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Traditional Chinese medicine — *Coptis chinensis* and *Coptis japonica* rhizome

Médecine traditionnelle chinoise — Rhizome de Coptis chinensis *et de* Coptis japonica

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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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.

Introduction

Coptis rhizome is used as traditional Chinese medicine in China. The rhizome of *Coptis japonica* Makino. is also used as herbal medicine in Japan and the Republic of Korea. As one of the most commonly used medicinal herbs, *Coptis* rhizome was listed in *Shennong materia Medica*. *Treatise on Febrile diseases* contains 113 prescriptions, including 12 containing *Coptis* rhizome.

However, there are still some concerns about the quality control of *Coptis* rhizome, outlined as follows, which affect the trade and use of this herb.

a) The harvesting and processing methods and techniques have not been standardized. Issues such as low efficiency, large interference from human factors and poor controllability seriously affect the quality of *Coptis* rhizome materials.

b) Even though many countries or regions, such as China, Japan, the Republic of Korea and Europe, have established pharmacopoeia standards for *Coptis* rhizome, the relevant requirements vary significantly, which limits the application of those standards in global trade.

c) The lack of quality standards for certain processed products, such as *Coptis* rhizome products processed with wine, ginger or *Euodia* fruit, makes it difficult to control their quality. This can affect the efficacy and safety of these products.

Coptis rhizome is ranked the fourth in the priority list of single herbal medicines for developing standards in ISO/TR 23975:2019. Thus, it is essential to develop an International Standard for *Coptis* rhizome to ensure consistency in the quality of *Coptis* rhizome and safe use of this herb and also to promote international trade.

In this document, the identification of commonly adulterated species of *Coptis* rhizome is also introduced.

As national implementation can differ, national standards bodies are invited to modify the values given in 5.5, 5.6 and 5.7 based on their national standards. Examples of national values are given in Annex C.

Traditional Chinese medicine — *Coptis chinensis* and *Coptis japonica* rhizome

1 Scope

This document specifies the minimum requirements and test methods for *Coptis* rhizome (the dried rhizome of *Coptis chinensis* Franch. and *Coptis japonica* Makino.).

This document applies to *Coptis rhizome* sold and used as Chinese materia medica (whole medicinal materials) and decoction pieces derived from these plants.

This document does not apply to the processed *Coptis rhizome*, including products traditionally processed with different methods, or its processing methods.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 18664, *Traditional Chinese Medicine — Determination of heavy metals in herbal medicines used in Traditional Chinese Medicine*

ISO/TS 21310, *Traditional Chinese medicine — Microscopic examination of medicinal herbs*

ISO 21371, *Traditional Chinese medicine — Labelling requirements of products intended for oral or topical use*

ISO 22217:2020, *Traditional Chinese medicine —Storage requirements for raw materials and decoction pieces*

ISO 22258, *Traditional Chinese medicine — Determination of pesticide residues in natural products by gas chromatography*

ISO 23723:2021, *Traditional Chinese medicine — General requirements for herbal raw material and materia medica*

3 Terms and definitions

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

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

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

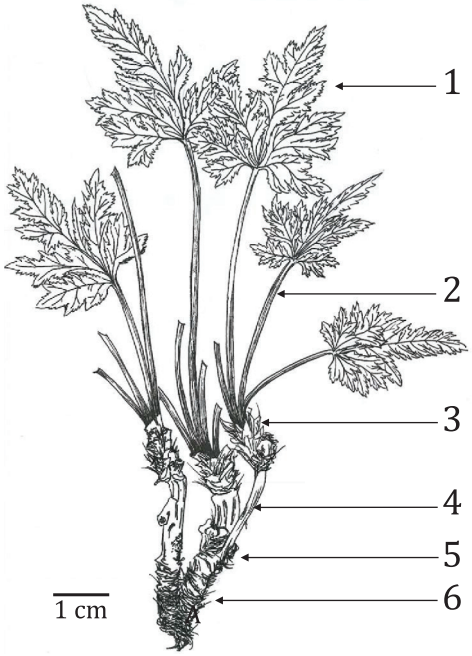
— IEC Electropedia: available at https://www.electropedia.org/

3.1
***Coptis* rhizome**
dried rhizome of *Coptis chinensis* Franch. and *Coptis japonica* Makino.

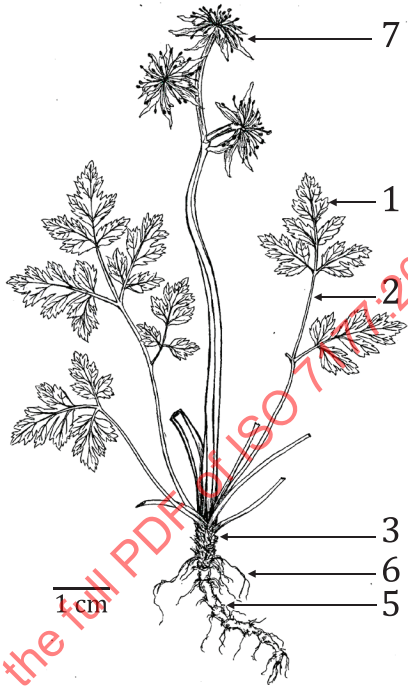
3.2
bridge piece
morphological feature of *Coptis* rhizome with its rhizome internodes as smooth as stem

4 Description

Coptis rhizome is the dried rhizome of *Coptis chinensis* Franch. and *Coptis japonica* Makino., collected in autumn, removed from rootlets and soil and dried, as shown in Figure 1.

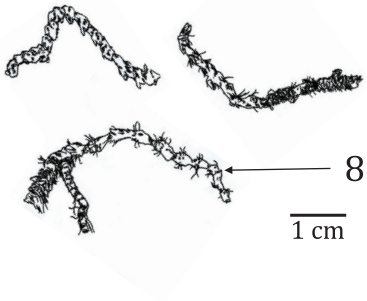


a) Plant of *Coptis chinensis* Franch.



b) Plant of *Coptis japonica* Makino.

c) *Coptis chinensis* rhizome



d) *Coptis japonica* rhizome

Key

1 leaf
2 petiolate
3 phyllode
4 bridge piece
5 rhizome
6 fibrous root
7 flower
8 nodular bump

Figure 1 — Structure of *Coptis* rhizome

5 Requirements

5.1 General characteristics

The following requirements shall be met before sampling:

a) *Coptis* rhizome shall be clean and free from leaves and foreign matter.

b) The presence of living insects, mouldy fruit and external contaminants which are visible to the naked eye shall not be permitted.

5.2 Morphological features of rhizome

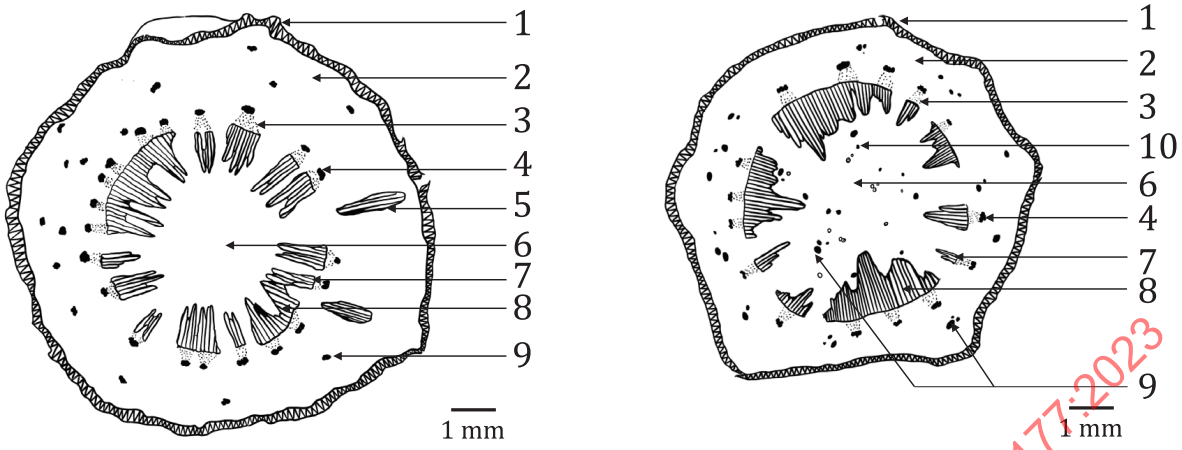
a) *Coptis chinensis* rhizome is gathered in a cluster, curved like chicken feet. A single rhizome is 3 cm to 6 cm long and 0,3 cm to 0,8 cm in diameter. The outer surface is greyish-yellow or yellowish-brown, rough, bearing irregular nodular bumps, fibrous roots and their residues. Some internodes are as smooth as the stem and commonly known as bridge pieces. The upper part mostly retains brown scale leaves. The apex often bears the remains of stems or petioles. The rhizome has a hard texture and its fracture is uneven. The bark is orange-red or dark brown. The wood is bright yellow or orange-yellow, radially arranged. The pith is sometimes hollow. It has a slight odour and a very bitter taste.

b) *Coptis japonica* rhizome is irregular and cylindrical. The rhizome is 2 cm to 4 cm long and 0,2 cm to 0,7 cm in diameter, slightly curved and short-branched. The outer surface is greyish yellow-brown, with ring nodes, without a bridge piece but with numerous remains of rootlets. The rhizome generally bears the remains of petiole at one end. It has a slight odour and an extremely bitter and lasting taste.

5.3 Microscopic identification

a) *Coptis chinensis* rhizome contains cork cells of several layers, covered on the outside by epidermis which is often withered. The cortex is broader; stone cells are singly scattered or grouped. The pericycle fibres are in bundles or accompanied by a few stone cells; both are yellow. Collateral vascular bundles are arranged in a ring. The xylem is yellow and lignified and the xylem fibres are well developed. The pith consists of parenchymatous cells, but stone cells are absent (see Figure 2 a).

b) *Coptis japonica* rhizome consists of a few stone cells in cortex and pith. The fractured surface is rather fibrous. Cork layer is light greyish brown. The cortex and pith are yellow-brown to reddish yellow-brown. The xylem is yellow to reddish-yellow (see Figure 2 b).



a) *Coptis chinensis* rhizome **b) *Coptis japonica* rhizome**

Key

1 cork layer
2 cortex
3 phloem
4 pericycle fibre
5 root-trace vascular bundle
6 pith
7 cambium
8 xylem
9 stone cells
10 starch grains

Figure 2 — Transverse section of *Coptis* rhizome

5.4 Thin-layer chromatography (TLC) identification

The thin-layer chromatography (TLC) of *Coptis* rhizome shall present fluorescent spots with the same colour and positions corresponding to the chromatogram of reference drug solution and one spot corresponding to the reference solution in the chromatogram.

5.5 Moisture

The content of water should be a mass fraction of ≤ 14,0 %.

5.6 Total ash

The content of total ash should be a mass fraction of ≤ 5,0 %.

5.7 Acid-insoluble ash

The content of acid-insoluble ash should be a mass fraction of ≤ 2,5 %.

5.8 Heavy metals

The content of heavy metals, such as arsenic, mercury, lead and cadmium, shall be determined.

5.9 Pesticide residues

The content of pesticide residues shall be determined.

5.10 Marker compounds

The content of marker compounds, such as berberine, epiberberine, coptisine and palmatine, should be determined as a mass fraction.

6 Sampling

Sampling shall be carried out in accordance with the method described in ISO 23723:2021, Clause 8.

7 Test methods

7.1 Macroscopic identification

The samples shall be examined by the naked eye in sunlight and also for smell and taste as described in 5.2.

7.2 Microscopic identification

The testing method specified in ISO/TS 21310 shall apply.

7.3 Thin-layer chromatography (TLC) identification

See Annex A for additional information.

7.4 Determination of moisture

The testing method specified in ISO 23723:2021, 7.2.1 shall apply.

7.5 Determination of total ash

The testing method specified in ISO 23723:2021, 7.2.3 shall apply.

7.6 Determination of acid-insoluble ash

The testing method specified in ISO 23723:2021, 7.2.3 shall apply.

7.7 Determination of heavy metals

The testing method specified in ISO 18664 shall apply.

7.8 Determination of pesticide residues

The testing method specified in ISO 22258 shall apply.

7.9 Determination of marker compounds

See Annex B for additional information.

8 Test report

For each test method, the test report shall specify the following:

a) all information necessary for the complete identification of the sample;

b) the sampling method used;

c) the test method used, with reference to this document;

d) the test result(s) obtained;

e) all operating details not specified in this document or regarded as optional, together with details of any incidents which could have influenced the test result(s);

f) any unusual features (anomalies) observed during the test;

g) the date of the test.

9 Packaging, storage and transportation

The packaging and transportation shall not transmit any odour or flavour to the product and shall not contain substances that could damage the product or constitute a health risk. The packaging shall be strong enough to withstand normal handling and transportation.

The storage conditions in ISO 22217:2020, 5.2.1 shall apply.

The products shall be protected from light, moisture, pollution and foreign substances during long-distance delivery. Carriers should be well ventilated so they remain dry and moisture-proof.

10 Marking and labelling

The method specified in ISO 21371 shall apply. The following items shall be marked or labelled on the packages:

a) all quality features indicated in Clause 5, determined in accordance with the methods specified in Clause 7;

b) gross weight and net weight of the package;

c) country, province or state of origin of the products;

d) date of production and expiry date of the products;

e) storage method;

f) any items required by the destination.

Annex A
(informative)

Thin-layer chromatography (TLC) identification of *Coptis* rhizome

a) Take 0,25 g of the powdered *Coptis* rhizome sample (through a 24-mesh sieve) with 25 ml of methanol, ultrasonicate for 30 min, filter and use the filtrate as the test solution.

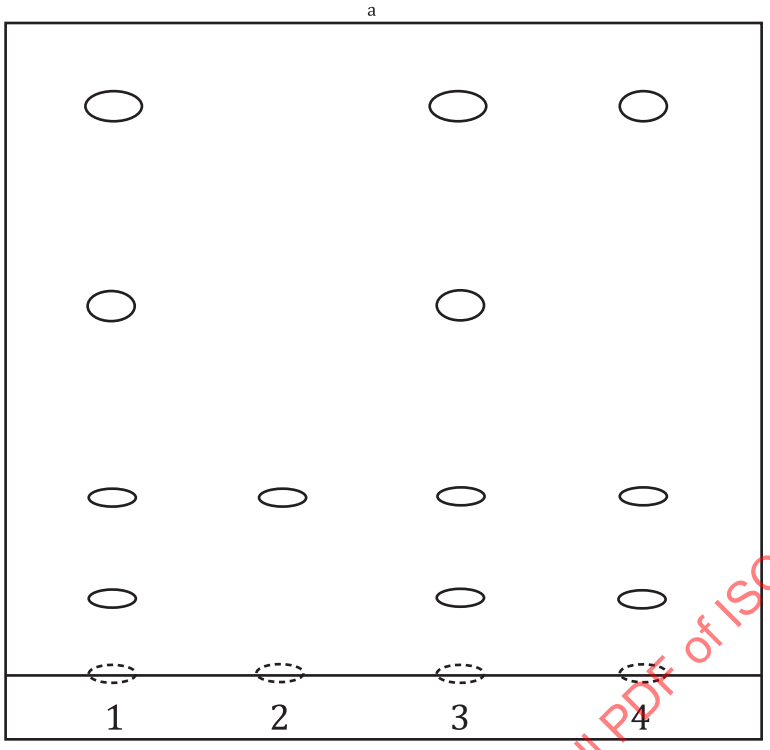
b) Prepare a solution of 0,25 g of powdered *Coptis chinensis* rhizome reference drug in the same manner as the reference drug solution.

c) Dissolve berberine chemical reference substance (CRS) in methanol to produce a solution containing 0,5 mg per ml as the reference solution.

d) Carry out the method for thin-layer chromatography using silica gel G as the coating substance and a mixture of cyclohexane, ethyl acetate, isopropanol, methanol, water and triethylamine (3:3,5:1:1,5:0,5:1) as the mobile phase.

e) Apply separately to the plate 1 µl of each of the solutions in a) to c). After developing in a chamber pre-equilibrated with vapour of strong ammonia for 20 min and removing the plate, dry in air and examine under ultraviolet light at 365 nm.

f) The fluorescent spots in the chromatogram obtained with the test solution correspond in position and colour to the fluorescent spots in the chromatogram obtained with the reference drug solution, and one of which corresponds to the spot in the chromatogram obtained with the reference solution. See Figure A.1.



Key

1 reference drug solution of *Coptis chinensis* rhizome
2 berberine CRS reference solution
3 *Coptis chinensis* rhizome test solution
4 *Coptis japonica* rhizome test solution
[a] Top of the plate.

Figure A.1 — TLC chromatograms of *Coptis chinensis* and *Coptis japonica* rhizome

Annex B
(informative)

Determination of marker compounds by high-performance liquid chromatography with an ultraviolet detector (HPLC-UV)

B.1 Preparation of test solution

a) Weigh accurately 0,2 g of the powdered *Coptis rhizome* sample (through a 24-mesh sieve) in a 100 ml stoppered conical flask.

b) Accurately add 50 ml of a mixture of methanol and hydrochloric acid (100:1), weigh and ultrasonicate for 30 min (power 250 W, frequency 40 kHz).

c) Cool and weigh again, replenish the loss of the solvent with methanol to volume and mix well.

d) Filter and accurately take 2 ml of filtrate in a 10 ml volumetric flask, add methanol to the mark and shake well.

e) Filter with 0,45 µm microporous membrane and use the successive filtrate as the test solution.

B.2 Preparation of reference solution

Weigh a quantity of berberine hydrochloride CRS accurately, dissolve in methanol to produce a solution containing 90,5 µg/ml as the reference solution.

B.3 Chromatographic conditions

a) Stationary phase: octadecylsilane bonded silica gel.

b) Column size: 4,6 µm, 5 mm × 150 mm.

c) Mobile phase A: a mixture of acetonitrile and 0,05 mol/l solution of potassium dihydrogen phosphate (50:50) (per 100 ml add 0,4 g of sodium dodecyl sulfate and adjust to pH 4,0 with phosphoric acid).

d) Flow rate: 1 ml/min.

e) Injection volume: 10 µl.

f) Temperature of column oven: 30 °C.

g) Detector: a spectrophotometer set at 345 nm.

B.4 Determination of marker compounds

Inject 10 µl each of the reference solution and the test solution into the column and determine the content of marker compound. For the high-performance liquid chromatography (HPLC) chromatograms of *Coptis chinensis* rhizome, see Figure B.1. With the peak area of berberine hydrochloride CRS as an external standard, calculate the contents of berberine, epiberberine, coptisine and palmatine. The mass fraction of four compounds of the sample being examined on the dried basis, w_{ti} (%), is calculated with Formula (B.1):

$$w_{ti} = (C_b \times A_{ti} \times 50 \times 10)/(A_b \times m_t \times 2 \times 1\,000\,000) \times 100 \quad \text{(B.1)}$$

where

C_b is the concentration of berberine CRS of the reference solution, in µg/ml;

A_{ti} is the peak area of test compound of the test solution, in mAU*s;

A_b is the peak area of berberine CRS of the reference solution, in mAU*s;

m_t is the mass of the test sample, in g.

Confirm these four peak locations by the relative retention time for four compounds in Table B.1. The retention time of the test compound peak of the sample, T_{ti}, is calculated with Formula (B.2):

$$T_{ti} = T_b \times f \quad \text{(B.2)}$$

where

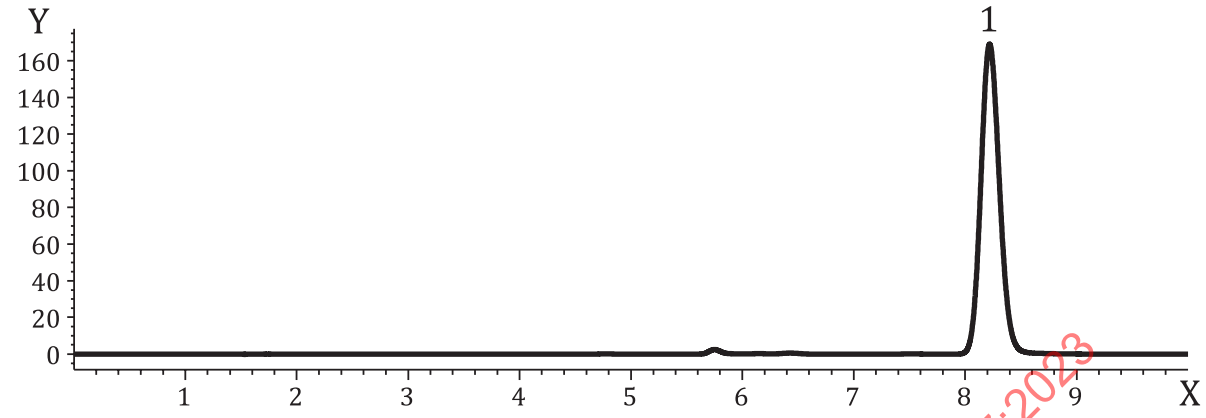
T_b is the retention time of berberine of the test solution, in min;

f is the relative retention time.

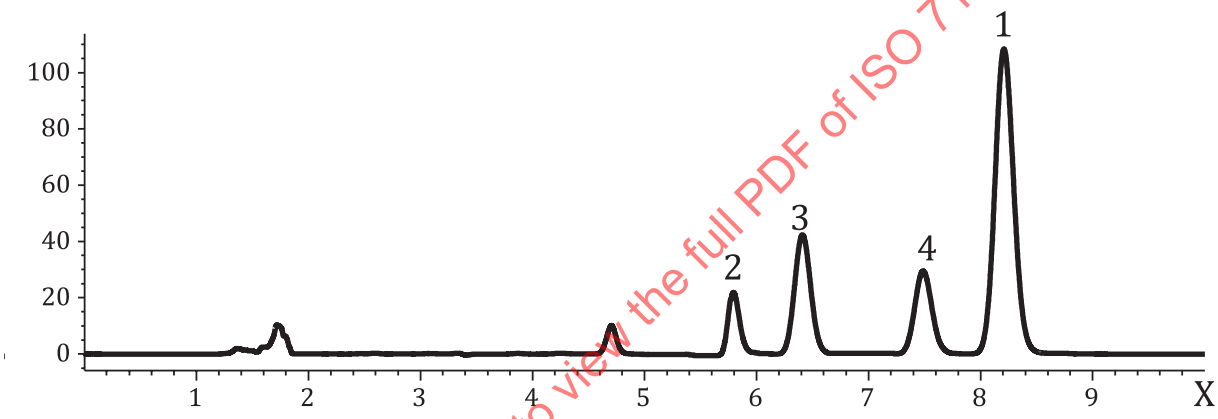
Table B.1 — The relative retention times for four compounds

Test compound peak	Relative retention time
Epiberberine	0,71
Coptisine	0,78
Palmatine	0,91
Berberine	1,00

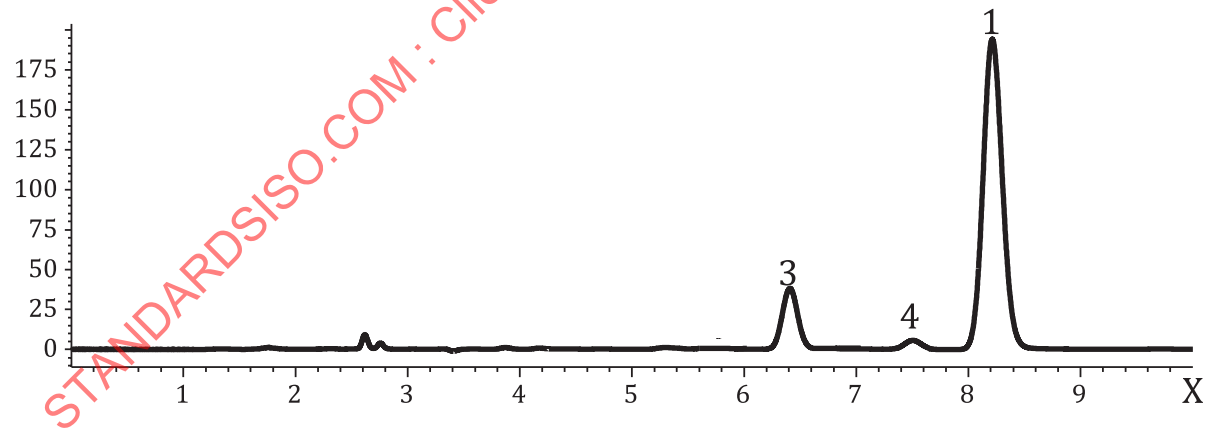
The relative retention time of the test peak should remain within ± 5 % of the value in Table B.1. Validate the methodology for accuracy, precision and repeatability if necessary.



a) Berberine CRS reference solution



b) *Coptis chinensis* rhizome test solution



c) *Coptis japonica* rhizome test solution

Key

X mAU
Y min
1 berberine
2 epiberberine
3 coptisine
4 palmatine

Figure B.1 — HPLC chromatograms of *Coptis* rhizome