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Traditional Chinese medicine — Determination of sulfur dioxide in natural products by titration

Médecine traditionnelle chinoise — Dosage du dioxyde de soufre dans les produits naturels par titrage

Reference number
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Foreword

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 249, *Traditional Chinese medicine*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Traditional Chinese medicine — Determination of sulfur dioxide in natural products by titration

1 Scope

This document specifies the determination method of sulfur dioxide in natural products used in traditional Chinese medicine, which includes the requirements of the device, chemicals, operational procedures and formula.

It is applicable to natural products of traditional Chinese medicine, including Chinese materia medica (whole medicinal materials) and decoction pieces derived from plants or animals.

It is not applicable to minerals used in traditional Chinese medicine.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
sulfur dioxide
toxic gas with a pungent, irritating smell, the chemical compound with the formula SO_2

3.2
sulfur dioxide residue
sulfur dioxide that remains in or on a natural product

Note 1 to entry: Sulfur dioxide residue is expressed as mg/kg.

3.3
maximum residue limit
MRL
highest level of sulfur dioxide that is permitted in or on a natural product

Note 1 to entry: Maximum residue limit is expressed as mg/kg.

3.4
acceptable daily intake
ADI
estimate of the amount of sulfur dioxide in natural products that can be safely consumed daily over a lifetime without adverse health effects

Note 1 to entry: ADI is expressed in milligrams of the sulfur dioxide, as it appears in the natural products, per kilograms of body mass per day (mg/kg/day).

3.5
acid-base titration
determination of the concentration of an acid or base by exactly neutralizing the acid or base with a base or acid of known concentration

4 Apparatus

4.1 Glass sample, cleaned thoroughly before use.

4.2 Electronic balance, with a minimum reading of 0,1 mg.

4.3 Separatory funnel, with a capacity of 50 ml or 100 ml.

4.4 Round-bottom flask, with a capacity of 500 ml or 1 000 ml.

5 Reagents

All reagents shall be of recognized chromatographic or analytical purity. Distilled water or water of equivalent purity or above, recently boiled, shall be used.

5.1 Distilled water.

5.2 Bromophenol blue, 1 g/l solution of bromophenol blue in ethanol (20 % volume fraction).

5.3 Hydrochloric acid, 6 mol/l solution of diluted hydrochloric acid. Dilute one volume of concentrated hydrochloric acid.

5.4 Hydrogen peroxide, 3 % volume fraction solution of diluted hydrogen peroxide, free from sulfate ions.

5.5 Sodium hydroxide, 0,1 mol/l solution of sodium hydroxide (standard volumetric solution).

6 Sampling and preservation of samples

6.1 Sampling

6.1.1 Laboratory samples

Raw material samples received by the laboratory shall be accompanied with complete information such as the source and time of collection of the samples. The samples for testing can include Chinese materia medica (whole medicinal materials) and decoction pieces derived from plants or animals.

6.1.2 Sample identification

When a sample is received, it shall be immediately assigned a unique label which will accompany it through all stages of the analysis to the reporting of the results. Samples shall be subject to the appropriate disposal review system and all records shall be accurately kept.

6.2 Sample pre-treatment and preservation

6.2.1 Sample pre-treatment

Before testing, the sample shall be dried and powdered. Samples shall be pre-treated as soon as possible and stored in a cool and dry place – if possible, in a refrigerator.

6.2.2 Sample storage

If samples cannot be analysed immediately, they shall be stored in a cool and dry place, away from the direct sunlight, and analysed within a few days.

7 Test method

7.1 Test procedure

a) Introduce 150 ml of water into the flask (see Figure 1, key 1), open the condenser pipe (key 3) and pass carbon dioxide through the whole system for 15 min at a rate of 100 ± 5 ml/min. To 10 ml of diluted hydrogen peroxide solution add 0,15 ml of a 1 g/l solution of bromophenol blue in ethanol (20 % volume fraction).

b) Add 0,1 M sodium hydroxide until a violet-blue colour is obtained, without exceeding the end point.

c) Place the solution in the test tube (key 4). Without interrupting the stream of carbon dioxide, remove the funnel (key 2) and introduce through the opening into the flask 25,0 g of the substance to be examined, with the aid of 100 ml of water.

d) Replace the funnel.

e) Close the tap of the funnel and add 80 ml of dilute hydrochloric acid to the funnel.

f) Open the tap of the funnel to allow the hydrochloric acid solution to flow into the flask, making sure that no sulfur dioxide escapes into the funnel by closing the tap before the last few millilitres of hydrochloric acid solution drain out.

g) Boil for 1 h. Open the tap of the funnel and stop the flow of carbon dioxide as well as the heating and the cooling of the water.

h) Transfer the contents of the test tube with the aid of a little water to a 200 ml wide-necked, conical flask.

i) Heat on a water-bath for 15 min and allow to cool.

j) Add 0,1 ml of a 1 g/l solution of bromophenol blue in ethanol (20 % volume fraction) and titrate with 0,1 M sodium hydroxide until the colour changes from yellow to violet-blue (V_1 ml).

k) Carry out a blank titration (V_2 ml).

NOTE 1 An alternative test method which is validated to be the same as this method can be used (see Table C.1).

NOTE 2 Annex B describes the method of gas chromatography to determine sulfur dioxide in natural products.

NOTE 3 Annex C provides an analysis of the similarities and differences of acid-base titration in different countries.

7.2 Calculation

Calculate the content of sulfur dioxide in parts per million using the following formula:

$$32\,030 \times (V_1 - V_2) \times (n/m)$$

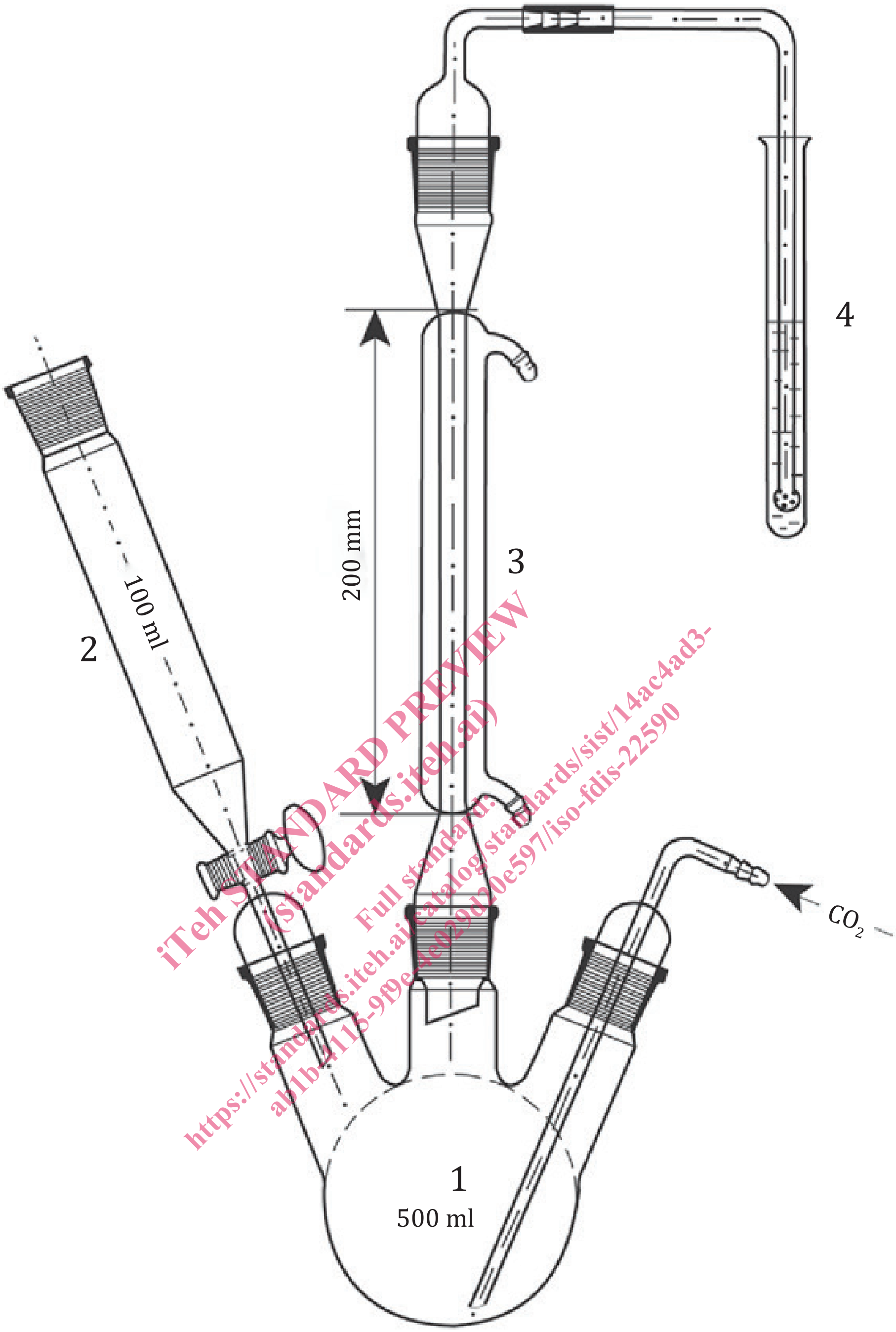
where

n is the molarity of the sodium hydroxide solution used as titrant;

m is the mass of the substances being examined;

V_1 is the volume of depleted sodium hydroxide of the test;

V_2 is the volume of depleted sodium hydroxide of the blank.



Key

1 three-neck round-bottom flask

2 funnel

3 condenser pipe

4 test tube

Figure 1 — Apparatus for the determination of sulfur dioxide

NOTE The limits of sulfur dioxide in different countries, regions and organizations and the calculated limits using target hazard quotients based on USEPA and WHO are shown in Annex A.

