The Development of Thimble Filter for the Purpose of Collecting Dust in Stack Gas

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1. Cellulose, borosilicate glass and silica glass fiber thimble filters were achieved and inspected for the purpose of collecting micron order particles of dust in stack gas.
2. After several experiments, thimble filters of the size 25 mmD×90 mmH among other different sizes was shown most adequate for the practical use.
3. These thimble filters were revealed to have excellent characters compared to those of traditional filters.

Background

For the past few decades, air and water pollution in Japan has been increasing rapidly and affecting the environment deeply.

To cope with the pollution, efforts have been made first to establish several national standards concerning the method of determining the degree of pollution as a Japanese Industrial Standard (JIS).

In measuring the dust concentration, and for a quantitative chemical analysis of heavy metals in stack gas, packed glass wool cartridges of several shapes have been used along with the JIS Z 8808, "Methods of Measuring Dust Contents in Stack Gas" (1970).

These cartridges, however, have many drawbacks which can be summarized as follows :

- 1) Techniques of homogeneous filling-up the cartridge with glass wool are very tedious.
- 2) Sulfur dioxide in stack gas is adsorbed

into the glass wool at various rates according to the nature of gas and glass wool.

- 3) Blanks of heavy metals contained in glass wool are high in many cases.
- 4) Weighing of collected dust is doubtful as to the gaining of a constant reliability.
- 5) Pressure drop of sampling gas flowing into the cartridge is great, according to the air resistance of glass wool clogged by dust.

Although the thimble filter has been produced since the 1930's for chemical uses, such as Soxhlet's extractor use under the name of cylindrical filter paper, it was in 1970 that the thimble filter was adopted for the first time as a collector of dust in the stack gas. Then, a glass fiber thimble filter was adopted in 1971 and a silica fiber thimble filter, in 1973, successively. These thimble filters seem to be superior to packed glass wool cartridges in several points, which may be summarized as follows :

- 1) Location : a) 10-6, Yotsuyakami-cho, Kawasaki-ku, Kawasaki ; b) 1-33, Yayoi-cho, Chiba ; c) 3-7, Hon-cho, Nihonbashi, Chuo-ku, Tokyo ; d) 6-21-1, Nagao, Tama-ku, Kawasaki ; e) 4-26, Ukima, Kita-ku, Tokyo.

- 1) It is easy to prepare the cartridge and fit it into the sampling apparatus.
- 2) Since only a standardized thimble filter is required for the mere collection of dust, the repeatability may be higher than that of a glass wool cartridge system.
- 3) The sampling gas flow rate is kept large, as the pressure drop of the flow may be minimized, and the sampling duration can be shortened.
- 4) Using a small-sized vacuum pump, the whole sampling system can be easily transported.
- 5) It is only necessary to determine the weight of the thimble filter. As the weight of the thimble filter is as light as 1/10 of the packed glass wool cartridge, the weighing is very easy and it is expected to minimize the weighing errors.
- 6) The thimble filter is packed up with a titanium sheath which is supported by a holder consisting of a silica spiral spring and the pedestal ring plate.

The sheath acts to prevent the destruction of the thimble filter when a dampened gas stream is sucked in and the dusts collected on the inside wall of the thimble filter get wet, clogging the micro-drainage.

The cartridge device with a cellulose, glass or silica glass thimble filter was first adopted in the JIS K-0097, "Method for Determination of Cadmium and Lead in Stack Gas," published on May 1, 1972. At that time, however, the titanium sheath and its holder were not adopted, bringing about some difficulties in the cases of dampened gas. In the JIS K-0096, "Method for Determination of Chromium and Manganese in Stack Gas," which is now under preparation, the titanium sheath and its holder are adopted for the first time, and the thimble filter is composed of silica glass fiber (95 % SiO_2).

This paper will discuss the manufacture and the applicability of these thimble filters and the results of the collection of dusts, etc. Analysis methods of metal components contained in stack gas according to JIS.

JIS Z-8808 "Methods of measuring dust contents in stack gas" will apply correspondingly to the principle of sampling for the purpose of measuring Pb, Cd, Cr and Mn concentrations in stack gas. As illustrated in Figure 1, this sampling train consists of the sample probe, vacuum line, vacuum pump and wet test meter. The packed glass wool cartridge method which has been used

Fig. 1. Sampling Train

Fig. 2. Cartridge Assembly

conventionally as a collection device is unsuitable for the metal component analysis because of many problems involved as mentioned earlier. Therefore, the author, *et al.*, who hinted at applying a glass fiber thimble filter or a silica glass fiber thimble filter to JIS for the metal component analysis purpose, have examined materials and an impure metal removing method *etc.* from various viewpoints, resulting in obtaining an excellent thimble filter. At the same time, the cartridge as a collection device was also examined so extensively that it was decided to adopt the cartridge as shown in Figure 2 as a standard cartridge.

Prior to the analysis, dusts collected in the thimble filter are pretreated in one of the following ways, and then Pb, Cd, Mn and Cr concentrations are measured with the atomic absorption spectrometry method.

Pb, Cd; In JIS K-0097

- 1) Decomposing method with nitric acid and hydrogen peroxide.
- 2) Extraction method with nitric acid only (in a case when any organic matters are not contained).
- 3) Extraction method with hydrochloric acid and hydrogen peroxide. After ashing

with a low temperature ashing device of the above three methods, a prescribed suitable pretreatment will be chosen according to the sample conditions, such as low organic matter content, high organic content, *etc.* Cr, Mn; In JIS K-0096, a Cr-containing sample is ashed in an electrical muffle furnace, and then fused with Na_2CO_3 and NaNO_3 . As for a Mn-containing sample, after it is ashed in an electrical muffle furnace, Mn is extracted with hydrochloric acid and hydrogen peroxide, thereby obtaining the sample solution.

Production Method of Thimble Filters

The production method of a thimble filter is shown in Figure 3 A, B, and C.

A. Cellulose Fiber Thimble Filters—

This thimble filter is the same as the thimble filter known as sohxlet's extractor use. The material is α -cellulose of 98 % purity.

B. Glass Fiber Thimble Filters—

The material of this thimble filter is usually the same as that used in the sampling of atmospheric particulate matters with a high-volume air sampler. The raw material is borosilicate glass fiber with a fiber diameter of $0.3 \mu\text{m}$, whose chemical composition is shown in

Fig. 3-A. Production Method of Cellulose Fiber Thimble Filter

(a) Toyo Tokushi Co., Ltd.

Fig. 3-B. Production Method of Borosilicate Glass Fiber Thimble Filter

(b) Nippon Glass Fiber Co., Ltd. MLF-4A

(c) Nippon Mining Co., Ltd. Special Grade

(d) Showa Denko Co., Ltd. Special Grade

(e) K. Oikawa, S. Ogawa, T. Tanaka, J. Yamauchi, Japan Society for Analytical Chemistry, Meeting at Hiroshima, Oct. 1968.

Fig. 3-C. Production Method of Silica Glass Fiber Thimble Filter

(f) Nippon Glass Fiber Co., Ltd. MLF-Q

(g) Nissan Chemical Ind. Co., Ltd. Alumina-sol No. 100

(h) Nippon Mining Co., Ltd.

Table I.

C. Silica Glass Fider Thimble Filters—

Conventionally, a thimble filter made of silica glass fiber have not been used for the purpose of atmospheric sampling or stack gas

sampling. However, since a thimble filter made of borosilicate glass fiber has a greater reactivity to the gas components, and since it is impossible to be used in a stack gas with temperatures exceeding 500°, filters

TABLE 1. Chemical Composition of the Borosilicate Glass Fiber

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	Na ₂ O	K ₂ O	B ₂ O ₃
68.63%	2.6%	0.13%	0.04%	7.31%	2.53%	13.5%	1.36%	3.50%

TABLE II. Chemical Composition of the Silica Glass Fiber

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	Na ₂ O
94.6%	2.54%	0.5%	0.02%	0.52%	0.27%

TABLE III. Physical Properties of Three Kinds of Thimble Filter

	Filter size mmD × mmH	Cellulose fiber	Borosilicate glass fiber	Silica glass fiber
Weight (g/thimble)	20 × 90	2.1	1.0	1.0
	25 × 90	2.8	1.7	1.6
	28 × 100	4.6	2.4	2.3
Thickness (mm)	20 × 90	1.2	1.6	1.6
	25 × 90	1.3	1.6	1.6
	28 × 100	1.4	1.7	1.9
Air resistance ^{a)} (sec/100 ml)		18	12	6
Tensile strength ^{b)} (g/15 mm)		6000	300	300
Bursting strength ^{c)} (kg/cm ²)		2.4	0.4	0.3

a) JIS-P 8117 air resistance of paper (φ 1 cm)

b) JIS-P 8113 tensile breaking strength of paper and paper board

c) JIS-P 8112 bursting strength of paper

made of silica glass fiber have been developed by the author, *et al.*²⁾ The chemical composition of a silica glass fiber is shown in Table II.

Physical Properties of Thimble Filters

With regard to the thimble filters made of cellulose fibers, borosilicate glass fibers and silica glass fibers, the following items concerning their physical properties were measured, and the results were shown in Table III.

- (1) Weight
- (2) Thickness (by a dial-gage type thickness meter)
- (3) Air resistance
- (4) Tensile strength
- (5) Bursting strength

The results show that a cellulose fiber thimble filter has the greatest weight, which is approximately twice as much as that of a borosilicate glass fiber thimble filter or of a

silica glass fiber thimble filter.

Similarly, an air resistance, which is regarded as an indicator of pressure drop, records the highest value for a cellulose fiber thimble filter. This value is reduced in the order of a borosilicate glass thimble filter first and then in a silica glass thimble filter. However, no trouble caused by this pressure drop will be expected when these thimbles filters are used in the common stack sampling procedure.

Filtration Characteristics of Thimble Filters

In general, the properties required for a filter are shown by the following three items;

- (1) a high collection efficiency
- (2) a low pressure drop
- (3) a high holding capacity

In order to determine the filtration characteristics of the above described thimble filters, the collection efficiency and pressure

2) K. Homma, K. Oikawa and R. Ogawa, *Kogai*, **7**, 58 (1972).

Fig. 4. Characteristic Curves on Collection Efficiencies of Thimble Filters Loaded with Lead Particle

silica glass fiber thimble filter : 28 mmD × 100 mmH
 filtering air flow rate 20 liter/min
 (a) lead aerosol dp : 0.15 μ m
 (b) lead aerosol dp : 0.30 μ m

drop during filtration were measured using lead fume with a particle diameter of submicron order. Simply diffused lead aerosol, which was used in this measurement, was yielded with fume generation equipment heated by a high-frequency wave (Figure 4 and 5).

When aerosol in stack gas is collected, an isokinetic sampling method is generally used. As the common stack gas velocity ranges from 5 to 20 m/sec, the suction air volume becomes 7.5 to 30 liter/min, using a sampling nozzle with a diameter of 6 mm.

Fig. 5. Characteristic Curves on Pressure Drop of Lead Particle Loaded Thimble Filters

$$\Delta P = BW^m$$

thimble : 28 mmD × 100 mmH
 flow rate 20 liter/min
 m : dust load g/cm²

- ① borosilicate Pb 0.15 μ m B:0.04 m :1.6
- ② silica Pb 0.15 μ m B:0.007 m :1.8
- ③ silica Pb 0.30 μ m B:0.002 m :1.7

The above measurement was taken place in such ranges.

Looking at the collection efficiencies classified by particle size, the test results show that a thimble filter made of glass fiber marks the highest efficiency, followed by a silica glass fiber thimble and a cellulose fiber thimble filter, in that order. (Table IV)

Concerning cellulose fiber thimble filters, no significant efficiency differences were

TABLE IV. Collection Efficiencies of Thimble Filters for Submicron Lead Aerosol

Particle size μ m	Kind of filter	Flow rate on filtration liter/min			
		7.5	10	15	20
0.07	cellulose	89 %	87 %	86 %	86 %
	silica glass	97.1	97.0	96.3	95.8
	borosilicate glass	99.1	99.3	99.8	99.3
0.13	cellulose	88	87	88	89
	silica glass	99.5	98.7	97.7	96.9
	borosilicate glass	99.8	99.8	99.8	99.8
0.25	cellulose	88	89	91	94
	silica glass	99.4	99.0	98.6	98.5
	borosilicate glass	99.91	99.92	99.93	99.93

TABLE V. Collection Efficiencies of the Silica Glass Fiber Thimble Filter for Submicron Lead Aerosol

Particle size μm	Filter size $\text{mmD} \times \text{mmH}$	Flow rate on filtration liter/min			
		7.5	10	15	20
0.15	20 $\times$ 90	99.5%	99.0%	98.0%	97.5%
	25 $\times$ 90	98.8%	98.2%	97.1%	95.9%
	28 $\times$ 100	99.8%	99.6%	99.2%	98.9%

detected among three kinds of particle sizes of submicron order used in this experiment. In the case of glass fiber thimble filters, which consists of the finest fibers, it was made clear that the larger the grain size and the flow rate became, the higher the collection efficiency, due to inertia. With regard to the collection efficiencies classified by the sizes of thimble filter made of silica glass fibers, as seen in Table V, the inertial collection effect to lead particles with a fine diameter of $0.15 \mu\text{m}$ became low, and the diffusion collection effect became predominant in turn, since silica glass fiber is thicker than borosilicate glass fiber and its packing density is low, and since the voids existing among the fibers are filled with colloidal alumina, which is used to increase the strength of silica glass fiber thimble filters. It is clearly shown by the experimental results that the larger the diameter and the filtration area, and the slower the flow rate on filtration, the higher the collection efficiency. Next, the results concerning the pressure drop caused by loading will be described. The pressure drop increment was obtained by subtracting the initial pressure drop, determined at the beginning from the pressure drop caused by accumulation, which was measured at a predetermined time interval. Then the relation between the loaded quantity and the values of pressure drop thus obtained was plotted. The results are illustrated in Figure 5. Each loaded quantity — pressure drop

curve can be abstracted in the following equation :

$$P = BW^m$$

Where, P : pressure drop increment, W : Weight of accumulated particles per thimble filter, B, m : constants given to each thimble filter. Though this scarce amount of data can not lead to a full conclusion on this experiment, it is estimated that no significant differences are found for the value of m , while the value of B differs greatly according to filter materials and collected particle sizes.

Metal Components Contained in Thimble Filters

When the metal components in the particle substances sampled from smoke conduit are analyzed quantitatively, it is impossible to separate the particle substances alone from the the thimble filter. Consequently, the analysis operation is usually performed by extracting the metal components into acid by treating the whole thimble filter to which the particle substances stick. In this case, the greater the amount of metals contained in the thimble filter, the greater the measuring error becomes. Therefore when a thimble filter is produced, the method of quickling which can minimize the acid soluble components is adopted. The size of the thimble filter used for the analysis is 20 mmD $\times$ 90 mmH. The amounts of Pb, Cd, Zn, Cu, Ni, Co and Fe are analyzed according to the manners described in JIS K-0097, while Cr and Mn are analyzed according to JIS K-0096.

TABLE VI. Metal Contents in the Thimble Filters by Acid Extraction Method

	Cellulose fiber		Borosilicate glass fiber		Silica glass fiber	
	$\mu\text{g}/\text{thimble}$	$\mu\text{g}/\text{g}$	$\mu\text{g}/\text{thimble}$	$\mu\text{g}/\text{g}$	$\mu\text{g}/\text{thimble}$	$\mu\text{g}/\text{g}$
Pb	4.6 (1.2) ^{a)}	2.3 (0.5)	3.6 (1.2)	3.7 (1.2)	12.8 (1.9)	11.3 (1.5)
Cd	0.1 (—)	0.1 (—)	0.1 (—)	0.1 (—)	0.1 (—)	0.1 (—)
Mn	0.7 (0.2)	0.4 (0.1)	0.6 (0.1)	0.7 (0.2)	1.6 (0.1)	1.4 (0.1)
Zn	7.0 (1.2)	3.5 (0.5)	3.8 (0.9)	3.9 (0.7)	8.6 (3.4)	7.6 (2.8)
Cu	6.1 (1.8)	3.0 (0.8)	4.8 (0.6)	4.9 (0.7)	18.6 (2.9)	16.5 (2.6)
Ni	0.8 (0.2)	0.4 (0.1)	1.2 (0.7)	1.3 (0.7)	2.3 (1.3)	2.0 (1.3)
Co	0.1 (—)	0.1 (—)	0.1 (—)	0.1 (—)	0.1 (—)	0.1 (—)
Fe	45 (8)	22 (4)	69 (31)	72 (33)	282 (44)	250 (39)
Cr	2.3 (1.7)	1.0 (0.4)	6.2 (2.3)	6.9 (2.5)	41.6 (5.4)	43.6 (6.1)

filter size : 20 mmD×90 mmH

^{a)} standard deviation

Table VI shows the results of this analysis. The following facts were found from these results. Cellulose fiber thimble filters and borosilicate glass fiber thimble filters contain an extremely small amount of metals which may cause no problem in the actual application. However, a large amount of Fe reaching to 250 μg per lg of the thimble filter was extracted, especially from free silica glass thimble filters. This is probably because silica glass filter, used as a material, is rich in iron components. Furthermore, Fe and Cu also marked a high concentration, but these do not hinder the ordinary experimental measurement.

Influence of Sulfur Dioxide

On sampling the stack gas, a large weight error has often been experienced because sulfur dioxide is adsorbed in glass wool filters and glass fiber filters. A. Matsuura, *et al.*³⁾ reported that the amount of the adsorbed

sulfur dioxide in a glass fiber filter is considerably large.

This was found by their experiment with discharged gas from a heavy oil burning boiler, using a thimble-type filter.

Wesley C. L. Hemeon, *et al.*⁴⁾ also reported the adsorption of sulfur dioxide as a result of an experimental collection of stack gas in the actual smoke conduit.

The author *et al.*⁵⁾ conducted a test to determine the adsorbed SO_2 quantity using an experimental device which is equipped with a differential thermal balance as indicated in Figure 6.

The results of the test are shown in Figure 7, thirty minutes after the temperature of gases which contain 1000 ppm of SO_2 were controlled to room temperature, 100, 200, 300 or 400°, the gas was flowed into the device at a flow rate of 0.35 liter/min, and after an hour, the rate of the weight increment of

3) A. Matsuura, *Kogai*, **8**, 80 (1973).4) Wesley C.L. Hemeon and Arthur W. Black, *J. Air Poll. Control Assoc.*, **22**, 516 (1973).5) Y. Tamori, M. Matsumoto, N. Kogure, K. Nakamura and K. Imagami, *J. Japan Soc. Air Poll.*, **8**, 6 (1973).

Fig. 6. Schematic Diagram of Experimental Apparatus

Fig. 7. Adsorption of 1000 ppm SO₂ Gas on Thimble Filters

△: borosilicate glass fiber thimble filter
×: silica glass fiber thimble filter

the filter to the weight measures prior to the gas introduction was calculated for each temperature level. The borosilicate glass fiber filter showed the largest adsorption at temperatures above 300°. The adsorption capacity of silica glass fiber filters showed a small adsorption quantity and no temperature reliability. Meanwhile, in both cases—using 2,000 ppm gas and in a heated atmosphere—the same tendency prevailed.

Heating Loss

It was once considered that a silica glass

fiber thimble filter could be used for the purpose of metal content analysis and was most suitable for dust collecting operations because of its weak adsorption of sulfur dioxide. However, the unexpected obstacle of heating loss, became evident through the process of experimentation. It is thought that the water contained in colloidal alumina, which is added to increase the binding force during the production process, gives rise to the heating loss. Alumina transforms with the increase of temperature as shown in Figure 8.

So far, the filter has been generally dried at a temperature of $110^{\circ} \pm 5^{\circ}$. However, Figure 6 indicates that if the filter is heated at a temperature of 1000–1100°, alumina can be converted into β -Al₂O₃ or α -Al₂O₃, resulting in the removal of almost all the water contained.

On the other hand, alumina which becomes a powdery substance because of this heating treatment loses its original binding effect. By heating it at a temperature of only 1000°, where alumina transforms to β -Al₂O₃, there may be danger of the binder becoming brittle.

Fig. 8. Transformation of Colloidal Alumina with Temperature

Therefore, the experiment shown in Figure 9 and 10 was conducted to obtain the optimum heating temperature and heating period. Figure 9 shows the relation between the heating period and heating loss at different temperatures. From this, we see that the filter cannot reach a constant weight condition after heat treatment for 7 hours at 400°, and even at 600° it still cannot, though the curve becomes less steep. Meanwhile, it is found that after 2 hours of heating at 800° no heating loss is seen.

That is, the filter is in a constant weight condition. Figure 10 shows the heating losses at different temperatures of the filter heat treated at 800° for 2 hours compared with the untreated filter. It is seen from this that a filter treated by heat at 800° has no heating loss at any temperature. On the contrary, untreated filter shows a sudden heating loss around 500°. This confirms that

Fig. 9. Heating Loss at 400°, 600° and 800° on Silica Glass Fiber Thimble Filter

○ : 400° × : 600° △ : 800°

Fig. 10. Effect of Heat Treatment (800°-2 hr) on Silica Glass Fiber Thimble Filter Compared with Untreated one

× : heat treatment (800°-2 hr)
○ : untreated

the heating loss can be minimized by filters treated by heat at a temperature of 800° for a period of 2 hours.

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