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(JTCCM/JSA)

**Determination of asbestos in building
material products — Part 3: Quantitative
analysis of containing asbestos by X-ray
diffraction method**

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Foreword

This translation has been made based on the original Japanese Industrial Standard established by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee according to the proposal for establishment of Japanese Industrial Standard submitted by Japan Testing Center for Construction Materials (JTCCM)/Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law. Consequently **JIS A 1481:2008** has been withdrawn and partially replaced with this Standard.

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Attention is drawn to the possibility that some parts of this Standard may conflict with patent rights, applications for a patent after opening to the public or utility model rights. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying any of such patent rights, applications for a patent after opening to the public or utility model rights.

JIS A 1481 series consists of the following 3 parts:

JIS A 1481-1 *Air quality — Bulk materials — Part 1: Sampling and qualitative determination of asbestos in commercial bulk materials*

JIS A 1481-2 *Determination of asbestos in building material products — Part 2: Sampling and qualitative analysis for judgement of existence of containing asbestos*

JIS A 1481-3 *Determination of asbestos in building material products — Part 3: Quantitative analysis of containing asbestos by X-ray diffraction method*

Determination of asbestos in building material products — Part 3 : Quantitative analysis of containing asbestos by X-ray diffraction method

Introduction

This Japanese Industrial Standard was established as **JIS A 1481** in 2006 and has gone through one revision since then, which was made in 2008. The purpose of this revision and establishment of this Standard as **JIS A 1481-3** is : to resolve the inconvenience in standard structure; to divide **JIS A 1481** into series of standards for the purpose of harmonization with the newly established related International Standards; and to partly revise the requirements of the analytical method specified. The comparison table between previous and current editions of this Standard on technically significant revisions is given in Annex D.

No corresponding International Standard has been established at this point.

1 Scope

This Standard specifies the method of determining asbestos content (mass fraction) (hereafter referred to as “asbestos content”) in building material products by the X-ray diffraction analysis method. It consists of procedures for preparation of secondary and tertiary analytical samples for quantitative determination, and the X-ray diffraction method employing the absorption correction by basal standard method.

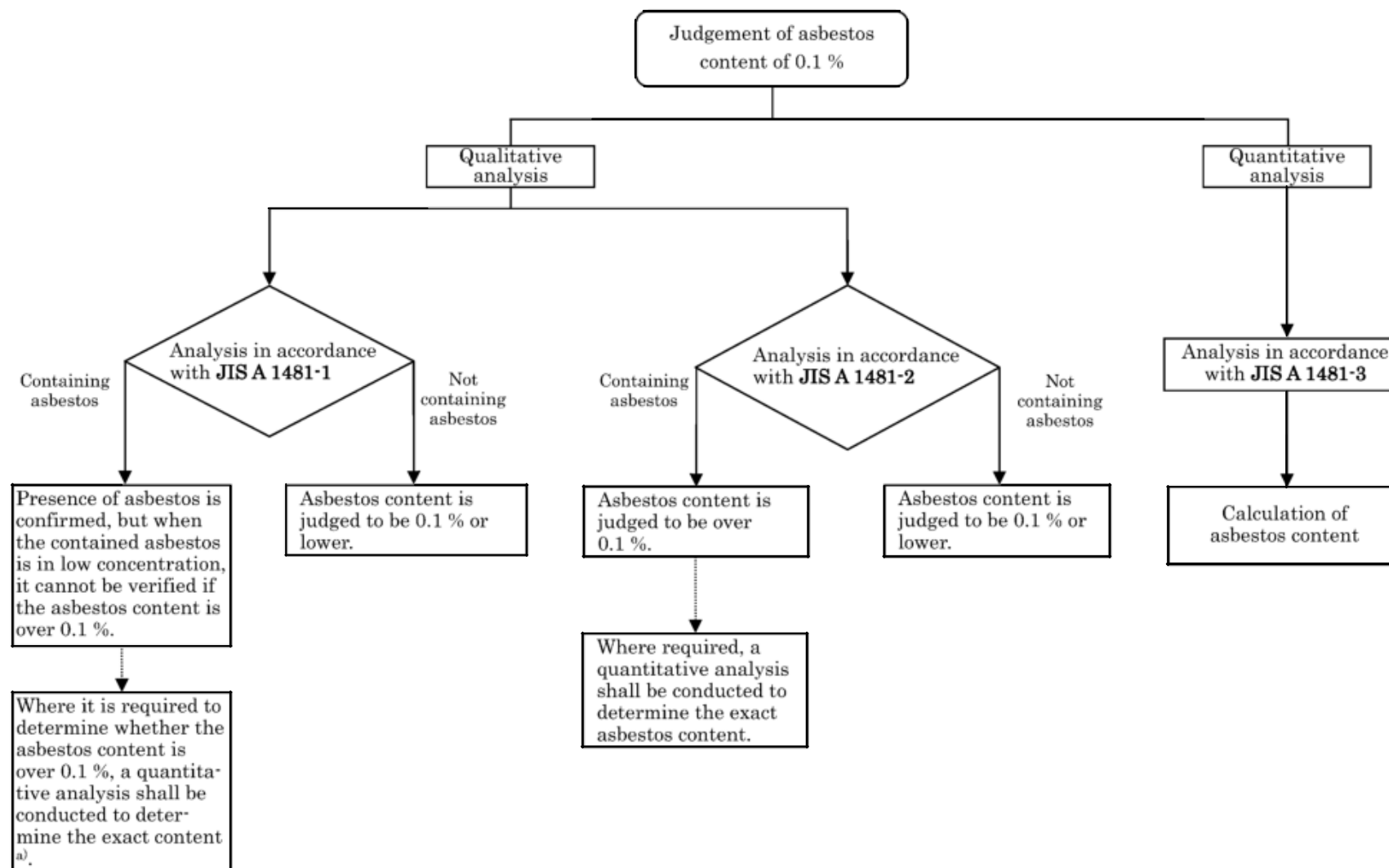
It is not applicable to natural minerals which can contain asbestos as impurities or any products that are made of such natural minerals.

For criteria to be applied for the analysis, those stipulated by the following domestic laws and regulations shall prevail over those specified in this Standard.

- Industrial Safety and Health Act
- Waste Management and Public Cleansing Act

NOTE 1 **JIS A 1481-1** is applicable to asbestos analysis for both building material products and natural minerals. **JIS A 1481-2** and **JIS A 1481-3**, on the other hand, are only applicable to asbestos analysis for building material products and not applicable to analysis for natural minerals which can contain asbestos or any products that are made of such natural minerals.

NOTE 2 Figure 1 shows the flow chart for judgement of asbestos content of 0.1 %.



Note ^{a)} **JIS A 1481-1** is applicable to asbestos analysis for both building material products and natural minerals. **JIS A 1481-2** and **JIS A 1481-3**, on the other hand, are only applicable to asbestos analysis for building material products and not applicable to analysis for natural minerals which can contain asbestos or any products that are made of such natural minerals.

Figure 1 Flow chart for judgement of 0.1 % asbestos content

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

- JIS A 1481-1 *Air quality — Bulk materials — Part 1 : Sampling and qualitative determination of asbestos in commercial bulk materials*
- JIS A 1481-2 *Determination of asbestos in building material products — Part 2 : Sampling and qualitative analysis for judgement of existence of containing asbestos*
- JIS K 1522 *Isopropyl alcohol (Isopropanol)*
- JIS K 8264 *Formic acid (Reagent)*
- JIS R 3503 *Glass apparatus for chemical analysis*

3 Terms and definitions, symbols and units

3.1 Terms and definitions

For the purpose of this Standard, the terms and definitions given in **JIS A 1481-2**, and the following apply.

3.1.1 secondary analytical sample for quantitative determination

sample prepared by treating the primary analytical sample with formic acid so that it can be used for quantitative analysis

3.1.2 tertiary analytical sample for quantitative determination

sample prepared by conditioning the secondary analytical sample for quantitative determination whose residual ratio exceeds 0.15 so that it can be used for quantitative analysis

3.1.3 residual ratio

the ratio of the amount (residue amount) of secondary analytical sample for quantitative determination to the amount of primary analytical sample

3.1.4 weight loss ratio

the ratio of the amount of the heat-treated primary analytical sample (weight loss) to the amount of the sample

3.1.5 integrated intensity

the area intensity (integrated value) of a specified X-ray diffraction line (peak)

3.2 Symbols and units

For the purpose of this Standard, the symbols and units shown in Table 1 apply.

Table 1 Symbols and units

Symbols	Designation	Units
M_1	Weighed value of primary analytical sample	mg
M_2	Weighed value of secondary analytical sample for quantitative determination	mg
M_3	Weighed value of tertiary analytical sample for quantitative determination	mg
A_s	Asbestos mass of secondary analytical sample for quantitative determination read in working curve	mg
A_{s1}	Asbestos mass of tertiary analytical sample for quantitative determination read in working curve	mg
C_i	Asbestos content of one analytical sample	%
C_t	Asbestos content of one tertiary analytical sample for quantitative determination	%
C	Asbestos content in building material products	%
r	Weight loss ratio (amount of primary analytical sample after heat-treatment/amount of sample)	—
V	Integrated intensity (applicable only to vermiculite)	Count
σ	Standard deviation of integral X-ray intensity of 10 measurements	—
a	Inclination of calibration curve	—
C_k	Minimum limit of detection of asbestos content	%
C_l	Minimum limit of determination of asbestos content	%
R	Correlation function	—
R^2	Decision function	—

4 Quantitative analytical method and principle

4.1 Summary of quantitative analytical method

The quantitative analysis for samples judged to be “containing asbestos” consists of the following main steps (see Figure 2).

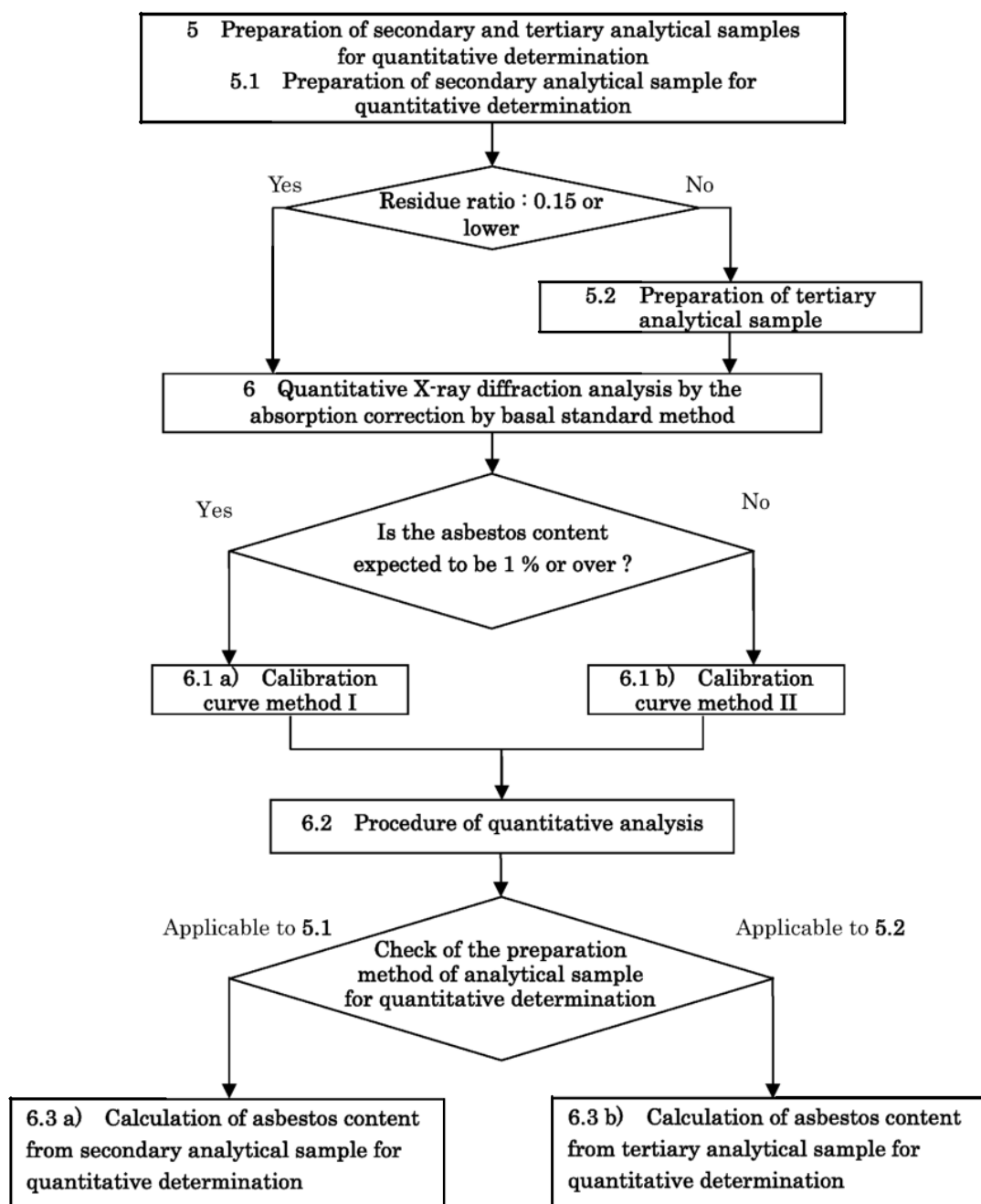
- a) **In the case of skipping the sample pre-treatment** If the analysis for asbestos presence conducted in accordance with **JIS A 1481-1** or **JIS A 1481-2** clearly indicates a high asbestos content (e.g. 5 % or over), the pre-treatment which is to be performed on the sample subsequently may be skipped, and the primary analytical sample may be directly used for the quantitative analysis of asbestos. In this case, disperse 10 mg of the primary analytical sample in dust-free water, and filter under reduced pressure using the reduced-pressure filtration apparatus of a 25 mm glass filter base fitted with a filter. Measure the asbestos in the sample filter of the apparatus and the diffraction intensity of the basal standard plate by the X-ray diffractometer, and determine the asbestos in the primary analytical sample by referring to the calibration curve of the standard asbestos sample prepared under the same conditions as the curve of the analytical sample by employing the absorption correction by basal standard method as specified in Annex B. See Annex A for conditions of X-ray diffractometer relating to asbestos analysis.

- b) **In the case of performing the sample pre-treatment** If the analysis for asbestos presence conducted in accordance with **JIS A 1481-1** or **JIS A 1481-2** clearly indicates a low asbestos content (e.g. less than 5 %), the following procedures shall be performed.
- 1) When the residual ratio is 0.15 or less, prepare the secondary analytical sample for quantitative determination, and determine the asbestos content in that sample by the X-ray diffraction method employing the absorption correction by basal standard method specified in Annex B, and calculate the asbestos content.
 - 2) When the residual ratio is over 0.15, prepare the tertiary analytical sample for quantitative determination, and determine the asbestos content in that sample by the X-ray diffraction method employing the absorption correction by basal standard method specified in Annex B, and calculate the asbestos content.

4.2 Principle of quantitative analytical method

Since the intensity of X-ray diffraction of asbestos is dependent on the amount of asbestos contained, the amount of asbestos in building material can be determined by comparing the intensity of X-ray diffraction of its analytical sample with that of the calibration curves prepared using a sample with a known mass of asbestos. As the intensity of the X-ray diffraction of asbestos is also influenced by the matrix of the sample, preparation of the analytical sample is performed with a measure taken so that the influence would be as small as possible. Further, correction is made for the decrease in intensity of the X-ray diffraction of asbestos which is caused by the X-ray absorption of the sample itself. The absorption correction by basal standard method adopted in this Standard minimises the influences explained above.

Quantitative analysis using an X-ray diffractometer can be carried out either by the internal standard method or by the standard addition method. These methods, though not high in precision when applied for asbestos analysis, are still effective in providing approximate values of asbestos content.



Flowchart for measurement of asbestos content in building material products

Figure 2 Summary of quantitative analytical method

5 Preparation of secondary and tertiary analytical samples for quantitative determination

5.1 Preparation of secondary analytical sample for quantitative determination

The preparation of secondary analytical sample for quantitative determination shall be as follows.

- a) Measure beforehand the mass of the fluorine resin binder glass fibre filter (hereaf-

ter referred to as “filter”) of 25 mm diameter to be used for the preparation of secondary analytical sample for quantitative determination and the X-ray diffraction intensity of basal standard material.

- b) Weigh accurately 100 mg (M_1 : weighed value of primary analytical sample) of primary analytical sample, and put it in a conical beaker specified in **JIS R 3503**. Add 20 ml of 20 % formic acid specified in **JIS K 8264** and 40 ml of dust-free water, and disperse them by using an ultrasonic cleaner for 1 min.
- c) Put them in a thermostatic bath set at $30\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ and vibrate continuously for 12 min.
- d) Filter under reduced pressure using the reduced-pressure filtration apparatus of glass filter base of 25 mm diameter fitted with a filter.
- e) After drying, weigh the mass (M_2 : weighed value of secondary analytical sample for quantitative determination) of the sample collected on filter and if the residual ratio (M_2/M_1) is found to be 0.15 or under, take it as the secondary analytical sample for quantitative determination. Prepare three such secondary analytical samples for quantitative determination per one sample. The reduced-pressure filtration apparatus of glass filter base and the filter shall have the same diameter as the sample stand of the X-ray diffractometer.

When the residual ratio (M_2/M_1) exceeds 0.15 in preparation of secondary analytical sample for quantitative determination, prepare a tertiary analytical sample for quantitative determination in accordance with 5.2.

5.2 Preparation of tertiary analytical sample for quantitative determination

The preparation of tertiary analytical sample for quantitative determination shall be as follows.

- a) If the residual ratio in the sample prepared in 5.1 exceeds 0.15, take 10 mg to 15 mg of the residue and disperse it in dust-free water.
- b) Subject the filter, which has been weighed, to filtration under reduced pressure, and dry.
- c) When drying is complete, weigh it, obtain the mass of dust (M_3 : weighed value of tertiary analytical sample for quantitative determination) and take it as the tertiary analytical sample for quantitative determination.
- d) Notes for preparation of tertiary analytical sample for quantitative determination are as follows.
 - 1) In the case of an inorganic component sample which can be reduced by heating again under the condition specified in 6.2 b) of **JIS A 1481-2**, the secondary analytical sample for quantitative determination may be prepared from the primary analytical sample after heating.
 - 2) Where the residual ratio in the sample prepared in 5.1 exceeds 0.15 and the residue amount is not sufficient for preparing tertiary analytical sample for quantitative determination, the residue obtained by newly performing the preparation