Method for sampling, etc. of feed, etc.
(Annex of “Operation Guide for Inspection of Feed, etc.”, May 10, 1977, 52-Chiku-B No. 793)

I  Method for sampling of feed, etc.
   1  Formula feed, etc.
      (1) Inspection target
          The size of the inspection target should be as follows. However, in an inspection to ensure
          safety, when a single portion delivered to a consumer is small, when the size of a production lot
          or sales lot is smaller than the quantity defined below, or in other cases, the inspection target can
          be reduced as necessary.
          i. Packaged feed
             (i) As for feed packaged in paper bags, about 50 to 1,000 bags of an identical production lot.
                 When an identical production lot cannot be distinguished, the number of bags should be 50
                 to 300.
             (ii) As for feed contained in large shipment containers (which refer to containers each
                 having a capacity of 200 kg or less, the same applied to 1 below), about 2 to 5 containers of
                 an identical production lot. When an identical production lot cannot be distinguished, the
                 number of containers should be 2 or 3.
          ii. Unpacked feed
             (i) For feed loaded on a bulk transport vehicle, the inspection target should be its load
                 capacity.
             (ii) For feed in a hopper wagon, the inspection target should be about 3 tons.
             (iii) For feed in a vessel, the inspection target should be the total amount loaded in the
                 hatch.
             (iv) For feed other than those described above, the inspection target should be about 1 ton or
                 more but not more than 20 tons of the same production lot. When the same production lot
                 cannot be distinguished, the inspection target should be 1 ton or more but no more than 6
                 tons. However, the following applies to single ingredient feed. For a main ingredient,
                 the inspection target should be 100 tons or more but not more than 1,000 tons, and for an
                 auxiliary ingredient, the inspection target should be 10 tons or more but not more than 100
                 tons.
      (2) Number of extractions or portions
          The number of extractions or portions should be as follows. It should be noted that the proviso
          in (1) applies to this case.
          i. Packaged feed
             (i) Feed packaged in paper bags
                 The number of bags listed in the table below is randomly extracted from the inspection
                 target by (1)-i-(i). However, when the maximum particle diameter of the feed for
                 inspection is 15 mm or greater (in case of a feed containing pellets, the diameter of the
                 pellets is 10 mm or greater), the number of bags extracted should be twice the said number,
and in the case of microbial tests, the number of bags should be 3, regardless of the maximum particle diameter of the feed for inspection.

<table>
<thead>
<tr>
<th>Size of inspection target</th>
<th>Number of bags extracted</th>
</tr>
</thead>
<tbody>
<tr>
<td>Less than 100 bags</td>
<td>4 bags</td>
</tr>
<tr>
<td>100 bags or more</td>
<td>5 bags</td>
</tr>
</tbody>
</table>

(ii) Feed contained in large shipment containers

Two containers are extracted randomly from the inspection target as stated in (1)-i-(ii). However, in case of microbial tests, 1 container should be extracted.

ii. Unpacked feed

(i) Single ingredient feed in a tank or a silo

Equally divide the content of the tank or silo into an upper layer, a middle layer, and a lower layer, and choose a portion from each of the 3 layers. In this case, the quantity of the portion should be 10 tons or more but not more than 15 tons for main raw materials, and 1 ton or more but not more than 1.5 tons for auxiliary materials.

(ii) Other feed

Five portions are randomly selected from the inspection targets stated in (1)-ii. However, when the maximum particle diameter of the inspection target feed is 15 mm or greater (in case of a feed containing pellets, the diameter of the pellets is 10 mm), 10 portions, and in case of a feed for microbial test, 3 portions regardless of the maximum particle diameter of the inspection target feed.

(3) Method for collecting samples

The method for collecting samples should be as follows. However, when precision similar to that obtained by the method as defined below can be obtained, an auto-sampler, etc. can be used to collect primary samples.

i. Packaged feed

(i) Feed for microbial tests

Open a container, stir the contents well with a shovel for collecting samples, and then directly collect 2 shovelfuls (for feed contained in a large shipment container, 6 shovelfuls) or more of samples from each of the containers and divide almost equally into 2 samples to prepare 250 g or more but not more than 500 g each of test samples and storage samples.

It should be noted that the shovel for collecting samples and the like should be disinfected previously with ethanol, etc., and sample containers used should be sterilized.

(ii) The feed other than (i) and pelleted feed and like feed having relatively uniform quality

Open the containers, stir the contents well, and then collect 3 shovelfuls (for feed contained in large shipment containers, 6 shovelfuls) or more samples from each of the containers in almost equal amounts to make 5 kg or more in total, which is used as a primary sample.

However, in case of a liquid feed, the amount of the primary sample should be 3 kg or more.

(iii) Feed other than (i) and (ii) packaged in paper bags
Using the entire content of each of the containers as a parent sample, transfer this sample to a piece of kraft paper, etc. to mix, and then section the sample into 12 sections or more but not more than 20 sections, collect a shovelful of sample from each section with a shovel listed in the table below, and combine this sample with that collected in a similar manner from each parent sample to make 5 kg or more in total, which is used as a primary sample. However, when the maximum particle diameter of the inspection target feed is 15 mm or greater (for a feed containing pellets, the diameter of pellets: 10 mm or greater), the entire contents of any two containers should be the parent sample, section the parent sample into 7 sections or more (35 sections or more in total, when using a shovel of JIS, No. 20) or 6 sections or more (30 sections or more in total, when using a shovel of JIS, No. 30), and collect a shovelful of sample from each section to make 6 kg or more in total, which is used as a primary sample.

<table>
<thead>
<tr>
<th>Maximum particle diameter</th>
<th>Shovel for increment collection</th>
</tr>
</thead>
<tbody>
<tr>
<td>10 mm or less</td>
<td>JIS, No. 10</td>
</tr>
<tr>
<td>15 mm or less</td>
<td>JIS, No. 15</td>
</tr>
<tr>
<td>20 mm or less</td>
<td>JIS, No. 20</td>
</tr>
<tr>
<td>30 mm or less</td>
<td>JIS, No. 30</td>
</tr>
</tbody>
</table>

(iv) Feed other than (i) and (ii) contained in large shipment containers
The entire content of each container is separately flowed or accumulated onto a sheet, etc., and 5 kg or more of samples are collected per portion from any 2 or 3 portions of the flowed or accumulated feed to make parent samples. Collect samples from each parent sample in a similar manner to that of (iii) to prepare 5 kg or more in total, which is used as a primary sample. However, when the maximum particle diameter of the inspection target feed is 15 mm or greater (for a feed containing pellets, the diameter of the pellets: 10 mm or greater), the samples collected from 2 portions of any 5 portions per container, 10 portions in total, of the flowed or accumulated feed to make parent samples, collect samples in a similar manner to that of a proviso in (iii) to make 6 kg or more in total, which is used as a primary sample.

ii. Unpacked feed

(i) Feed for microbial tests
Samples are collected by the method according to i-(i) from the 3 portions extracted according to (2)-i-(ii), and test samples and storage samples each weighing 250 g or more but not more than 500 g are prepared.

(ii) Feed other than (i) which are single ingredient feed in a tank or silo
At the time of loading or unloading, the 3 portions extracted by the method of (2)-i-(i) are each sectioned into 5 sections, and one shovelful of the sample is collected from each section with the shovel JIS, No. 50 to make 10 kg or more in total, which is used as a primary sample.
(iii) Other feed

Five kilograms or more of the sample is collected from a portion to make 5 [10 when the maximum particle diameter of the inspection target feed is 15 mm or greater (for a feed containing pellets, the diameter of the pellets: 10 mm or greater)] parent samples, then the primary sample is collected by the method according to i-(iii).

However, when the quality is considered relatively uniform, the sample can be collected from any 5 portions in an amount of 500 g or more per portion, and these can be mixed to make 5 kg or more in total, which is used as a primary sample.

(4) Method for reducing samples

The primary sample collected by the method of (3) is reduced by the method described below, preparing a test sample and a storage sample. However, when a precision similar to that obtained by the method prescribed in the following can be obtained, the samples can be prepared using a riffle sampler or the like.

i. Liquid feed

After the primary sample is mixed well, it is reduced using a suitable container, and 500 g or more but not more than 1 kg of a test sample and a storage sample are prepared respectively.

ii. Non-liquid feed

In case of a primary sample having a maximum particle diameter of 5 mm or less (including feed with a particle diameter greater than 5 mm that have been pulverized to a particle diameter of 5 mm or less and then mixed), it is transferred onto a piece of kraft paper, etc. and mixed well, and then sectioned into 16 sections, and a shovelful of the sample is collected from each section with the shovel defined in JIS, No. 5, and 500 g or more but not more than 1 kg of a test sample and a storage sample are prepared respectively.

In case of a primary sample having a maximum particle diameter of 5 mm or greater, increment reduction is performed by the method according to (3)-i-(iii), and 750 g or more but not more than 1.5 kg of a test sample and storage sample are prepared respectively.

However, when the maximum particle diameter of the inspection target feed is 15 mm or greater, the entire volume of the primary sample should be pulverized, then it is transferred onto a piece of kraft paper, etc. and mixed well, and then sectioned into 16 sections, and a shovelful of the sample is collected from each section with the shovel defined in JIS, No. 5, and 500 g or more but not more than 1 kg of a test sample and a storage sample are prepared respectively.

2 Feed additives

(1) Inspection target

The size of the inspection target should be as follows. It should be noted that the proviso of 1-(1) applies to this case.

i. Feed additives packaged in paper bags

The inspection target should be about 5 bags or more but not more than 100 bags of the same production lot.
ii. Feed additives contained in large shipment containers (referring to containers each having a capacity of 50 kg or more. The same applies to (2).)

The inspection target should be about 2 containers or more but not more than 5 containers of the same production lot.

(2) Number of extraction

Number of extraction should be as follows. It should be noted that the proviso of 1-(1) applies to this case.

i. Feed additives packaged in paper bags

Bags are randomly extracted from the inspection target by (1)-i. The number of bags is listed in the table below.

However, in case of a sample for microbial test, the number of bags extracted is 3, regardless of the size of the inspection target.

<table>
<thead>
<tr>
<th>Size of inspection target</th>
<th>Number of bags extracted</th>
</tr>
</thead>
<tbody>
<tr>
<td>Less than 50 bags</td>
<td>3 bags</td>
</tr>
<tr>
<td>50 bags or more</td>
<td>4 bags</td>
</tr>
</tbody>
</table>

ii. Feed additives contained in large shipment containers

Two containers are randomly extracted from the inspection target under (1)-ii. However, in case of a sample for microbial test, the number of bags extracted is 1.

(3) Method for collecting samples

The method for collecting samples should be as follows. However, when precision similar to that obtained by the method as defined below can be obtained, an auto-sampler, etc. can be used to collect primary samples.

i. Feed additives for microbial test

1-(3)-i.-i should be followed.

ii. Feed additives other than i. in which the quality is considered relatively uniform.

Open the containers, stir the contents well, and collect almost equal amounts of 3 shovelfuls (for a feed contained in a large shipment container, 5 shovelfuls) or more of samples from each container with the shovel for collecting samples. Combine the samples to make 3 kg or more in total, which is used as a primary sample.

However, in case of a liquid feed additive, the amount of the primary sample should be 2 kg or more.

iii. Feed additives other than i. and ii. packaged in paper bags

Using the entire content of each container as a parent sample, transfer this onto a piece of kraft paper, etc. and mix it, section the parent sample into 12 sections or more but 20 sections or less, collect a shovelful of the sample from each section with the shovel defined in JIS, No. 5, and combine the samples collected in a similar manner from each parent sample to make 3 kg or more of the sample, giving a primary sample.

iv. Feed additives other than i. and ii. contained in large shipment containers

The entire content of each container is separately flowed or accumulated onto a sheet, etc., a
sample is collected from any 2 portions of the flowed or accumulated feed additive in an amount of 3 kg or more per portion to make parent samples, and the samples collected from each parent sample in a similar manner to that of iii to make 3 kg or more, which is used as a primary sample.

(4) Method for reducing samples

The primary sample collected by the method of (3) is reduced in the manner described below to prepare a test sample and a storage sample. However, when precision similar to that obtained by the method as defined below can be obtained, the test sample and storage sample can be prepared using a riffle sampler, etc.

i. Liquid feed additives

After the primary sample is mixed well, it is reduced using a suitable container, and 250 g or more but not more than 500 g of a test sample and a storage sample are prepared.

ii. Non-liquid feed additives

The primary sample is transferred onto a piece of kraft paper, etc. and mixed well, and then sectioned into 16 sections, and a shovelful of the sample is collected from each section with the shovel defined in JIS, No. 3, and 250 g or more but not more than 500 g of a test sample and a storage sample are prepared respectively.

3 Other feed, etc.

In case of a feed or a feed additive to which the method for sampling, etc. of 1 or 2 cannot be applied, a test sample and a storage sample are prepared by determining the inspection target according to the prescription of 1-(1) or 2-(1), determining the number of extraction, etc. according to the prescription of 1-(2) or 2-(2), following the methods prescribed in 1-(3) or 2-(3) and the prescription of 1-(4) or 2-(4) as precisely and uniformly as possible.

II Method for storing samples

1 Containers used to put in samples should be clean, damp-proof, and tightly sealable.

2 When a sample is put in a container, an indication label, etc. attached to the inspected feed, etc. or a copy of the same should be enclosed therein, or the name and other information for specifying the feed, etc. should be written on the container, and the container should be then tightly sealed.

3 The storage sample should be placed in a storage envelope, required informations including inspection site, etc. should be filled, and inspection officials and inspectee’s signatures, stamps, and tally-impressions should be affixed.

However, in the case that the storage sample has been prepared in a way that of a proviso in 1-1-(4)-(ii), and that the inspectee, its officers and employees, or other stakeholders are not able to attend the tally-impression of the storage envelope, inspection officials perform the tally-impression and bear the name and seal, upon obtaining the consent of the inspectee about it.

4 Test samples and storage samples should be transported in a way avoiding quality change and stored in a cool, dark place.
Inspection of pesticides in feed

Director of Animal Products Safety Division, Food Safety and Consumer Affairs Bureau, Ministry of Agriculture, Forestry and Fisheries of Japan

Recently, in order to cope with the introduction of the so-called positive list system concerning restrictions on residues of pesticides, etc. in foods, the Ordinance of the Ministry concerning standards and specifications of feed and feed additives has been revised, and the standard of residual pesticides in feed has been newly established. While we have been requesting the implementation of the inspection of feed, etc. according to the “Operation Guide for Inspection of Feed, etc.” (52-Chiku-B No. 793 as of May 10, 1977, hereinafter referred to as “Operation Guide”), it has been determined that the inspection of the residual pesticides in feed should be performed according to the methods described below. Please review the methods and take thorough measures for proper and facilitated implementation of the inspection service.

Note

1 Sampling of feed
   1 Formula feed
      The Operation Guide should be followed.
   2 Imported feed grain
      The sampling method is as described below. The proviso of I-1-(1) in the Annex of the Operation Guide applies to this case.
      (1) Inspection target
           For imported grains loaded in bulk on a ship, the inspection target is the lot set on a silo basis.
      (2) Method for collecting samples
           Samples should be collected so that the sample well represents the entire lot by using an auto-sampler, etc. when loaded in a silo, dividedly in 100 times at proper time intervals using an increment scoop (JIS No. 5 for corn; JIS No. 3 for milo, barley, wheat, rye and oats) to make a primary sample in total. For the grains that are already loaded in a silo, samples are collected by a similar manner when transferred to another silo.
      (3) Method for reducing samples
           The total amount of the primary sample is pulverized. The pulverized sample is mixed well, and is then reduced using an appropriate container to prepare a test sample and a storage sample of 500 g or more but not more than 1 kg.
      (4) Notes on reduction and preparation of samples
           At the reduction and preparation in (3), when the inspectee, its officers and employees, or other
stakeholders are not able to attend the reduction, the inspectee’s consent should be obtained in advance concerning implementing reduction and preparation conducted by the inspection officials according to the procedure (3).

3 Hay

Hay should be sampled in the manner described below. The proviso of I-1-(1) in the Annex of the Operation Guide applies to this case.

(1) Inspection target
For the hay packaged into about 25- to 60-Kg bales (hereinafter referred to as “compact bale”), the inspection target is 1 container or about 50 or more but not more than 500 bales from the same production or import lot.
For the hay packaged into about 100-Kg or larger bales (hereinafter referred to as “big bale”), the inspection target is 1 container or about 10 or more but not more than 50 bales from the same production lot or import lot.

(2) Method for collecting samples
For compact bales, 20 or more bales are randomly extracted, and 50 g or more are collected per bale to make 1 kg or more of a sample.
For big bales, 4 or more bales are randomly extracted, and 250 g or more are collected per bale to make 1 kg or more of a sample.
This sample is mixed well, and is equally divided into 2 portions to prepare a test sample and a storage sample of 500 g or more but not more than 1 kg.
When collecting samples from the bales, 10 to 20 g per spot, including the core portion so that the sample well represents the bale, are collected from a number of spots in the bale.

II Measures based on the results of the analysis

1 Cases with analytical values higher than standard values
When the results of the analysis exceed the standard values, the following measures should be taken:

(1) Formula feed and hay
Instructions, etc. to recall the inspection target lots should be given to feed manufacturers, etc. Required measures should be taken based on “Manual on Handling of Cases of Violation Concerning Feed Safety” (15-Shoan No. 991 as of August 22, 2003).

(2) Imported feed grains
Instructions, etc. to recall the inspection target lots should be given to feed importers, etc. Since it is determined that the handling of formula feed, etc. manufactured using said feed should be individually judged based on the data of the transition to livestock products, etc., consultation with the Animal Products Safety Division should be sought in such cases.

2 Cases other than 1
Manufacturers, importers, etc. of said feed are notified of the results of the analysis.