

## General method for the determination of arsenic – Silver diethyldithiocarbamate photometric method

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# General method for the determination of arsenic – Silver diethyldithiocarbamate photometric method

#### 1 SCOPE

This International Standard specifies a general method for the photometric determination of arsenic, using silver diethyldithiocarbamate.

#### 2 FIELD OF APPLICATION

The method is applicable to the determination of quantities of arsenic (As), contained either in all the test solution or in the aliquot portion taken for the determination, of between 1 and 20  $\mu$ g.

#### **3 PRINCIPLE**

Reduction of the arsenic by zinc in a hydrochloric acid medium, with the formation of arsine.

Absorption of the arsine in a solution of silver diethyldithiocarbamate in pyridine.

Photometric measurement of the purplish-red colour produced by the colloidally dispersed silver at the maximum of the absorption curve (wavelength approximately 540 nm).

NOTE – The reaction of the formation of the colloidal silver is :

 $AsH_3 + 6 Ag(DDTC) = 6 Ag + 3 H(DDTC) + As(DDTC)_3$ 

#### **4 REAGENTS**

All the reagents, and the zinc in particular, shall be free from arsenic or have a very low arsenic content.

Distilled water, or water of equivalent purity, shall be used in the test.

**4.1 Hydrochloric** acid,  $\rho$  approximately 1,19 g/ml, about 38 % (*m/m*) solution, or approximately 12 N.

**4.2 Silver diethyldithiocarbamate**, [Ag(DDTC)], 5 g/l solution in pyridine.

Dissolve 1 g of silver diethyldithiocarbamate in pyridine ( $\rho$  approximately 0,980 g/ml) and dilute to 200 ml with the same pyridine.

Store the solution in a tightly sealed, dark glass bottle, protected from light.

This solution remains stable for about 2 weeks.

4.3 Arsenic standard solution, 0,100 g/l.

Weigh, to the nearest  $0,000 \ 1 \ g$ ,  $0,132 \ 0 \ g$  of arsenic trioxide  $(As_2O_3)$  and transfer it to a beaker of suitable capacity (for example, 100 ml). Dissolve the arsenic trioxide in about 2 ml of 50 g/l sodium hydroxide solution. Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask. Wash the beaker several times, collecting the wash water in the volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 100  $\mu$ g of As.

4.4 Arsenic standard solution, 2,50 mg/l.

Transfer 25,0 ml of the arsenic standard solution (4.3) to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix. Prepare this solution immediately before use.

1 ml of this standard solution contains 2,5  $\mu$ g of As.

4.5 Absorbent cotton wool saturated with lead acetate.

Dissolve 50 g of lead acetate trihydrate  $[Pb(C_2H_3O_2)_2.3H_2O]$  in 250 ml of water. Saturate the absorbent cotton wool with this solution, remove the excess solution by allowing it to drain and dry the cotton wool under vacuum at room temperature.

Store it in an air-tight container.

4.6 Potassium iodide, 150 g/l solution.

Dissolve 15 g of potassium iodide in water, dilute to 100 ml and mix.

4.7 Tin(II) chloride, hydrochloric acid solution.

Dissolve 40 g of tin(11) chloride dihydrate  $(SnCl_2.2H_2O)$  in a mixture of 25 ml of water and 75 ml of the hydrochloric acid solution (4.1).

**4.8 Zinc shot**, 0,5 to 1 mm, or any other form of zinc which has been shown, by experiment, to give equivalent results under the specified test conditions (see Annex).