

JIS

JAPANESE INDUSTRIAL STANDARD

**Measuring method for
airborne fibrous particles**

JIS K 3850^{—1995}

Translated and Published

by

Japanese Standards Association

Printed in Japan

In the event of any doubt arising,
the original Standard in Japanese is to be final authority.

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Measuring method for airborne
fibrous particles

K 3850-1995

1. Scope This Japanese Industrial Standard specifies the measuring method by counting airborne fibrous particles in measurement of performance of an air cleaner, control of exhaust gas, other contamination control, etc.

Remarks 1. There are many kinds of airborne fibrous particles. Since the analysis by counting thereof is carried out by using an optical microscope and an electron microscope according to the purpose, the measuring method is specified respectively therefor.

2. The following standards are cited in this Standard.

JIS B 7551 Variable area flowmeters

JIS K 1522 Isopropyl alcohol (isopropanol)

JIS K 3802 Technical terms for membranes and membrane processes

JIS K 8034 Acetone

JIS K 8101 Ethanol (99.5)

JIS K 8271 Xylene

JIS K 8322 Chloroform

JIS R 3702 Cover glass for microscope

JIS R 3703 Slide glasses for microscope

JIS Z 8122 Contamination control - Vocabulary

JIS Z 8401 Rules for rounding off of numerical values

JIS Z 8761 Method of measurement by variable area flowmeters

JIS Z 8808 Methods of measuring dust concentration in flue gas

3. The units and numerical values given in { } in this Standard are based on the traditional units and appended for informative reference.

2. Definitions For the purpose of this Standard, in addition to those given in JIS K 3802 and JIS Z 8122, the following definitions shall apply.

- (1) fibrous particles Particles of at least 3 aspect ratio (length/width) which float in the air.
- (2) asbestos Fibrous silicate minerals (chrysotile) belonging to a serpentine group, and fibrous silicate minerals (amosite, crocidolite, tremolite, actinolite, and anthophyllite) belonging to an amphibole group among minerals forming rock. It is also called ISIWATA (Japanese).
- (3) fiber concentration The number of fibrous particles contained in the air of a specific volume (cm^3 or l). Its unit is expressed by piece/ cm^3 (f/ cm^3) or piece/ l (f/ l).
- (4) isokinetic sampling Suction of air at the same flow rate as that of the air by making a filter holder face to the stream of the air when a sample is taken from the air flowing through a duct or the like at a specific velocity.
- (5) polycarbonate filter A filter for sampling particles wherein uniform micropores of $0.02\ \mu\text{m}$ to $2.0\ \mu\text{m}$ diameter are provided on a polycarbonate made membrane.
- (6) backup filter A filter placed on the back side for the purposes of reinforcing a sampling filter, prevention of contamination, etc. when sampling airborne particles with sampling filter. A glass fiber filter, an acetylacetate membrane filter, etc., are used.
- (7) glass fiber filter A woven cloth wherein thin glass fibers of about $0.2\ \mu\text{m}$ to $10\ \mu\text{m}$ diameter are laminated. There are glass fiber filters of various kinds of sampling efficiency as a particle sampling filter.
- (8) polyvinyl alcohol (PVA) A water soluble high-molecular compound of about 2000 polymerization degrees, which is expressed by $(-\text{CH}_2\text{CHOH}-)_n$.
- (9) eyepiece graticule A circular transparent glass plate which is installed near the eyepiece of an optical microscope, and projects necessary information (the range for limiting a visual field, standard scale, etc.) in the visual field so that the observation and counting with the microscope can be easily carried out. Various eyepiece graticules are available according to the purpose. An example of the eyepiece graticule for counting asbestos is given in Informative reference figure 1.

3. Classification of measuring methods The method for measuring fibrous particles shall be classified according to each principle as follows:

3.1 Measuring method using optical microscope

- (1) Objective range In this measuring method, which is for measuring the fiber concentration of fibrous particles including asbestos, it can be regarded as the fiber concentration of asbestos especially in measurement in a plant wherein asbestos is handled, a discharge opening of an air cleaner wherein sampling of asbestos is known, etc. However, in this measuring method, since the asbestos is difficult to be identified by discriminating it from other fibrous particles, when only the fiber concentration of asbestos is desired to be obtained from the mixture of asbestos and other fibrous particles, the method using an electron microscope in 5.2 applies.
- (2) Summary Fibrous particles floating in the air are sampled with the filter specified in 4.1 (1), and the filter is made transparent. Thereafter, the number of fibrous particles on the filter is counted with the optical microscope specified in 5.1.1 to measure the fiber concentration.

3.2 Measuring method using electron microscope

- (1) Objective range This is a measuring method wherein the fiber concentration of fibrous particles such as asbestos or the like. It is effective especially when asbestos and other fibers are counted while they are being identified, or when fibers finer than those measured with an optical microscope are contained.
- (2) Summary Fibrous particles such as asbestos or the like which float in the air are sampled with the filter specified in 4.1 (1). It is converted to an electron microscope observation sample, and the class, number, size, etc. of asbestos and other fibers on the observation sample are measured with an electron microscope. In this method, the objective range, sample preparation method, etc. are different according to the type of filter and the type of the electron microscope, the summary thereof is shown in Table 1.

Table 1. Summary of objective range or the like of method using electron microscope

Method	Electron microscope to be used	Type of filter	Sample preparation method	Summary of sample preparation	Objective range and features of method (Informative reference)
SEM method	SEM+EDS	Poly-carbonate filter	SEM-method 1 (poly-carbonate filter method)	Sample airborne particles by filtration under reduced pressure with a poly-carbonate filter, cut off a part of it, and conductively treat it to make a sample.	Though handling of the filter is slightly difficult, the microscopic image is easily observed and its counting is easy. Further, the type of fiber can be identified to some extent from the chemical composition.
		Cellulose ester filter (membrane filter)	SEM-method 2 (low temperature incineration method)	A part of an acetyl acetate membrane filter wherein airborne particles are sampled by filtration under reduced pressure is bonded to a gold vacuum-evaporated slide or a nickel sheet with acetone vapor, and incinerated with a low temperature incineration apparatus to make a sample.	Since the sample can be observed under the same condition as that for the method in 3.1 and as the filter burns off, the fiber comes to be easily visible, the same filter as that for an optical microscope and a TEM method can be observed. Further, the type of fiber can be identified to some extent from the chemical composition.

Table 1. (continued)

Method	Electron micro-scope to be used	Type of filter	Sample preparation method	Summary of sample preparation	Objective range and features of method (Informative reference)
TEM method	TEM+EDS		TEM-method 1 (PVA and carbon extraction method)	A part of an acetyl-acetate membrane filter wherein airborne particles are sampled by filtration under reduced pressure is bonded to a slide glass with acetone vapor to be incinerated at a low temperature. A residual particle face is hemmed with a cellophane tape. Polyvinyl alcohol (PVA) solution is dripped thereon, spreaded, and dried. After drying, the PVA membrane is peeled off, reversed, and carbon is vacuum-evaporated thereon. The PVA membrane is dissolved by floating on hot water. The remained floating carbon membrane is scooped up with a nickel mesh to make a sample.	The asbestos and fibrous particles of the same size as that which can be identified with an optical microscope can be also effectively measured by this method. The observed visual field is extremely easily visible, and the asbestos fiber can be almost identified to its type from the chemical composition and crystalline structure.

Table 1. (continued)

Method	Electron microscope to be used	Type of filter	Sample preparation method	Summary of sample preparation	Objective range and features of method (Informative reference)
			TEM-method-2 (suspension and re-filtration method)	A part of an acetyl-acetate membrane filter wherein airborne particles are sampled by filtration under reduced pressure is bonded to a slide glass with acetone vapor and incinerated at a low temperature. The slide glass is put into isopropyl alcohol solution, and residual particles are suspended again with an ultrasonic dispersing device. The suspension is filtered under reduced pressure onto a polycarbonate filter, and carbon is vacuum-evaporated thereon. A part thereof (3 mm square) is cut off and placed on a nickel mesh grid. The polycarbonate filter is dissolved with chloroform to make a sample.	Since the number of fibers in each visual field can be adjusted in treating the sample in addition to the abovementioned, restriction of sampling can be reduced. It is suitable for measurement in surroundings of a comparatively low density.

Remarks 1. The filter shall be as specified in 4.1 (1).

2. SEM, which is the abbreviation of scanning electron microscope, shall be as specified 5.2.1.
3. EDS, which is the abbreviation of energy dispersion X-ray spectrometer, shall be as specified in 5.2.1.
4. TEM, which is the abbreviation of transmission electron microscope, shall be as specified in 5.2.1.

4. Collection of airborne fibrous particles

4.1 Sample collector The sample collector shall be composed of a filter,

a filter holder, a flowmeter, a suction pump, and a connecting tube as given in Fig. 1.

- (1) Filter The filter shall be the circular cellulose ester filter (membrane filter) of at least 99 % collection efficiency for particles of 0.3 μm particle size.

Further, in the case of the polycarbonate filter method of the scanning electron microscope specified in 5.2.2 (a), it shall be the filter wherein gold or carbon is vacuum-evaporated onto a polycarbonate filter of 0.8 μm pore diameter.

Informative reference: The membrane filter used for measurement of asbestos is generally that of 0.8 μm pore diameter.

- (2) Filter holder A filter holder corresponding to the size of a filter is used, which is so constructed that the filter can be easily detached or attached and perfectly sealed.

Remarks: The open face type filter holder given in Fig. 2 is used⁽¹⁾ ⁽²⁾.

Further, when a sample is taken in the air flowing at a specific velocity in a duct or the like, the filter holder for isokinetic sampling as given in Fig. 3 (extracted from an example of the dust collector using circular filter paper in Fig. 12 of JIS Z 8808) is referred to.

Notes ⁽¹⁾ When the open face type filter holder type 2 as given in Fig. 2 is used, cares shall be taken for deformation, breakage, etc. of filter paper fitted as a backup.

⁽²⁾ When the polycarbonate filter is used, a glass fiber filter or a membrane filter is used as a backup filter for the purposes of reinforcement and prevention of contamination.

- (3) Flowmeter As the flowmeter, the area flowmeter or integrating flowmeter specified in JIS B 7551 is used.

When the area flowmeter is used, the flow rate measuring method specified in JIS Z 8761 applies.

Remarks: The integrating flowmeter is used when a sample is taken from the air flowing at a specific velocity principally in a duct or the like.

- (4) Suction pump The suction pump shall have the intended sample volume and suction pressure, be free from pulsation, and withstand continuous operation for a long time.
- (5) Connecting tube As the tube⁽³⁾ connecting the filter holder, flowmeter, and suction pump, capable of withstanding suction pressure shall be used.

Note ⁽³⁾ Cares shall be taken for sealing the joint part of the connecting tube.

Fig. 1. Structure of sample collector

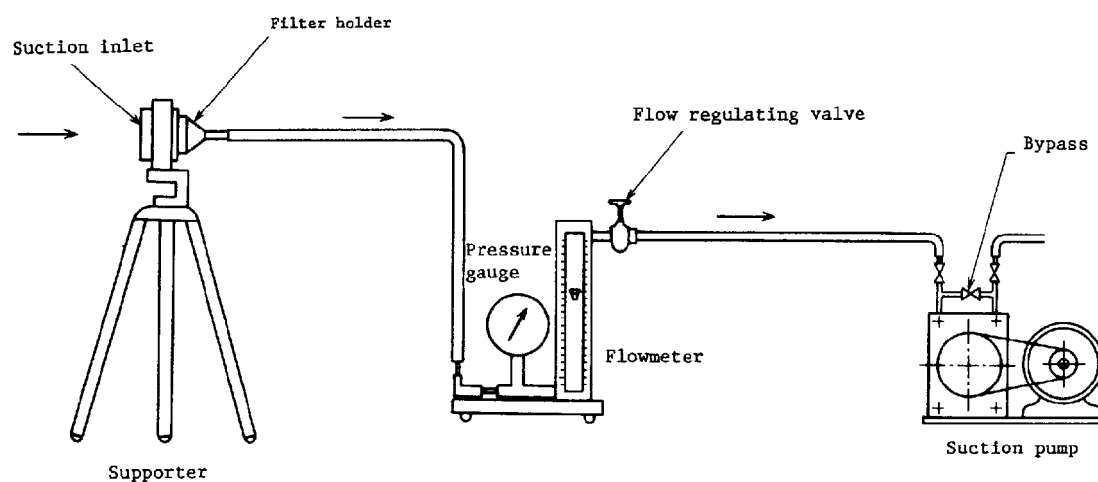
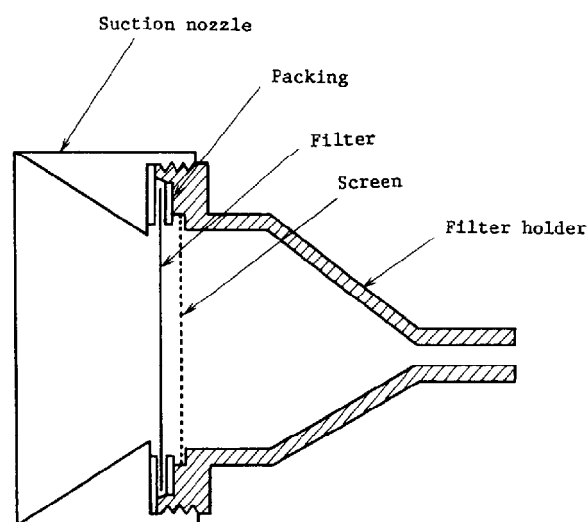


Fig. 2. Example of open face type filter holder

Type 1



Type 2

