IS 7017 (1973): Method of colorimetric determination of traces of heavy metals by dithizone [CHD 1: Inorganic Chemicals]

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Indian Standard

METHOD FOR COLORIMETRIC DETERMINATION OF TRACES OF HEAVY METALS BY DITHIZONE

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INDIAN STANDARDS INSTITUTION
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Indian Standard
METHOD FOR COLORIMETRIC DETERMINATION OF TRACES OF HEAVY METALS BY DITHIZONE

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Indian Standard

METHOD FOR COLORIMETRIC DETERMINATION OF TRACES OF HEAVY METALS BY DITHIZONE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 16 August 1973, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

0.2 Heavy metals when present in traces may be determined as lead. This standard prescribes the determination of heavy metals (as lead) by dithizone extraction. This provides a most satisfactory means of separating traces of heavy metals and determining them as lead. When interfering elements are known to be present, the method should be suitably modified.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS:2-1960*.

1. SCOPE

1.1 This standard prescribes the colorimetric method of determination of traces of heavy metals (as lead) by dithizone method.

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise; 'pure chemicals' and distilled water (*see* IS:1070-1960†) or demineralized water (demineralized through ion exchange method) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

3. APPARATUS

3.1 Nessler Cylinders — 50 ml capacity (*see* IS:4161-1967‡).

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*Rules for rounding off numerical values (*revised*).

†Specification for water, distilled quality (*revised*).

‡Specification for Nessler cylinders.

4. REAGENTS

4.1 Standard Lead Solution — Dissolve 0·40 g of lead nitrate [$Pb(No_3)_2$] in water containing 2 or 3 ml of concentrated nitric acid and make up the volume to 1 000 ml with water. Transfer 10 ml of this solution to a volumetric flask, add 2 or 3 ml of concentrated nitric acid and dilute with water to 1 000 ml. One millilitre of this solution contains 2·5 μg of lead (as Pb). The diluted solution shall be freshly prepared.

4.2 Dilute Ammonium Hydroxide — 1 N, approximately.

4.3 Reagent A — Dissolve 25 g of triammonium citrate or 22 g of citric acid and 4 g of hydroxylamine hydrochloride in about 200 ml of water. Add dilute ammonium hydroxide to bring the *p*H to 8·5. Dilute the solution to 500 ml. Purify this solution by extracting with 15 ml portions of 0·01 percent dithizone solution until the final colour of the dithizone extract is green. Wash the aqueous remainder portion three times with 25 ml portions of chloroform and finally with one 25 ml portion of carbon tetrachloride.

4.4 Dithizone (Diphenyl thiocarbazone or Phenylazothionoformic Acid) Solution — Dissolve 0·01 g of dithizone in 100 ml of carbon tetrachloride, shaking intermittently for 1 hour. Allow to stand overnight and shake once again before using. This shall be kept in a cool and dark place. This gives a 0·01 percent solution. Filter, if necessary. Dilute 10 ml of this solution to 100 ml with carbon tetrachloride in a 100-ml volumetric flask. This shall be prepared fresh before determination. This gives a solution of 0·001 percent.

NOTE 1. — Carbon tetrachloride used should be further purified. One litre of carbon tetrachloride is extracted with two portions of 25 ml of dilute ammonium hydroxide and then kept over 100 g of activated carbon. Before use, it is decanted and distilled at about 80°C over a little fresh lime.

NOTE 2. — Sometimes dithizone solid and its 0·01 percent solutions deteriorate on storage. The 0·01 percent solution should, therefore, be tested before further dilution, by shaking 2 ml of the solution with 5 ml of 1 percent ammonium hydroxide. If the organic layer is only faintly yellow under these conditions, the solution may be used. If it is deeply coloured it shall be discarded and fresh solution be prepared. The solution as well as the reagent should be stored in a refrigerator and exposure to sunlight should be avoided during analytical work. To increase the stability of 0·01 percent solution, it should be covered with a thin aqueous layer saturated with sulphur dioxide.

4.5 Thymol Blue Indicator Solution — 1 percent (*m*/*v*) solution in rectified spirit.

5. PROCEDURE

5.1 Take several aliquots of standard lead solution into a series of separating funnels, add 5 ml of water and 10 ml of the reagent A. Then add

2 drops thymol blue indicator and bring the pH of the solution to 8·5 by addition of dilute ammonium hydroxide. Add 5 ml of the dithizone solution and shake well for about 10 seconds. Drain the organic layers into stoppered Nessler cylinders.

5.1.1 Prepare the solution of the material as prescribed in individual material specification. Take a suitable aliquot of the prepared solution, and transfer to a separating funnel. Develop the colour as prescribed in 5·1. Drain the organic layer into a stoppered Nessler cylinder, add 10 ml of dilute ammonium hydroxide, and shake for about 10 seconds. Compare the colour developed with those of the standard solutions. Note the volume of the standard with which the colour of the test solution matches.

NOTE — If the colour of the test solution is intermediate between two standard solutions, then the experiment is repeated by taking more number of standard solutions in that range and exact colour matching is arrived at.

5.2 Calculation

$$\text{Heavy metals (as Pb), percent by mass} = \frac{100 \times V \times f}{M}$$

where

V = volume in ml of standard lead solution matching with the test solution,

f = mass in g of heavy metals (as Pb) equivalent to 1 ml of standard lead solution, and

M = mass in g of the material in the aliquot taken for the test.

INDIAN STANDARDS
ON
CHEMICAL REAGENTS AND GENERAL METHODS OF TESTS

IS:	
2088-1971	Methods for determination of arsenic (*first revision*)
2263-1962	Methods of preparation of indicator solutions for volumetric analysis
2316-1968	Methods of preparation of standard solutions for colorimetric and volumetric analysis (*first revision*)
2317-1963	Method for gravimetric determination of sulphates
2362-1963	Determination of water by the Karl Fischer method
3225-1965	Methods for preparation of buffer solutions
4016-1966	Density-composition tables for aqueous solutions of sodium hydroxide
4048-1966	Density-composition tables for aqueous solutions of sulphuric acid
4284-1967	Method for volumetric determination of iron
4285-1967	Method for volumetric determination of calcium
4542-1968	Colorimetric methods for determination of iron
4730-1968	Method for determination of density of liquids
5194-1969	Method for determination of nitrogen-Kjeldhal method
5298-1969	Method for determination of distillation range and of distillation yield
5305-1969	Method for volumetric determination of phosphorus
5741-1970	Methods for determination of *p*H
5762-1970	Methods for determination of melting point and melting range
5813-1970	Method for determination of crystallizing point
5949-1970	Methods for volumetric determination of calcium and magnesium using EDTA
6361-1971	Methods for colorimetric determination of phosphorus
7017-1973	Method for colorimetric determination of traces of heavy metals by dithizone