeated with 5 mL of diluted HCl and then with 5 mL distilled water. Acid extracts are combined in a 100-mL beaker and is heated on a hot plate (cupboard) to expel any chloroform, and is continued to boil until the volume is reduced to approx. 25 mL. The solution is cooled and transferred to a 50-mL volumetric flask.

To the solution in the flask are added the chemicals in order, mixing after each addition, a) 2 mL of sodium citrate solution; b) 2 mL of bromine water until there is a marked yellow colour; wait 10 minutes, c) just enough ammonia solution to destroy the bromine colour and 1 mL of excess ammonia solution; d) 4 mL of disodium dimethylglyoxime solution. Make up the solution to the mark with water.

After 15 minutes measure the absorbance of the solution at 450 or at 480 nm. against water as blank. Use 4- or 5-cm cells.

Calibration Graph

Measure appropriate amounts of nickel Standard solution, covering the range 0 to 100 µg of Ni, into a series of 50-mL volumetric flasks. To each add 20 mL of dluted HCl and proceed as for the test solution. Measure the absorbances of solutions, and constract a graph relating the absorbances to the microgrammes of Ni. Molar absorptivity : $\varepsilon_{450}= 14x10^3$

Evaluation of the data

From a previously prepared calibration graph, read the number of milli(micro)grammes of nickel equivalent to the observed absorbances of the blank and test solutions and calculate the amount of nickel in the sample.

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