International Standard 6678

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Phosphoric acid for industrial use — Determination of lead content — Atomic absorption spectrometric method

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Foreword

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It has been approved by the member bodies of the following countries :

Australia	Germany, F.R.	Poland
Austria	Hungary	Portugal
Belgium	India	Romania
Brazil	Italy	South Africa, Rep. of
China	Korea, Rep. of	Switzerland
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Egypt, Arab Rep. of	Netherlands	United Kingdom
France	Philippines	USSR

No member body expressed disapproval of the document.

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Phosphoric acid for industrial use — Determination of lead content — Atomic absorption spectrometric method

1 Scope and field of application

This International Standard specifies an atomic absorption spectrometric method for the determination of lead in phosphoric acid for industrial use.

The method is applicable to phosphoric acids having lead contents greater than 0,1 mg/kg.

2 Reference

ISO 4285, *Phosphoric acid for industrial use — Guide to sampling techniques.*

3 Principle

Extraction of the lead from a diluted test portion into a solution of diethylammonium diethyldithiocarbamate in xylene. Separation of the xylene extract and aspiration of the extract into the acetylene/air flame of an atomic absorption spectrometer.

4 Reagents and materials

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

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

4.2 Xylene.

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

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

Dissolve 2,5 g of diethylammonium diethyldithiocarbamate in 250 ml of the xylene (4.2).

4.4 Ascorbic acid, 100 g/l solution.

Prepare immediately before each series of tests.

4.5 Lead, standard solution corresponding to 1,4 g of lead per litre.

Weigh, to the nearest 0,000 1 g, 0,559 8 g of lead nitrate $Pb(NO_3)_2$, and dissolve in about 100 ml of water. Transfer the solution quantitatively to a 250 ml one-mark volumetric flask. Dilute to the mark and mix.

1 ml of this standard solution contains 1,4 mg of Pb.

4.6 Lead, standard solution corresponding to 7 mg of lead per litre.

Transfer 5,00 ml of the standard lead solution (4.5) to a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix.

1 ml of this standard solution contains 7 µg of Pb.

Prepare this solution on the day of use.

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

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

5 Apparatus

Ordinary laboratory apparatus, which shall be of lead-free glass and previously washed with hot dilute hydrochloric acid solution and water, and

5.1 Atomic absorption spectrometer, fitted with a lead hollow-cathode lamp and an acetylene/air burner.

6 Procedure

6.1 Test portion

Weigh, to the nearest 0,005 g, 2,5 g of technical grade phosphoric acid or 25,0 g of phosphoric acid of high purity (see ISO 4285).

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

6.2 Blank test

At the same time as the determination, carry out a blank test, following the same procedure and using the same quantities of reagents as for the determination, but omitting the test portion.

6.3 Preparation of calibration graph

6.3.1 Preparation of calibration solutions

Into each of six stoppered 250 ml separating funnels, place 9 ml of the hydrochloric acid solution (4.1), approximately 150 ml of water and 5 ml of the ascorbic acid solution (4.4). Add to each funnel the volumes of the standard lead solution (4.6) given in the following table.

Standard lead solution (4.6)	Corresponding mass of lead
ml	µg
0,00*	0
1,00	7
2,00	14
3,00	21
4,00	28
5,00	35

* Blank test on the reagents for calibration.

Dilute each solution to 200 ml with water. Add 5,00 ml of the diethylammonium diethyldithiocarbamate solution (4.3). Stopper each funnel and shake for 30 s. Allow the layers to separate, discard the aqueous layer and transfer the xylene layer to a 10 ml glass beaker.

Complete recovery of the xylene layer is not necessary.

6.3.2 Spectrometric measurements

Set up the spectrometer (5.1) as described in the appropriate instruction manual and allow to stabilize. Adjust the wavelength to about 217 nm or, alternatively, to about 283 nm, and set the sensitivity and slit width according to the characteristics of the instrument. Adjust the air and acetylene pressures according to the requirements of the aspirator and the burner, so as to give a gently oxidizing flame when xylene is being aspirated at a rate of 4 to 5 ml/min.

Aspirate into the flame the xylene solution obtained from each calibration solution and measure the absorbance. Ensure that the aspiration rate is constant throughout the series of measurements. Aspirate xylene after each measurement.

6.3.3 Plotting the graph

Plot a graph having, for example, the masses, in micrograms, of lead (Pb) in the calibration solutions as abscissae, and the corresponding values of absorbance, corrected for the absorbance of the blank test on the reagents for calibration, as ordinates.

6.4 Determination

6.4.1 Preparation of the test solution

Dissolve the test portion (6.1) in approximately 150 ml of water. Add 9 ml of the hydrochloric acid solution (4.1). Transfer the solution quantitatively to a 250 ml separating funnel, and add 5 ml of the ascorbic acid solution (4.4). Dilute to 200 ml with water and mix. Add 5,00 ml of the diethylammonium diethyldithiocarbamate solution (4.3) and shake for 30 s. Isolate the xylene layer as described in 6.3.1

6.4.2 Spectrometric measurements

Measure the absorbances of the extracts from the test solution and from the blank test solution following the procedure specified in 6.3.2 starting from the second paragraph "Aspirate into the flame the xylene solution . . .".

7 Expression of results

The lead content, expressed in milligrams per kilogram, is given by the formula

$$\frac{m_1 - m_2}{m_0}$$

where

m_1 is the mass, in micrograms, of lead corresponding to the absorbance of the test solution;

m_2 is the mass, in micrograms, of lead corresponding to the absorbance of the blank test solution;

m_0 is the mass, in grams, of the test portion (6.1).

8 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 in the standard to which reference is made, or regarded as optional.