

Chapter 2 Sample preparation, and others

[Summary of sample preparation method]

Sample preparation is highly important to perform analytical procedures. Errors caused by sample preparation may be greater than those by analysis, depending on objectives and subjects of analysis. Therefore, sample preparation has to be done very carefully.

The amount of sample used for determination is usually 1–10 g (10–50 g for mycotoxins, pesticide residues, and others), while the amount of parent sample is 20 kg to several tons. In most analyses, a portion of the sample is analyzed, and the results are used to represent the total composition of the sample.

Regardless of parent sample size, the following method is actually used: the total sample is divided into many smaller segments, whose respective small portions are mixed. The method in which portions are extracted from a parent sample is called sample division method. Such methods include using a riffle sampler (Fig. 2-1) and conical quartering (a method in which a sample is piled in a cone shape, and pushed down vertically from the top. Then, the flat sample is divided into quarters and the opposite two portions are mixed to be used as one sample. This method tends to cause errors.)

Dry matter (%), the term described in the database of the Japan Standard Feed Ingredients Chart, represents the contents (%) of respective components in the dry matter converted from the contents (%) of the respective components other than water in the original sample, according to the following conversion formula:

$$\text{Contents (Respective components) in dry matter (\%)} = A \times \frac{100}{100 - B}$$

A: Contents in the original sample (respective components) (%)

B: Water content in the original sample (%)

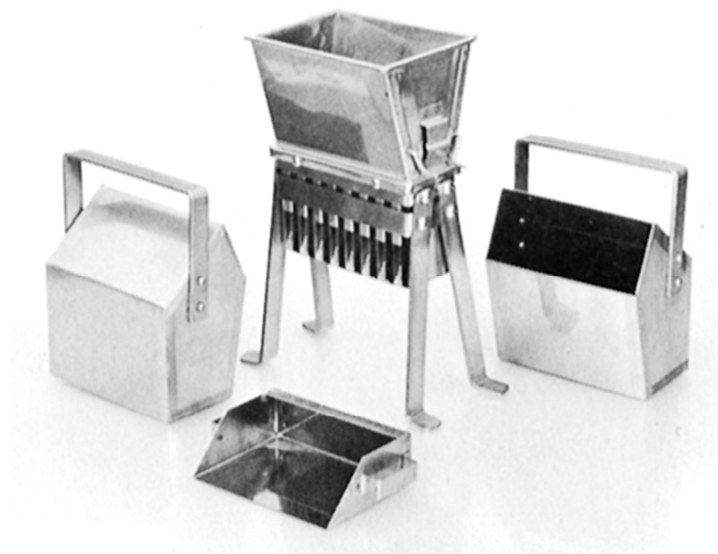


Fig. 2-1 Riffle sampler

The main part is made of stainless steel and the inside is finished to be flat and smooth. The switching blades attached to the upper sample dish prevent the falling sample from remaining inside and allow the sample to completely fall into the receiver.

[Method listed in the Analytical Standards of Feed]

1 Collection and storage of samples [1, Chapter 2, Analytical Standards of Feed]

Samples shall be collected and stored in accordance with the “Method for sampling, etc. of feed, etc.”, annex of “Operation Guide for Inspection of Feed, etc.” (May 10, 1997, 52-Chiku-B No. 793, Notice of Director of Livestock Industry Bureau of Ministry of Agriculture and Forestry).

2 Sample preparations [2, Chapter 2, Analytical Standards of Feed]

Unless otherwise specified, samples for analysis^[1] shall be prepared as follows and stored in a tight container^[2], such as a stoppered glass bottle.

In sample preparation, procedures are performed promptly in order to avoid an increase or decrease of water content in samples or chemical changes of samples.

- (1) For a dry sample, comminute the sample^[3], and put it through a 1-mm sieve^[4] to mix it well.
- (2) For a wet sample^[5], mix the sample, and then divide it by a riffle sampler to separate the required amount of not less than 200 g, and then weigh the separated sample.^[6] Dry the sample at not more than 60 °C, leave it to stand in a room^[7] to be air-dried (hereinafter referred to as pre-dry), weigh it again, and prepare the sample according to the procedures (1). Then, convert the analytical value of the sample into the contents of the original sample.^[8]
- (3) When a sample cannot be comminuted because of high fat content^[9], mix the sample, and then divide it by a riffle sampler to separate the required amount of not less than 200 g, and then weigh the separated

sample.^[6] Pound it in a mortar and transfer it to a beaker. Wash the sample adhered to the mortar with diethyl ether, add the washings to the beaker, leave the beaker covered with aluminum foil to stand, and decant the diethyl ether into a 1,000–2,000-mL volumetric flask.

Pour diethyl ether on the undissolved residue in the beaker, leave the beaker covered with aluminum foil to stand for 1 day, and decant the solution into the volumetric flask. Then, filter the undissolved residue through a large filter paper (No. 5A), which was previously weighed, wash the filtrate with diethyl ether^[10], and add the washings into the volumetric flask (hereinafter referred to as pre-extraction ^{*1}).

Next, air-dry the undissolved residue with the filter, dry it at 60–80 °C, leave it to stand in a room^[7], and air-dry it.^[11] Again, weigh it and then prepare the sample by the method (1). Convert the analytical value of the sample into the content of the original sample.

*1. See “3.1 Crude fat” in Chapter 3.

[Notes and precautions]

[1] Samples for the inspection of feed should be collected from the inspection target lots in accordance with the methods defined in the attached “Operation Guide of inspection of Feeds, etc”.

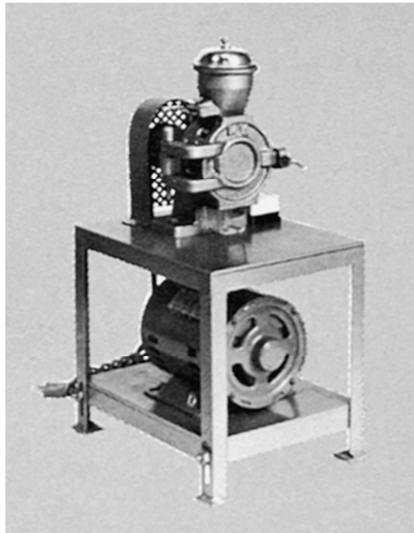
As for the methods of sampling and sample division by JIS, see JIS M 8100 “Particulate materials -- General rules for methods of sampling.”

[2] The Japanese Pharmacopoeia specifies a container which “protects the contents from extraneous solids or liquids and from efflorescence, deliquescence, or evaporation under ordinary or customary conditions of handling, shipment, and storage.” Such containers include glass bottles, plastic containers, polyethylene bags, and cans.

[3] Usually, a Wiley mill or a rotor speed mill (ultra centrifugal mill) is used to comminute feed (Fig. 2-2).

For some samples, a ball mill, a Yagen grinder, a hand-turned chopper, a household blender (or a coffee mill), a mortar, and others are used.

Samples with high water or fat content cannot be comminuted using the above mills, except mortar, without pre-treatment.



Wiley mill



Ultra centrifugal mill

Fig. 2-2 Mills

- [4] See JIS Z 8801 “Test sieves”.
- [5] This procedure is available only when components to be determined are stable against heat.
- [6] It can be weighed using a crude balance with sensitivity not less than 0.1 g.
- [7] Dried samples are left to stand for 1–2 days in a room to reach near equilibrium to air humidity.
- [8] The contents (%) of respective components in the pre-dried sample are converted into the contents (%) of respective components other than water in the original sample according to the following conversion formula:

$$\text{Contents of respective components in the original sample (\%)} = \frac{100 - A}{100} \times B$$

A: Loss by pre-drying (%)

B: Contents of respective components in the sample undergoing pre-drying (%)

- [9] It is rarely observed in meat meal. Samples with high water content are air-dried, and then delipidated.
- [10] Collect both filtrates and washings in the volumetric flask. When crude fat is not to be determined, collection of extracts is not required.
- [11] The contents (%) of respective components in the pre-extracted sample are converted into the contents (%) of respective components in the original sample according to the following conversion formula:

$$\text{Contentst of respective components in the original sample (\%)} = \frac{100 - A}{100} \times B$$

A: Loss by pre-extraction (%)

B: Contents of respective components in the pre-extracted sample