

# **REGULATION ON SAMPLING AND ANALYSIS METHODS FOR OFFICIAL CONTROL OF FEED**

**Law of Authorization:** 5996

**Official Journal of Publication:** 27.12.2011-28155

## **Objective and scope**

**ARTICLE 1** – (1) The objective of this Regulation is to lay down:

- a) the sampling so as to determine the composition of and additives and undesired materials in feed except for microorganisms during the official control of feed,
- b) the preparation of samples for analysis, and the communication of results,
- c) the analysis methods to be applied to the official control of feed,
- ç) the analysis methods to be applied for the determination of the components of animal origin during the official control of feed,
- d) the calculation of the energy values of the mixed feed intended for poultry,
- e) the analysis methods to be utilized for the control of the illegal presence of the feed additives use of which is prohibited.

## **Legal Basis**

**ARTICLE 2** – (1) This Regulation has been prepared

- a) on the basis of Article 31 of the Law No. 5996 of 11/6/2010 on Veterinary Services, Plant Health, Food and Feed, and
- b) in line with the EC Commission Regulation No.152/2009 of 27/1/2009 on the Methods of Sampling and Analysis for the Official Control of Feed.

## **Definitions**

**ARTICLE 3** – (1) For the purposes of this Regulation, the following definitions shall apply:

- a) Separator: Tools which are utilized to separate the samples into equal parts during the preparation of primary sample, reduced sample and laboratory samples.
- b) Reduced sample: Sample which is obtained from aggregate sample by means of reduction technique and represents the aggregate sample.
- c) Ministry: The Ministry of Food, Agriculture and Livestock.
- ç) Primary sample: Amount which is taken from a point of the lot from which samples are collected.
- d) Hand tools: Shovel, sampling probe and similar tools which are utilized to collect samples manually.
- e) Laboratory sample: Sample which is taken from homogenized aggregate sample or reduced sample.
- f) Mechanical tools: Tools which are utilized to collect samples from moving feed.
- g) Sampling staff: Person authorized by the Ministry to conduct official controls.
- ğ) Sampled lot: Feed which can, by its very nature, represent an example and a unit.

h) Sampling tools and devices: Tools and devices which are made of materials that will not cause contamination in the sampled feed and the Ministry deems appropriate to be used.

i) Sampling probe: Sampling instrument in various sizes which has long grooves or partition and complies with the size of the particle of the feed and lot sampled.

i) Aggregate sample: Sample which is obtained through combining and mixing the primary samples taken from the sampled lot homogeneously.

#### **General sampling provisions**

**ARTICLE 4** – (1) Hand tools, mechanical tools and separators shall be utilized so as to collect samples from solid feed.

(2) Number and amount of samples required to be collected have been given in Annex-1 according to the characteristic and size of the lot to be sampled.

(3) Sampling tools and devices, surfaces where samples are to be prepared and sampling vessels should be clean and dry. Samples shall be collected and prepared in the possible shortest time in order to prevent any changes in the characteristic of feed and probable contamination, and necessary measures shall be taken accordingly.

#### **Collection of primary samples**

**ARTICLE 5** – (1) Primary samples to be collected for the control of products or materials which can homogeneously disperse within feed shall be randomly taken from the sampled lot in approximately equal amounts.

a) With regards to bulk feed, the sampled lot shall be divided into approximately equal parts conjecturally and by rule of thumb, and each part shall be randomly sampled as many as primary samples as specified under Paragraph A.2 in Annex-1. Sampling may be carried out during loading or unloading when required.

b) With regards to packaged feed, necessary packages shall be selected in numbers provided for under Paragraph A.2 in Annex-1, and an amount of each package shall be sampled by means of shovel and similar hand tools. Sampling may be carried out after packages are individually unloaded when required. In the case that the collected sample contains rigid parts, they shall be shredded and mixed up.

c) With regards to liquid or semi-liquid feed which is homogenized or can be homogenized, the number of vessels specified under Paragraph A.2 in Annex-1 shall be taken into consideration. The content shall be homogenized, and samples shall be collected from each vessel in required numbers. Sampling may be carried out while unloading the content.

c) With regards to liquid or semi-liquid feed which cannot be homogenized, the number of vessels specified under Paragraph A.2 in Annex-1 shall be taken into consideration, and sampling shall be carried out at different levels. Sampling may be carried out while unloading the content, but the part on top of the vessel shall be removed. Total volume of the collected sample cannot be less than 10 litres in any case.

d) With regards to feed blocks and mineral licking blocks, blocks or mineral licking blocks shall be selected in numbers specified under Paragraph A.2 in Annex-1, and an amount of each block or mineral licking block shall be sampled.

(2) With regards to primary samples to be collected for the control of products or undesired materials such as aflatoxin, claviceps purpurea, castor oil plant and crotalaria, a legume species which do not homogeneously disperse within feed, the lot to be sampled shall be separated into approximately equal parts conjecturally and by rule of thumb according to the number of samples specified under Paragraph B.3 in Annex-1. In case of more than one part which has been conjecturally divided, primary samples in approximately equal amounts shall be taken from different areas of each part in numbers specified Paragraph B.2 in Annex-1. Total amount of primary samples taken from each part for aggregate samples cannot be more than 4 kilograms. Primary samples taken from different parts cannot be transformed into aggregate sample.

#### **Preparation of aggregate samples**

**ARTICLE 6** – (1) Primary samples taken for the control of the products or materials which can homogeneously disperse within feed shall be combined and mixed in order to form aggregate sample.

(2) Primary samples collected from each part for the control of products or undesired materials such as aflatoxin, claviceps purpurea, castor oil plant and crotalaria, a legume species, which do not homogeneously disperse within feed, shall be combined and mixed in order to form aggregate sample in numbers specified under Paragraph B.3 in Annex-1. Aggregate sample taken from each part which has been determined conjecturally and by rule of thumb shall be coded in accordance with the part from which it has been taken.

#### **Preparation of laboratory samples**

**ARTICLE 7** – (1) An aggregate sample shall be carefully and meticulously mixed in order to obtain a homogeneous sample. Aggregate sample may be reduced to 2 kilograms or 2 litres by means of mechanical or automatic separator or quartering method and therefore the aggregate sample may be obtained if required.

(2) Minimum two samples which comply with the number specified under Paragraph A.4 in Annex-1 or Paragraph B.4 in Annex-1 and bear the same characteristics shall be formed so as to be submitted to laboratory. Each sample shall be separately put into vessels. All necessary measures shall be taken in order to prevent the probable contamination during storage and transportation or the problems that may decay the sample and alter its composition.

#### **Packaging of laboratory samples**

**ARTICLE 8** – (1) Samples collected for official controls shall be sealed and labelled. Sealing shall be carried out in such a way that package cannot be opened without damaging the seal in order to ensure the safety of sample.

#### **Records of samples**

**ARTICLE 9** – (1) The sample and the part to which the sample belongs shall be recorded in such a way as not to cause confusion and hesitation.

#### **Submission of samples to laboratory**

**ARTICLE 10** – (1) Minimum one sample of the samples taken from each aggregate sample and necessary information related to the required analysis shall be submitted to the competent laboratory for analysis in the shortest time possible.

#### **Preparation of samples for analysis**

**ARTICLE 11** – (1) Feed sample which is collected and submitted to the competent authority in accordance with this Regulation shall be weighed and sampled in amounts specified by analysis method. This amount should be of homogeneous nature and represent the sample.

(2) The points to consider during the preparation of samples for analysis are specified below:

- a) Samples shall be prepared in accordance with technique specified in the analysis methods applied.
- b) All of the procedures followed shall be carried out in such a way as to prevent probable contamination and alteration in the composition as far as possible.
- c) Grinding, mixing and sieving operations which are to be carried out so as to ensure that sample is minimally exposed to weather and light shall be conducted as fast as possible. Mills and grinders which may prominently cause the sample to heat up cannot be utilized.
- ç) Mainly feed sensitive to heat shall be manually grinded. The attention shall be paid to the fact that the tools utilized do not constitute a contamination source for the sample in terms of trace metals.
- d) In the case that it is impossible to hold the moisture content of the sample and therefore some changes occur in the moisture content thereof, determination of moisture shall be performed before and after the preparation in accordance with the technique described in Subparagraph (a) of Paragraph 1 of Article 13.

(3) Sample shall be divided into sub-samples in adequate number for analysis and reference by means of appropriate separation techniques while preparing it for analysis. Coning and quartering methods shall not be applied to divide the sample into sub-samples. Reference sample shall be kept in an appropriate, clean, dry and air-tight vessel. Minimum 100-gram sub-samples shall be prepared according to characteristics of feed as specified below:

- a) With regards to feed that can be grinded, it shall be grinded, when required, unless another technique is specified by analysis method; however, excessive grinding shall be avoided during grinding process. All of the grinded sample shall be sieved through a 1 mm<sup>2</sup> mesh sieve (according to ISO R565). The sieved sample shall be mixed and put into an appropriate, clean, dry and air-tight vessel. In the case that sample is to be collected for analysis, the sample shall be re-mixed before weighing process.
- b) With regards to the feed that can be grinded after drying process, sample shall be dried until its moisture content reduces to the range of 12% - 8% in accordance with the pre-drying process described in the method under Sub-paragraph (a) of Paragraph 1 of Article 13 where another technique is not specified by analysis method. Further processes shall be carried out as specified under Sub-paragraph (a).
- c) With regards to liquid or semi-liquid feed, sample shall be put into an appropriate, clean, dry and air-tight vessel. In the case that sample is to be collected for analysis, the sample shall be mixed just before weighing process.
- ç) With regards to other feed which it is not possible to prepare as specified above, other different techniques may be applied to the preparation of them according to their characteristics. However, the amount taken for analysis shall be homogeneous and such as to represent the sample to be analyzed.

(4) With regards to the storage of samples, samples shall be stored at an appropriate temperature which may not cause any alteration in their composition. Mainly vitamins or the samples which are collected so as to analyze the light-sensitive materials shall be kept in a brown glass vessel.

#### **Provisions regarding the tools and chemicals utilized for analyses**

**ARTICLE 12** – (1) The chemicals utilized should be analytically pure unless another form is specified by analysis method. Blind test shall be conducted to control the purity of the chemicals utilized to analyze the materials in trace quantities. It may be required to purify chemicals further according to the results obtained.

(2) Water shall be utilized to prepare a solution, diluents and rinsing and washing solutions unless a special solution, diluents or any other substance is not specified by analysis method. The water to be utilized for such actions shall be distilled or demineralised. In the case that a special purification is required by analysis method, purification shall be performed as specified by analysis method in terms of the water to be utilized.

(3) It is a requirement for the tools and materials utilized at laboratory to be clean and comprise of those specified by analysis methods.

#### **Analysis methods**

**ARTICLE 13** – (1) The Ministry shall determine and publish on the web page of the Ministry the analysis methods related to

- a) the control of the composition of mixed feed and feedstuffs,
- b) the control of the level of feed additives,
- c) the control of the undesired materials in feed,
- ç) the official control to determine the components of animal origin in feed,
- d) the control of the feed additives which are prohibited for use,
- e) the calculation of the energy values of the mixed feed intended for poultry,

(2) Various methods may be applied to the extraction process. In the case that an extraction method other than those specified by analysis method is applied, this method shall provide extraction efficiency equal to the method specified by analysis method for the product in question.

(3) Cleaning process may be performed by means of various methods. In the case that a method other than those specified by analysis method is applied, this method shall provide cleanliness efficiency equal to the method specified by analysis method for the product in question.

(4) Other methods which are different from those described in this Regulation and scientifically accepted and allowed by the Ministry may be applied, where necessary, within the scope of the Regulation on Establishment, Duties, Responsibilities, Procedures and Principles of Food Control Laboratories.

#### **Communication of analysis results**

**ARTICLE 14** - (1) Analysis result shall be written in reasonable figures as specified by analysis method. Result may be updated according to the moisture content prior to preparation where necessary in the case that an alteration occurs in the moisture content of the sample delivered to laboratory.

(2) The method applied shall be included in the relevant report.

(3) The relevant report shall contain the result obtained in the analysis report, the sample studied and number of repetitions. The result value shall be determined by averaging the sample studied and the number of repetitions. Parallel operation shall be performed on minimum two samples. In the case that analysis is conducted for undesired materials, one more study shall not be required if the value which is obtained as a result of the analysis determined to be conducted on the first sample is lower than the value specified in the specification on the condition that quality procedures should be followed. With regards to the control of a material's content or the value declared, one more study shall not be required if the analysis result obtained from the first sample studied confirms the content and the value declared and stands at an acceptable variation range on the condition that quality procedures should be followed.

(4) Expanded measurement uncertainty and recovery correction shall be taken into account during the determination and evaluation of undesired materials such as dioxin and dioxin like polychlorinated biphenyl (PCBs) in feed. In the case that the analysis result of undesired materials is higher than the maximum level determined, the feed in question shall be considered to be inappropriate. This evaluation shall be performed in the cases where it is possible to calculate the measurement uncertainty and recovery correction according to analysis method. Such evaluations shall not be performed since they are not appropriate for microscopic analyses. With regards to the reporting of analysis results, measurement uncertainty and recovery rate shall be determined as specified below:

a) Recovery level shall be determined in the correction applied for recovery. The correction for recovery shall not be required in the cases where it stands at 90% to 110%.

b) With regards to the equation of ' $x \pm U$ ', " $x$ " shall refer to the analysis result and " $U$ " to the expanded measurement uncertainty. Coverage factor 2 shall be used for 95% confidence interval. However, it is not required to include the measurement uncertainty and recovery correction in the analysis result report if the value obtained as a result of the analysis determined is lower than the half of the value specified in the specification on the condition that quality procedures should be followed.

#### **Entry into Force**

**ARTICLE 15** – (1) This Regulation shall enter into force on the date it is published.

#### **Enforcement**

**ARTICLE 16** – (1) The provisions of this Regulation shall be enforced by the Ministry of Food, Agriculture and Livestock.

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