International Standard 7110

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Ammonium bicarbonate (Ammonium hydrogencarbonate) for industrial use (including foodstuffs) — Determination of lead content — Flame atomic absorption method

Bicarbonate d'ammonium (Hydrogénocarbonate d'ammonium) à usage industriel (y compris les industries alimentaires) — Dosage du plomb — Méthode par spectrométrie d'absorption atomique dans la flamme

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Foreword

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INTERNATIONAL STANDARD ISO 7110-1985 (E)

Ammonium bicarbonate (Ammonium hydrogencarbonate) for industrial use (including foodstuffs) — Determination of lead content — Flame atomic absorption method

1 Scope and field of application

This International Standard specifies a flame atomic absorption spectrometric method for the determination of the lead content of ammonium bicarbonate (ammonium hydrogencarbonate) for industrial use (including foodstuffs).

The method is applicable to products containing more than 0,1 mg/kg of lead.

2 Principle

Dissolution of a test portion, acidification with hydrochloric acid and elimination of carbon dioxide. Complexing and extraction of the lead with a solution of diethylammonium diethyldithiocarbamate in xylene.

Aspiration of the solution into an acetylene-air flame. Measurement of the absorption of the 217 nm line or, alternatively, of the 283,3 nm line emitted by a lead hollow-cathode lamp.

3 Reagents and materials

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

3.1 Hydrochloric acid, ϱ approximately 1,19 g/ml, 38 % (m/m) approximately, containing not more than 0,005 mg of lead per kilogram.

3.2 Xylene, mixed isomers, ϱ 0,860 to 0,870 g/ml.

WARNING — Xylene is flammable and toxic by inhalation. Avoid contact with skin and eyes.

3.3 Diethylammonium diethyldithiocarbamate, 10 g/l solution in xylene.

Dissolve 2,5 g of diethylammonium diethyldithiocarbamate $[(C_2H_5)_2N\text{-}CSSNH_2(C_2H_5)_2]$ in 250 ml of the xylene (3.2).

3.4 L-Ascorbic acid, (ascorbic acid) 100 g/l solution.

Dissolve 10 g of ascorbic acid in water and dilute to 100 ml.

Prepare this solution at the time of use for each series of tests.

3.5 Lead, standard solution corresponding to 1,000 g of lead per litre.

Weigh, to the nearest 0,001 g, 1,600 g of lead(II) nitrate $[Pb(NO_3)_2]$, previously dried at 105 °C and cooled in a desiccator, and place in a beaker of suitable capacity. Dissolve in a small quantity of water to which has been added 1 ml of nitric acid solution, ϱ approximately 1,40 g/ml. Transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 1 mg of Pb.

3.6 Lead, standard solution corresponding to 0,005 g of Pb per litre.

Take 5,00 ml of the standard lead solution (3.5), place in a 1 000 ml one-mark volumetric flask, add 1 ml of nitric acid solution ϱ approximately 1,40 g/ml, dilute to the mark and mix.

1 ml of this standard solution contains 5 µg of Pb.

Prepare this solution at the time of use for each series of tests.

3.7 Acetylene, compressed (for example from a cylinder).

3.8 Air, compressed (for example from a cylinder).

3.9 3,3'-Dibromophenolsulfonphtaleine (Bromophenol red), indicator paper.

4 Apparatus

For this determination, use lead-free glassware, which has been cleaned by washing thoroughly with nitric acid solution and then rinsing with water.

Ordinary laboratory apparatus and

4.1 Atomic absorption spectrometer, fitted with a burner fed with acetylene and air.

4.2 Lead hollow-cathode lamp.

5 Procedure

5.1 Test portion

Weigh, to the nearest 0,1 g, 25 g of the laboratory sample.

If a preliminary test shows a lead content above the range covered by the calibration graph, reduce the mass of the test portion accordingly.

5.2 Preparation of the calibration graph

5.2.1 Preparation of the calibration solutions.

Into each of a series of seven 250 ml separating funnels, place 9 ml of the hydrochloric acid (3.1), about 150 ml of water and 5 ml of the L-ascorbic acid solution (3.4).

Then add the volumes of the standard lead solution (3.6) indicated in the following table.

Standard lead solution (3.6)	Corresponding mass of Pb
ml	µg
0*	0
1,0	5
2,0	10
3,0	15
4,0	20
5,0	25
6,0	30

* Calibration blank reagent test.

Treat the contents of each separating funnel as follows. Dilute to about 200 ml with water, add 5,0 ml of the diethylammonium diethyldithiocarbamate solution (3.3), stopper and shake well for about 2 min. Allow the phases to separate, discard the aqueous phase and collect the organic phase in a small beaker of 10 to 20 ml capacity.

NOTE — It is not necessary to collect the organic phase quantitatively.

5.2.2 Adjustment of the apparatus (4.1) fitted with the lamp (4.2)

Switch on the apparatus (4.1) for a sufficient time beforehand to achieve stability. Adjust the wavelength in the region of 217 nm or, alternatively, in the region of 283,3 nm, and also the attenuation and the slit to suit the characteristics of the apparatus. Adjust the air and acetylene pressures according to the characteristics of the aspirator and the burner, so as to give an oxidizing flame when xylene is being aspirated at a rate of 2 to 3 ml/min.

5.2.3 Spectrometric measurements

Aspirate the series of calibration solutions (5.2.1) successively into the flame and measure the absorbance for each. Take care to keep the rate of aspiration constant throughout the preparation of the calibration graph.

NOTE — Aspirate xylene through the burner after each measurement.

5.2.4 Plotting the calibration graph

Plot a graph having, for example, the number of micrograms of lead introduced during the preparation of the calibration solutions as abscissae and the corresponding values of the measured absorbances as ordinates.

5.3 Determination

5.3.1 Preparation of the test solution

Dissolve the test portion (5.1) in water and add the hydrochloric acid (3.1) until the colour of the indicator paper (3.9) changes from yellow to red. Add an excess of 9 ml of the hydrochloric acid, heat gently in order to eliminate most of the carbon dioxide, then cool. Transfer the solution quantitatively into a separating funnel of capacity 250 ml, add 5 ml of the L-ascorbic acid solution (3.4), dilute, if necessary, to about 200 ml with water and mix. Then add 5,0 ml of the diethylammonium diethyldithiocarbamate solution (3.3). Stopper the funnel and shake well for about 2 min. Allow the phases to separate, discard the aqueous phase and collect the organic phase in a small beaker.

5.3.2 Spectrometric measurements

5.3.2.1 Preliminary measurement

Carry out a preliminary measurement on the test solution (5.3.1) using the procedure specified in 5.2.3 and in conjunction with the spectrometric measurements on the calibration solutions (5.2.1).

NOTE — Use for this measurement the minimum quantity of test solution.

5.3.2.2 Bracketed measurement

Carry out a second measurement on the test solution (5.3.1) in conjunction with measurements on two bracketing solutions, the concentrations of which do not differ by more than 2 µg of Pb.

Prepare these bracketing solutions using the conditions specified in 5.2.1 and using appropriate quantities of the standard lead solution (3.6).

6 Expression of results

The mass of lead, m_1, in the test solution, expressed as micrograms of Pb, is given by the equation

$$m_1 = m_2 + (m_3 - m_2) \times \frac{A_0 - A_1}{A_2 - A_1}$$

where

m_2 is the mass, in micrograms, of Pb contained in the weaker bracketing solution;

m_3 is the mass, in micrograms, of Pb contained in the stronger bracketing solution;

A_0 is the absorbance of the test solution (5.3.1);

A_1 is the absorbance of the weaker bracketing solution;

A_2 is the absorbance of the stronger bracketing solution.

The lead content, expressed as milligrams of Pb per kilogram, is given by the formula

$$\frac{m_1}{m_0}$$

where m_0 is the mass, in grams, of the test portion (5.1).

7 Test report

The test report shall include the following particulars:

a) an identification of the sample;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard, or regarded as optional.