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WS

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

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Determination of iodine in serum - As3+
(English Translation)

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard was drafted. China Centers for Disease Control and Prevention, Endemic Disease Control Center, Xiamen City, Fujian Province Center for Disease Control and Prevention, Shanxi Provincial Institute of Endemic Disease Control.

1 Scope

This standard specifies the arsenic-cerium catalytic spectrophotometric method for the determination of iodine in serum.

This standard applies to the determination of total iodine concentration in serum.

2 Principle

Serum samples were digested with perchloric acid-sodium chlorate solution, and the catalytic effect of iodine on the oxidation-reduction reaction of arsenic and cerium.

$\text{H}_3\text{AsO}_3 + 2\text{Ce}^{4+}$

$\text{H}_2\text{O} + \text{H}_3\text{AsO}_4 + 2\text{Ce}^{3+}$

During the reaction, yellow Ce^{4+} is reduced to colorless Ce^{3+} . The higher the iodine content, the faster the reaction speed, and the less Ce^{4+} remaining;

The reaction temperature and time were determined, and the absorbance value of the remaining Ce^{4+} in the system was measured colorimetrically to determine the iodine content.

3 Instruments

3.1

Digestion temperature control heating device. constant temperature digestion instrument (temperature control point accuracy $130\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, temperature difference between holes $\leq 1\text{ }^{\circ}\text{C}$).

3.2

Constant temperature water bath. The temperature control accuracy is $\pm 0.3\text{ }^{\circ}\text{C}$.

3.3

Spectrophotometer, 1cm cuvette.

3.4

Glass test tube. 15mmx100mm or 15mmx120mm.

3.5

Quantitative pipettes. 100 μL , 1000 μL , 5000 μL ;

Quantitative glass pipettes. 5 mL and 10 mL.

3.6

Stopwatch.

3.7

Constant temperature drying oven.

3.8

Centrifuge.

4 Reagent

4.1

Unless otherwise specified, the reagents used in this standard are analytical reagents, and the experimental water should meet the secondary water specifications in GB/T 6682.

4.2

Perchloric acid (HClO_4 , 70% ~ 72%), excellent grade pure.

4.3

Sodium chlorate (NaClO_3 , $M = 106.4$).

4.4

concentrated sulfuric acid (H_2SO_4 , $\rho_{20} = 1.84\text{g/mL}$), excellent grade pure.

4.5

Arsenic trioxide (As_2O_3 , $M = 197.8$).

4.6

Sodium chloride (NaCl , $M = 58.4$), excellent grade pure.

4.7

Sodium hydroxide (NaOH , $M = 40.0$).

4.8

Ammonium cerium sulfate [$\text{Ce}(\text{NH}_4)_4(\text{SO}_4)_4 \cdot 2\text{H}_2\text{O}$, $M = 632.6$] or ammonium cerium sulfate tetrahydrate [$\text{Ce}(\text{NH}_4)_4(\text{SO}_4)_4 \cdot 4\text{H}_2\text{O}$, $M = 668.6$].

4.9

Potassium iodate (KIO_3 , $M = 214.0$), reference reagent or standard substance.

5.1 Sodium chlorate solution [$c(\text{NaClO}_3) = 2.0\text{mol/L}$]

After weighing 106.5g of sodium chlorate (4.3) and dissolving it in 400 mL of pure water, add pure water to 500mL, and store it in a refrigerator (4 °C) for 12 Months.

5.2 Sulfuric acid solution [$c(\text{H}_2\text{SO}_4) = 2.5\text{mol/L}$]

Take 140mL of concentrated sulfuric acid (4.4) and slowly add it to 700mL of pure water. Stir while adding. After cooling, dilute to 1000mL with pure water.

5.3 Arsenite solution [$c(\text{H}_3\text{AsO}_3) = 0.025\text{mol/L}$]

Weigh 2.5g of arsenic trioxide (4.5) and 3.0g of sodium hydroxide (4.7) into a 1L beaker, add about 30mL of pure water and stir until full Partially dissolve, add about 500mL of pure water to the beaker, and add 40.0g of sodium chloride (4.6), stir until completely dissolved, and then slowly add.200mL Sulfuric acid solution (5.2), make up to 1000 mL with pure water at room temperature, store in a brown bottle, and store at room temperature for 6 months.

5.4 Cerium ammonium sulfate solution [$c(\text{Ce}^{4+}) = 0.025\text{mol/L}$]

Weigh 15.8g of cerium ammonium sulfate (4.8) or 16.7g of cerium ammonium sulfate tetrahydrate $[\text{Ce}(\text{NH}_4)_4(\text{SO}_4)_4 \cdot 4\text{H}_2\text{O}]$ dissolved in 700mL sulfuric acid solution (5.2), make up to 1000 mL with pure water, store in a brown bottle, and store at room temperature for 6 months.

5.5 Iodine standard stock solution [$\rho(\text{I}) = 100\mu\text{g/mL}$]

Accurately weigh 0.1686g of potassium iodate that has been dried to a constant weight at $105^\circ\text{C} \sim 110^\circ\text{C}$ in a volumetric flask, add pure water to dissolve, and make up to volume with pure water.

1000 mL. Store in tightly closed brown bottle, and store in refrigerator (4°C) for 6 months.

5.6 Iodine standard intermediate solution [$\rho(\text{I}) = 10\mu\text{g/mL}$]

Pipette 10.00 mL of iodine standard stock solution (5.5) into a 100 mL volumetric flask, and make up to volume with pure water. Store tightly the brown bottle can be stored in the refrigerator (4°C) for 1 month.

5.7 Iodine standard use series solution [$\rho(\text{I}) = 0\mu\text{g/L} \sim 300\mu\text{g/L}$]

Pipette iodine standard intermediate solution (5.6) 0 mL, 0.50 mL, 1.00 mL, 1.50 mL, 2.00 mL, 2.50 mL, and 3.00 mL before use Place them in 100 mL volumetric flasks and make up to volume with pure water. The iodine concentrations of this standard series of solutions are $0\mu\text{g/L}$, $50\mu\text{g/L}$, $100\mu\text{g/L}$, $150\mu\text{g/L}$, $200\mu\text{g/L}$, $250\mu\text{g/L}$, and $300\mu\text{g/L}$.

6 Sample collection and storage

Use a disposable vacuum non-coagulation blood collection tube to collect not less than 2mL of blood, leave it at room temperature for 0.5h, and centrifuge at 3000r/min for 10min.

The separated serum was placed in a polyethylene plastic tube with a stopper and hermetically sealed to prevent evaporation. Can be stored for 7 days at room temperature (20°C) and at 4°C it can be stored for 1 month, and frozen (-20°C) for at least 3 months