भारतीय मानक

जल और अपशिष्ट जल के नमूने लेने (भौतिक एवं रसायनिक) तथा परीक्षण पद्धतियाँ

भाग 47 सीसा

(पहला पुनरीक्षण)

Indian Standard

METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER

PART 47 LEAD

(First Revision)

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BUREAU OF INDIAN STANDARDS MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG NEW DELHI 110002

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Price Group 4

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Environmental Protection Sectional Committee had been approved by the Chemical Division Council.

Lead is a serious cumulative body poison. Natural waters seldom contain more than 20 μ g/l, although values as high as 400 μ g/l have been reported. Lead in a water supply may come from industrial, mine and smelter discharges or from the dissolution of old lead plumbing. Tap waters that are soft, acidic and not suitably treated may contain lead resulting from an attack on lead service pipes. It is toxic and therefore, a stringent limit has been specified for lead in potable water.

Also, lead is to be specially tested when pollution/plumbo solvency is suspected. Therefore, the test for lead is essential. These tests serve to determine whether the lead content of potable water and waste water is within the acceptable limit or not.

In the preparation of this standard, considerable assistance has been derived from American Standard Test Methods (ASTM Annual Book Section 11, 1983) and Analytical Chemical Acta, 164 (1984) 1-21.

The composition of the technical committee responsible for the formulation of this Indian Standard is given in Annex A.

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

Indian Standard

METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER

PART 47 LEAD

(First Revision)

1 SCOPE

This standard prescribes the following three methods for determination of lead:

*

- a) Atomic absorption method (Direct),
- b) Atomic absorption method (Chelation Extraction), and
- c) Differential Pulse Anodic Stripping Voltammetry (DPASV).

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No. Title

- 7022 (Part 1): Glossary of terms relating to 1973 water sewage and industrial effluents: Part 1
- 7022 (Part 2): Glossary of terms relating to 1979 water, sewage and industrial effluents: Part 2

3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 7022 (Part 1): 1973 and IS 7022 (Part 2): 1979 shall apply.

4 APPLICATION

Depending upon the concentration range and interference levels, choice of the method is made. When the concentration levels are below $500 \mu g/l$, pre-concentration is carried out either by chelation and extraction prior to atomic absorption spectrophotometer (AAS) or by deposition on a mercury drop electrode as in DPASV method. For dissolved lead content, filtration through 0.45 μ m membrane filter is required.

5 SAMPLING AND PRESERVATION

The sampling bottles should be cleaned thoroughly with dilute nitric acid (6 N) prior to final

rinsing with water. The water samples should be collected and stored preferably in polypropylene or chemically resistant glass containers. For preservation, the samples should be acidified with concentrated nitric acid (2 ml of AR grade nitric acid in 1 litre of the sample just to bring down the pH to below 2). For dissolved lead filter the sample in the field and acidify the filtrate with nitric acid to a pH of 2 or lower.

NOTE — Avoid excess nitric acid. Add 5 ml of 0.1 N iodine solution to avoid losses of volatile organo lead compounds during handling and digestion of samples.

6 PURITY OF REAGENTS

6.1 Unless otherwise specified, only AR grade chemicals should be used for all the tests.

6.2 Lead free distilled water should be used for preparing standards, and reagent solution.

7 ATOMIC ABSORPTION METHOD (DIRECT)

7.1 Principle

The lead content of the sample is determined by directly aspirating the sample into the flame of an atomic absorption spectrophotometer.

This method is applicable in the range from 1.0 to 10.0 mg/l of lead. However, the concentration range will vary with the sensitivity of the instrument used.

7.2 Interferences

Other metals usually do not interfere. However, high concentrations of calcium do interfere and give high values for lead. In these cases the chelation-extraction procedure should be used. Background correction should be applied.

7.3 Apparatus

7.3.1 Atomic absorption spectrophotometer with air-acetylene flame.

7.3.2 Hollow-cathode lamps or electrodeless discharge lamps for use at 283.3 nm.

7.4 Reagents

7.4.1 Hydrochloric Acid — Concentrated.

7.4.2 Nitric Acid - Concentrated.

7.4.3 Nitric Acid - Diluted (1:499).

7.4.4 Lead Solutions

7.4.4.1 Stock lead solution

Dissolve 1.599 9 g of lead nitrate in a mixture of 10 ml or concentrated nitric acid and 100 ml of water and dilute to 1 litre (1 ml = 1.0 mg of Pb).

7.4.4.2 Standard lead solution

Dilute 100 ml of lead stock solution to l litre with dilute nitric acid (1:499)(1 ml = 0.1 mg of Pb).

7.5 Procedure

7.5.1 To 100 ml portion of the acidified sample add 0.5 ml of nitric acid, 5 ml of concentrated hydrochloric acid and heat it not to boil but to reduce the volume to 20 ml in a well-ventilated hood. Cool and filter the sample and make up to 100 ml in a standard flask. Aspirate the sample solution and measure the absorbance at 283.3 nm. Aspirate nitric acid (1:499) prior to sample aspiration.

7.5.2 Prepare a reagent blank and sufficient standards containing 1.0, 2.5, 5.0, 7.5 and 10.0 mg/l of lead by diluting suitable volume of the standard solution with nitric acid (1:499) and repeat as above. Aspirate the solutions and measure the absorbance.

7.6 Calculation

Construct a standard calibration graph by plotting the absorbance versus mg of lead concentration of each standard. Read the concentration of the sample from the graph.

Lead,
$$(mg/1) = \frac{M}{V} \times 1000$$

where

M = mass o lead present in mg in the sample, and

V = volume of sample in ml.

8 ATOMIC ABSORPTION METHOD (CHELATION – EXTRACTION)

8.1 Principal

Lead is chelated with pyrrolidine dithiocarbamic acid and extracted with chloroform. The extract is treated with hot nitric acid after

evaporating to dryness, meated with hot hydrochloric acid and diluted with water to a specified volume. An aliquot is aspirated into the air-acetylene flame of the spectrophotometer. For total recoverable lead an acid digestion procedure is done prior to chelation or aspiration.

This method is applicable for concentration range from 100-1 000 μ g/l of lead.

NOTE — The lower range of determination to the extent of 0.001 mg/l may be obtained by graphite system.

8.2 Interferences — Same as in 7.2.

8.3 Apparatus — Same as in 7.3.

8.4 Reagents

8.4.1 Hydrochloric Acid — Concentrated.

8.4.2 Hydrochloric Acid — Diluted (1:2).

8.4.3 Hydrochloric Acid — Diluted (1:49).

8.4.4 Nitric Acid — Concentrated.

8.4.5 Pyrrolidine Dithiocarbamic Acid Chloroform Reagent

36 ml of pyrrolidine is mixed with 1 litre of chloroform. The solution is cooled and 30 ml of carbon disulphide is added in small fractions with continuous stirring. Dilute with two litres of chloroform and store in a cool and dark place. The reagent is stable for atleast six months.

NOTE — As components of this mixture are highly toxic and flammable, prepare and use in a fumehood.

8.4.6 Sodium Hydroxide Solution

Dissolve 100 g of sodium hydroxide in water and dilute to 1 litre with water.

8.4.7 Chloroform

8.4.8 Bromophenol Blue Indicator Solution

Dissolve 0.1 g of bromophenol blue in 100 ml of 50 percent ethanol or *iso*propanol.

8.4.9 Lead Solutions

8.4.9.1 Stock lead solution

Dissolve 1 599 9 g of lead nitrate in a mixture of 10 ml of concentrated nitric acid and 100 ml of water and dilute to 1 litre (1 ml = 1.0 mg of Pb).

8.4.9.2 Standard lead solution

Dilute 100 ml of lead stock solution to 1 litre with dilute nitric acid (1:499)(1 ml = 0.1 mg of Pb).

8.4.10 Water Saturated Chloroform

Mix one part of chloroform with one part of water in a separatory funnel. Shake 30 times and let separate. Discard aqueous layer. Save chloroform layer.

8.5 Procedure

8.5.1 For dissolved lead, filter 100 ml of the sample through 0.45 μ m membrane filter paper. For total lead, add 5 ml of concentrated hydrochloric acid and evaporate the solution to 15 to 20 ml. Cool and filter the sample through acid washed filter paper. Make up to 100 ml in a volumetric flask. Add to this solution or the filtrate (in case of dissolved lead) 2 drops of bromophenol blue indicator solution and mix. Adjust the pH by adding sodium hydroxide solution till a blue colour persists. Add diluted hydrochloric acid (1:49) drop by drop until the colour just disappears; then add 2.5 ml in excess to bring the pH to 2.3-2.5. Add 10 ml of pyrrolidine dithiocarbamic acid - chloroform reagent and shake well. After the phases separate out, collect the chloroform phase by taking care to avoid any trace of water in the flask. Repeat the extraction till the chloroform layer becomes colourless with fresh 6 to 7 ml portion of chloroform; combine the extracts and make up the volume to 25 ml. Aspirate the organic extracts directly into the the flame (zeroing the instrument on a water saturated chloroform blank) and record absorbance. To avoid problems associated with instability of extracted metal complexes. immediately, determine after extraction. Alternatively evaporate the extracts just to dryness and dissolve the residue by dropwise addition of 2 ml of concentrated nitric acid by holding the beaker at an angle. Again evaporate to dryness and add 2 ml of hydrochloric acid (1:2) and heat for 1 minute. Cool and make up the solution in a 10 ml standard flask. Aspirate the sample and measure the absorbance.

8.5.2 Prepare a reagent blank and sufficient standards containing 100, 200, 400, 500, 700, 900 and 1 000 $\mu g/l$ of lead by diluting a suitable volume of the standard solution with 100 ml of water and repeat as above. Aspirate the solution and measure the absorbance.

8.6 Calculation

8.6.1 Construct a standard calibration graph by plotting the absorbance versus the micrograms of lead. Read the concentration of the samples from the curve.

Lead,
$$\mu g/l = \frac{M}{V} \times 1000$$

where

- $M = \text{mass of lead present in } \mu g \text{ in the sample,}$ and
- V = volume of sample in ml.

9 DIFFERENTIAL PULSE ANODIC STRIP-PING VOLTAMMETRY (DPASV)

9.1 Principle

Lead is deposited on a hanging mercury drop at a negative (– ve) potential of – 0.6 V versus saturated calomel electrode (SCE). Then the lead is stripped back into the solution by applying a positive (+ ve) potential scan. The anodic current peak which is measured is representative of the lead concentration in the sample. For total dissolved lead the sample is filtered through a 0.45 μ m membrane filter paper prior to acidification and analysis. This method is applicable in the concentration range 1.0 to 100 μ g/1 of lead.

9.2 Interferences

Selenium interferes when it is present in excess of 50 μ g/l. This may be overcome by adding ascorbic acid which reduces selenium (IV) to selenium metal. Iron (III) interferes when present at levels greater than lead. However, this may be overcome by warming the solution with hydroxylamine. Also, the presence of any other neighbouring stripping peaks which is less than 100 mV from that of the lead will interfere.

9.3 Apparatus

9.3.1 Polarographic instrumentation capable of performing differential pulse work.

9.3.2 Hanging Mercury Drop Electrode

9.3.3 Platinum Counter Electrode

9.3.4 Saturated Calomel Reference Electrode

9.3.5 Magnetic Stirrer Control Unit, Stirring Bar

9.4 Reagents

9.4.1 Hydrochloric Acid -- Concentrated.

9.4.2 Nitric Acid — Concentrated.

9.4.3 Nitric Acid — Diluted (1:1).

9.4.4 Lead Solutions

9.4.4.1 Stock lead solution

Dissolve 0.319 8 g of lead nitrate in water containing 1 ml of concentrated nitric acid. Dilute to one litre with water (1 ml = 200 μ g of Pb).

9.4.4.2 Intermediate lead solution

Dilute 10 ml of lead stock solution and 1 ml of nitric acid to one litre with water ($1 \text{ ml} = 2 \mu g$ of Pb).

9.4.4.3 Standard lead solution

Dilute 10 ml of lead intermediate solution and 1 ml of concentrated nitric acid to 100 ml with water ($1 \text{ ml} = 0.2 \ \mu \text{g}$ of Pb).

This solution should be prepared just before use for preparing the working standards.

9.4.5 Amalgamated Zinc

Cover 10 g of granular zinc with water and add 2 drops of concentrated hydrochloric acid. Then add 5 to 8 drops of mercury with continuous shaking.

9.4.6 Purified Nitrogen

Boil 2 g of ammonium meta vanadate with 25 ml of concentrated hydrochloric acid. Dilute to 250 ml and transfer to the scrubber. Add 10 to 15 g of amalgamated zinc. Pass nitrogen gas through the scrubber for removal of traces of oxygen and through distilled water for washing any traces of scrubber chemicals (Fig. 1).

9.5 Procedure

9.5.1 Clean all the glasswares and the voltammetric cells by soaking them overnight in concentrated nitric acid and rinse them thoroughly with distilled water. If total dissolved lead alone is to be determined, the sample should be filtered through 0.45 μ m membrane filter paper. For total recoverable lead, digest the sample with 3 ml each concentrated hydrochloric acid and nitric acid. Evaporate the solution to 15 to 20 mf. Cool and make up to 100 ml in a volumetric flask. Take 10 ml of the sample in the polarographic cell and deaerate for 15 minutes. The cell should be covered with nitrogen gas during the experiment (Fig. 2) Generate a new droplet of mercury and put the stirrer on. Connect the cell and deposit at -0.6 V versus SCE for 3 minutes. Stop the stirrer and wait for 30 seconds. Start the anodic scan with the following settings:

Initial potential	0.6 V vs SCE
Scan rate	5 mV/sec
Scan direction	+ ve
Modulation amplitude	25 m V
Current range	1 — 10 µA
Drop time	0.5 sec
Display direction	ve
Low pass filter	Off position
Mode	Differential pulse
Scan range	-0.6 V to -0.15

Measure the current peak height (I_1). Add 20 μ l of standard lead solution and deaerate for 5 minutes. Repeat as above. Measure the current peak height (I_2).

9.6 Calculation

$$C_{\text{sample}}, \, \mu g/l = \frac{I_1 \, V \, C_{\text{std}} \, \times \, 1 \, 000}{I_2 \, v + (I_2 - I_1) \, V}$$

where

V

- I_1 = current peak height for the sample,
- I_2 = current peak height for the sample + standard,
- $v = volume of standard added (20 <math>\mu$ l),
 - = volume of the sample solution,
- $C_{\rm std}$ = concentration of the standard solution added, and
- $C_{\text{sample}} = \text{concentration of lead in the sample.}$



FIG. 1 SCRUBBER ASSEMBLY FOR NITROGEN PURIFICATION





5

ANNEX A

(Foreword)

COMMITTEE COMPOSITION

Environmental Protection Sectional Committee, CHD 012

Representing

Central Pollution Control Board, Delhi

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Memb**ers**

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DR M. ATCHAYYA (Alternate)

Ministry of Rural Development

Bharat Heavy Electrical Ltd, Hyderabad

National Environmental Engineering Research Institute (CSIR), Nagpur

Indian Council of Agricultural Research, New Delhi National Council for Cement & Building Materials, New Delhi

Shriram Institute for Industrial Research, Delhi

National Thermal Power Corporation Ltd, New Delhi

Central Scientific Instruments Organization (CSIR), Chandigarh

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Jawaharlal Nehru University, New Delhi National Institute of Occupational Health (ICMR), Ahmadabad

Dharmsi Morarji Chemical Co Ltd, Bombay

(Continued on page 7)

(Continued from page 6) Members SHRI P. S. RAMANATHAN SHRI D. N. V. RAO SHRI R. J. BUCH (Alternate)

SHRI K. J. BUCH (Alternate) DR S. ROUTH DR J. C. NUHAWAN (Alternate) SHRI P. R. SAMADDAR SHRI P. K. SEN (Alternate) SHRI S. C. SHARMA SHRI R. N. GUPTA (Alternate) SHRI M. P. SINGH SHRI N. C. TIWARI (Alternate) SHRI M. SUBBA RAO DR T. CHANDINI (Alternate) SHRI R. M. SUNDARAM SHRI C. KRISHNA RAO (Alternate) SHRI R. PARTHASARTHY (Alternate) SHRI R. PARTHASARTHY (Alternate) SUPERINTENDING ENGINEER EXECUTIVE ENGINEER (Alternate) SHRI J. M. TULI SHRI S. N. CHAKRABARTI (Alternate)

D_R R. K. SINGH, Director (Chem) Representing

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National Test House, Calcutta

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National Environment Engineering Research Institute (CSIR), Nagpur

Delhi Water Supply & Sewage Disposal Undertaking, New Delhi

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Central Ground Water Board, New Delhi

Industrial Toxicology Research Centre (CSIR), Lucknow

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This Indian Standard has been developed from Doc: No. CHD 012 (0170

Amendments Issued Since Publication		
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AMENDMENT NO. 1 OCTOBER 2000

TO

IS 3025 (PART 47): 1994 METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER

PART 47 LEAD

(First Revision)

(*Page* 1, *clause* 2) — Insert the following at the appropriate place:

'3025 (Part 1): 1986 Methods of sampling and test (physical and chemical) for water and wastewater : Part 1 Sampling'

(Page 2, clause 7.5.1, line 6) — Insert the words 'using dilute nitric acid' after the words '100 ml'.

(Page 4, clause 9.6) — Substitute the following for the existing formula:

 $C_{\text{sample, mg/l}} = \frac{V_{\text{std}} \times C_{\text{std}}}{V_{\text{sample}}} \times \frac{I_1}{I_2 - I_1}$

(CHD 12)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 2 APRIL 2003 TO

IS 3025 (PART 47): 1994 METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER

PART 47 LEAD

(First Revision)

(Page 1, clause 1) — Substitute the following for the existing:

'1 SCOPE

This standard prescribes the following four methods for determination of lead:

- a) Atomic absorption method (Direct);
- b) Atomic absorption method (Chelation-Extraction);
- c) Differential Pulse Anodic Stripping Voltammetry (DPAV); and
- d) Dithizone method.'

(Page 4, clause 9.6) — Insert the following new clause after 9.6:

'10 DITHIZONE METHOD

10.1 Principle

An acidified sample containing microgram quantities of lead is extracted with dithizone solution in chloroform. The extraction is carried out in the presence of strong ammoniacal citrate-cyanide reducing agent (pH 10 to 11.5). The quantity of lead present in the sample is determined spectrophotometrically by measuring the absorbance at 510 nm of the chloroform extract containing the lead dithizonate complex.

10.2 Minimum Detection Limit

1.0 µg Pb/10 ml dithizone solution (extract).

10.3 Interference

This method uses a high pH, mixed colour and single dithizone extraction. The method is without interference. In strongly ammoniacal citrate-cyanide solution (pH 10 to 11.5) dithizones of Sn (11) and Tl (1) are unstable and extracted only

Amend No. 2 to IS 3025 (Part 47): 1994

partially. Further, a modification of the method allows for detection and elimination of these interferences.

10.4 Apparatus

10.4.1 Spectrophotometer for use at 510 nm with a path length of 1 cm or longer.

10.4.2 pH meter

10.4.3 Standard Volumetric Glasswares

10.4.4 TEF Beaker, 100 ml for Acid Digestion.

10.4.5 Separatory Funnels, 250 ml and 500 ml.

10.4.6 All glasswares are to be cleaned with 1:1 HNO₃, and rinsed thoroughly with distilled water.

10.5 Reagents

10.5.1 Quality of Reagents

Only analytical or equivalent grade reagents, unless specified otherwise, are to be used. All reagents are to be prepared in lead-free distilled water.

10.5.2 Stock Lead Solution

Dissolve 0.159 9 g lead nitrate [(Pb(NO₃)₂, minimum purity, 99.5 percent (w/w)] in about 200 ml of water. Add 10 ml concentrated HNO₃ and dilute to 1 000 ml with water, 1.0 ml of this solution will contain 100 µg of Pb.

10.5.3 Standard Lead Solution

Dilute 2.0 ml of stock lead solution to 100 ml with water, 1.0 ml of this solution will contain 2 μ g of Pb.

10.5.4 Nitric Acid --- Concentrated (18N).

10.5.5 Nitric Acid -Dilute -20 percent, v/v.

10.5.6 Ammonium Hydroxide — Concentrated (14N).

10.5.7 Ammonium Hydroxide - Dilute 10 percent, v/v and 1 percent, v/v.

10.5.8 Citrate-Cyanide Reducing Solution

Dissolve 200 g anhydrous ammonium citrate [(NH4)2 HC6H5O7] 10 g anhydrous sodium sulphite (Na2SO3), 5 g hydroxylamine hydrochloride

(NH₂OH.Hcl), 20 g potassium cyanide (KCN) in water and dilute to 500 ml, and mix with one litre of concentrated NH₄OH .

CAUTION — KCN is a poisonous solution. Handle with extreme care and do not pipette by mouth.

10.5.9 Stock Dithizone Solution

Dissolve 25 mg dithizone in about 50 ml chloroform (CHCl₃) taken in a 200 ml beaker and filter through Whatman No. 42 (or equivalent) filter paper. Collect the filtrate and two washings (10 ml each) in a 250 ml conical flask. Transfer the combined filtrate to a 500 ml separatory funnel. Add about 100 ml 1 percent (ν/ν) NH4OH solution, shake moderately for about 1 min. Transfer the CHCl₃ layer to another 250 ml separatory funnel retaining the orange-red aqueous layer in the 500 ml separatory funnel. Repeat the extraction (of the CHCl₃ layer) with 100 ml of 1 percent (ν/ν) NH4OH solution, transfer the CHCl₃ layer to another 250 ml separatory funnel and the aqueous layer to the original 500 ml separatory funnel containing the first extract. One more repetition, of extraction and transferring to the main aqueous layer is carried out.

To the combined aqueous extract in the 500 ml separatory funnel add 1:1 HCl in 2 ml portions, mixing after each addition, until dithizone precipitation is complete and the solution is no longer orange-red. Extract the precipitated dithizone with three 25 ml portions of CHCl₃. Dilute the combined extract to 250 ml with CHCl₃, 1 ml of this solution will contain 100 μ g of dithizone.

10.5.10 Working Dithizone Solution

Dilute 100 ml stock dithizone solution to 250 ml in a standard volumetric flask with CHCl₃, 1 ml of this solution will contain 40 μ g of dithizone.

10.6 Procedure

10.6.1 Sample Digestion

Digest all samples for dissolved and total lead as per standard digestion procedure using HNO₃H₂SO₄ and HNO₃ - HClO₄.

10.6.2 To 100 ml acidified sample (pH_2) add 20 ml of dilute (20 percent, v/v) HNO₃, filter if required through a filter paper (Whatman No. 41 or equivalent), and transfer it to a 250 ml separatory funnel. Add 60 ml ammoniacal citrate-cyanide solution, mix and cool to room temperature. Add 10 ml of dithizone working solution. Shake the stoppered funnel vigorously for about 30 s, allow to stand (to get two separate layers). Discard 1-2 ml CHCl₃ layer and then fill the absorption

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cell. Measure the absorbance at 510 nm using working dithizone solution as reagent blank.

10.7 Calibration Curve

Plot a calibration curve using at least five standard lead solutions, after adding 50 ml ammoniacal citrate-cyanide solution to the individual lead standard solutions and extracting the same with 10 ml of dithizone working solution.

10.8 Calculation

Same

: :::

mg Pb/litre = $\frac{\mu g Pb (in 10 ml extract obtained from the calibration curve)}{\mu g Pb/litre}$

Volume of sample (ml)

10.9 Precision and Accuracy

Using the dithizone method, lead at the level of 0.026 mg/l can be recovered with 4.8 percent relative standard deviation and 15 percent relative error.

(CHD 12)