

ICS 71.060.01



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 23947.1-2009

**Inorganic chemicals for industrial use -- General method for
determination of arsenic -- Part 1: Silver diethyldithiocarbamate
photometric method**

无机化工产品中砷测定的通用方法 第1部分：二乙基二硫代氨基甲酸银光度法
(English Translation)

Issued on: June 2, 2009

Implemented on: February 1, 2010

Issued by: General Administration of Quality Supervision, Inspection and

Table of Contents

Foreword	3
1 Scope	5
2 Principle	5
3 Reagents	5
4 Apparatus	6
5 Operation steps	8
6 Presentation of results	10
Appendix A (Normative) Use of needle-shaped zinc	11
Appendix B (Informative) Ions that interfere with the determination	12

Foreword

GB/T 23947 Inorganic chemicals for industrial use - General method for the determination of arsenic content is divided into two parts.

-- Part 1: Silver diethyldithiocarbamate photometric method;

-- Part 2: Gutzeit method.

This part is Part 1 of GB/T 23947.

This part was modified in relation to ISO 2590:1973 General method for the determination of arsenic - Silver diethyldithiocarbamate photometric method (English version).

This part was drafted according to ISO 2590:1973. The clauses of this standard correspond to the clauses of the international standard.

Since the international standards cited in ISO 2590:1973 have been abolished, this standard has been modified when adopting ISO 2590:1973. The technical difference is that the abolished international standards are not cited.

For ease of use, this part has made the following editorial changes.

-- In order to be consistent with Chinese inorganic chemical product series standards, "Inorganic chemicals for industrial use" is added to the standard name;

-- The foreword of the international standard is deleted;

-- "This international standard" is replaced with "this standard";

-- The decimal point "." replaces the comma "," that was used as a decimal point;

-- The "Appendix" in the international standard is changed to "Appendix A";

-- Informative Appendix B is added for guidance.

Appendix A of this part is normative and Appendix B is informative.

This part was proposed by China Petroleum and Chemical Industry Association.

This part is under the jurisdiction of the Subcommittee on Inorganic Chemical Industry of the National Technical Committee on Chemical of Standardization Administration of China (SAC/TC63/SC1).

Inorganic chemicals for industrial use - General method for determination of arsenic - Part 1: Silver diethyldithiocarbamate photometric method

1 Scope

This part of GB/T 23942 specifies the general method for the determination of arsenic in inorganic chemicals for industrial use -- the silver diethyldithiocarbamate spectrophotometric method.

This method is applicable to an entire test solution or a test portion taken for determination with arsenic (As) content of 1 µg~20 µg.

2 Principle

The arsenic is reduced to arsine with zinc in a hydrochloric acid medium, and then the arsine is absorbed by a silver diethyldithiocarbamate pyridine solution. The purple-red color produced by the dispersed silver colloid is photometrically measured at the maximum absorption wavelength (about 540 nm). The reaction equation for the formation of silver colloid is as follows.

3 Reagents

All reagents, especially zinc, shall contain no arsenic or very little arsenic. The water used in the experiment shall be distilled water or water of equivalent purity.

3.1

Hydrochloric acid. The density is 1.19 g/mL; the mass fraction is about 38%, or the concentration is 12 mol/L.

3.2

Silver diethyldithiocarbamate [Ag (DDTC)]. 5 g/L pyridine solution.

Dissolve 1 g of silver diethyldithiocarbamate in pyridine (with a density of about 0.980 g/mL), and dilute the solution to 200 mL with the same pyridine. Store it in a sealed brown bottle away from light. The shelf life of this solution is two weeks.

3.3

Arsenic standard solution. 0.100 g/L.

Weigh 0.132 g of arsenic trioxide (As_2O_3), accurate to 0.0001 g, place it in a beaker of appropriate volume (e.g., 100 mL), and dissolve it in about 2 mL of 50 g/L sodium hydroxide solution; transfer all of it to a 1000 mL volumetric flask, rinse the beaker with water several times, transfer the washing liquid to the volumetric flask, dilute with water to the scale, and mix well.

1 mL of this standard solution contains 100 μg of arsenic (As).

3.4

Arsenic standard solution. 2.50 mg/L.

Pipette 25.0 mL of arsenic standard solution (3.3) into a 1000 mL volumetric flask, dilute to the volume, and mix well. This solution shall be prepared before use.

1 mL of this standard solution contains 2.5 μg of arsenic (As).

3.5

Lead acetate absorbent cotton.

Dissolve 50 g of lead acetate trihydrate [$\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$] in 250 mL of water. Soak the absorbent cotton with this solution, remove the excess solution, and dry it under a vacuum at room temperature.

Store it in an airtight container.

3.6

Potassium iodide solution. 150 g/L.

Dissolve 15 g of potassium iodide in water and dilute to 100 mL, then mix well.

3.7

Stannous chloride. hydrochloric acid solution.

Dissolve 40 g of stannous chloride dihydrate ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) in a mixed solution of 25 mL of water and 75 mL of hydrochloric acid (3.1).

3.8 Zinc particles. 0.5 mm~1 mm; or any shape of zinc that has been proven through

tests to produce the same results under the specified test conditions (see Appendix A).

4 Apparatus

Glass containers used for arsenic determinations shall be rinsed with hot concentrated sulfuric acid (be careful when doing this), then rinsed with water and dried completely.

4.1

Glassware with a ground glass joint for arsenic release and total absorption.

The apparatus is shown in Figure 1 and consists of the following parts.

4.1.1

Conical flask. 100 mL; it is used for arsenic release.

4.1.2

Connecting pipe. It is used for absorbing hydrogen sulfide.

4.1.3 15-ball absorber.

4.2

Spectrophotometer.

4.3 Photoelectric absorption meter. It is equipped with a filter with a maximum transmittance at 520 nm~560 nm.

5 Operation steps

WARNING -- Since pyridine is toxic and has a foul odor, it is recommended to handle it with caution in a fume hood.

5.1 Weighing samples and preparing test solutions

Weigh the specimen according to the amount specified in the standard of the product to be tested, and treat the specimen according to the following conditions to obtain the test solution.

In general, the total volume of the test solution prepared or the volume of the test solution taken for determination is 40 mL, containing 1 µg~20 µg of arsenic and 10 mL of hydrochloric acid (3.1). Before the potassium iodide solution is added, the acidity of the test solution shall be 3 mol/L. Nitrate ions must not be present in the test solution.

In addition, certain elements (cobalt, mercury, silver, copper, molybdenum, cesium, etc.) will affect the formation of arsine. If necessary, the operating steps shall indicate how to take into account the effects of their presence.

NOTE: When the test solution is sulfuric acid medium, the acidity of 40 mL test solution shall be 1.9