

ICS 59.080.01

CCS W 04



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

GB/T 43821-2024

Textiles - Determination of lead release

纺织品 铅释放量的测定

Issued on: March 15, 2024

Implemented on: October 1, 2024

**Issued by: State Administration for Market Regulation;
Standardization Administration of the PRC**

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0 Warning

A warning on the first page of the body states that the persons using this document are to have practical experience of regular laboratory work, that the document does not point out all the possible safety problems, and that it is the responsibility of the user to take suitable safety and health measures and to ensure compliance with the conditions laid down by the relevant national laws and regulations.

1 Scope

This document describes test methods for determining the lead released by textiles and by the accessories of textile products after treatment with artificial saliva, using an inductively coupled plasma optical emission spectrometer (ICP-OES), a graphite furnace atomic

absorption spectrometer (GFAAS) or an inductively coupled plasma mass spectrometer (ICP-MS).

It applies to the testing of textiles and of the accessories of textile products, such as buttons and zips.

2 Normative references

The contents of the following documents constitute indispensable provisions of this document through normative reference in the text. For dated references, only the edition corresponding to that date applies; for undated references, the latest edition, including all amendments, applies.

The list comprises GB/T 6682 Water for analytical laboratory use - Specification and test methods; GB/T 18886-2019 Textiles - Tests for colour fastness - Colour fastness to saliva; GB/T 30156 Method for corrosion and abrasion of coated accessories of textile products; GB/T 30158-2013 Determination of nickel release from accessories of textile products; and GB/T 40139 Measurement of material surface area - Hyperspectral imaging three-dimensional area measurement method.

3 Terms and definitions

This document has no terms and definitions that need to be defined.

4 Principle

After preparation the specimen is placed in artificial saliva and shaken; the lead concentration in the artificial saliva after shaking is determined with an inductively coupled plasma optical emission spectrometer, a graphite furnace atomic absorption spectrometer or an inductively coupled plasma mass spectrometer, and the lead release of the specimen per unit of time is calculated together with the tested surface area of the specimen. The lead release is expressed in micrograms per square centimetre per hour.

5 Reagents or materials

Unless otherwise specified, all reagents are of guaranteed grade and the water is grade one water as specified in GB/T 6682.

Nitric acid: 65 % by mass fraction, density 1.40 g/mL.

Lead standard stock solution at 1 000 µg/mL: 0.160 g of lead nitrate is weighed on the electronic balance, dissolved with 10 mL of 10 % nitric acid solution by volume, transferred to a 100 mL volumetric flask and diluted to the mark with 10 % nitric acid solution by volume, giving a stock solution of 1 000 µg/mL; when turbidity, precipitate or a change of colour appears during storage, the stock solution is prepared afresh. Lead standard

substances or standard sample stock solutions certified and issued with a reference material certificate by the State may also be used. A note records that the lead standard stock solution keeps for 6 months at ordinary temperature, 15 °C to 25 °C.

Lead standard intermediate working solution at 10 µg/mL: 1 mL of the lead standard stock solution is accurately transferred to a 100 mL volumetric flask and diluted to the mark with 1 % nitric acid solution by volume; when turbidity, precipitate or a change of colour appears during storage, it is prepared afresh. A note records that the intermediate working solution keeps for 2 weeks at ordinary temperature, 15 °C to 25 °C.

Lead series standard working solutions: a measured quantity of the intermediate working solution is accurately transferred to a 100 mL volumetric flask and diluted to the mark with 1 % nitric acid solution by volume to give a series of suitable concentration. For determination with an inductively coupled plasma optical emission spectrometer the series may be made up at 0.00 µg/mL, 0.02 µg/mL, 0.04 µg/mL, 0.20 µg/mL, 0.40 µg/mL, 0.80 µg/mL and 1.00 µg/mL; for determination with a graphite furnace atomic absorption spectrometer or an inductively coupled plasma mass spectrometer the series may be made up at 0.00 µg/mL, 0.01 µg/mL, 0.02 µg/mL, 0.04 µg/mL, 0.05 µg/mL, 0.08 µg/mL and 0.10 µg/mL. The lead series standard working solutions are used as soon as they are prepared.

Bismuth internal standard stock solution at 1 000 µg/mL or 100 µg/mL: a bismuth standard substance or standard sample stock solution certified and issued with a reference material certificate by the State. A note records that it keeps for 12 months at ordinary temperature, 15 °C to 25 °C. Bismuth internal standard working solution: a measured quantity of the stock solution is accurately transferred and diluted stepwise with 1 % nitric acid solution by volume to a suitable concentration, for instance 0.005 µg/mL, and is used as soon as it is prepared.

Artificial saliva: prepared according to 4.3 of GB/T 18886-2019, kept away from light, valid for 1 week. Degreasing solution: a suitably diluted neutral detergent, for instance an aqueous solution of sodium dodecylbenzene sulfonate at a mass fraction of 0.5 %. Wax or lacquer: free of lead, used to coat the non-tested surfaces of the specimen so as to prevent lead being released from those surfaces during the test.

6 Apparatus

An inductively coupled plasma optical emission spectrometer, a graphite furnace atomic absorption spectrometer or an inductively coupled plasma mass spectrometer.

A constant temperature water bath shaker, with the temperature controlled at (37 +/- 2) °C and a shaking frequency of (60 +/- 5) r/min.

An electronic balance with a scale division of 0.001 g; a steel rule with a measuring range of 0 mm to 200 mm and a scale division of 1 mm; covered containers fitted with screw seal caps and made of non-metallic, lead-free and nitric acid resistant materials, such as glass,

polypropylene, polytetrafluoroethylene and polystyrene, which before use are soaked for at least 4 h in 5 % nitric acid solution by volume; and volumetric flasks of 50 mL, 100 mL and other sizes.

7.1 Preparation of the test specimens

Textiles - sampling: the textile specimen is cut to (10.0 ± 0.5) cm² or to another suitable size. As far as possible three specimens of the same batch are taken for the test. A note records that fabrics are mainly flexible materials and that, on the reasonable assumption that any part of the fabric can be sucked or put into the mouth by infants and children, the size of the fabric is not restricted.

Textiles - measurement of the tested area: the tested area of the textile specimen is measured accurately and calculated with the steel rule.

Textile accessories - sampling: the parts of the accessories of textile products that can be put into the mouth by infants and children are sampled and tested. In order to reach the analytical sensitivity of the instrument and the minimum sample intake, the tested area of the specimen is at least 0.5 cm²; where necessary, identical specimens may be tested together to reach that minimum area. As far as possible three specimens of the same batch are taken for the test. A note records that an accessory is considered able to be put into the mouth by infants and children when any one dimension of the accessory, height, length or width, is less than 5 cm, or when any one dimension of a detachable or protruding part that could reasonably be assumed to come into contact with infants and children is less than 5 cm.

Textile accessories - measurement of the tested area: the tested area of the accessory is measured according to A.1 in Annex A of GB/T 30158-2013 or according to GB/T 40139.

Abrasion simulation: for coated accessories of textile products, that is electroplated, painted, polymer or similar coatings, reference is made to FZ/T 01163 to identify whether the coating of the textile accessory contains lead. Accessories with lead-free coatings are corroded and abraded according to GB/T 30156 and, where possible, the whole item is treated; accessories with lead-containing coatings are not subjected to the corrosion and abrasion treatment.

Masking: where the specimen has non-tested surfaces, that is faces that cannot be put into the mouth of infants and children, those surfaces are removed or protected so that they do not come into contact with the artificial saliva, in order to avoid lead being released from them; one or more layers of the lead-free wax or lacquer are applied to the non-tested surfaces.

Degreasing: at room temperature the prepared textile accessory is placed in the degreasing solution and stirred gently for 2 min, rinsed with water and dried in air. The degreasing step applies only to the parts of the specimen from which excess grease has to be removed.