Commission Regulation (EU) No 691/2013 of 19 July 2013 amending Regulation (EC) No 152/2009 as regards methods of sampling and analysis (Text with EEA relevance)

COMMISSION REGULATION (EU) No 691/2013

of 19 July 2013

amending Regulation (EC) No 152/2009 as regards methods of sampling and analysis

(Text with EEA relevance)

THE EUROPEAN COMMISSION,

Having regard to the Treaty on the Functioning of the European Union,

Having regard to Regulation (EC) No 882/2004 of the European Parliament and of the Council of 29 April 2004 on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules[(1)], and in particular Article 11(4) thereof,

Whereas:

(1) Commission Regulation (EC) No 152/2009 of 27 January 2009 laying down the methods of sampling and analysis for the official control of feed[(2)] acknowledged the need to update the provisions concerning sampling to take into account the recent developments in the way feed are produced, stored, transported and marketed.

(2) The sampling for the official control of pesticide residues in and on feed of plant and animal origin has to be performed in accordance with Commission Directive 2002/63/EC of 11 July 2002 establishing Community methods of sampling for the official control of pesticide residues in and on products of plant and animal origin and repealing Directive 79/700/EEC[(3)]. The sampling requirements provided for in Directive 2002/63/EC are minimum requirements and the sampling requirements provided for in this Regulation are in general at least equal to or stricter than these minimum requirements with the exception of the size of the final sample for certain commodities. With the inclusion of the provisions as regards the size of the final sample for the control of pesticide residues, the methods of sampling provided in this Regulation can also be applied for the control of pesticide residues.

(3) Commission Regulation (EU) No 619/2011[(4)] lays down the methods of sampling and analysis for the official control of feed as regards presence of genetically modified material for which an authorisation procedure is pending or the authorisation of which has expired. For sampling, Regulation (EU) No 619/2011 makes reference to the provisions of Regulation (EC) No 152/2009 whereby it establishes specific provisions as regards the size of sample. The amendments introduced by this Regulation include specific provisions as regards the size of samples; therefore the methods of sampling provided for by Regulation (EC) No 152/2009 as amended by this Regulation should also be applied to the control of compliance with Regulation (EU) No 619/2011.

(4) A period of time is needed to put the new method of sampling in place.

(5) Regulation (EC) No 152/2009 should therefore be amended accordingly.

(6) The measures provided for in this Regulation are in accordance with the opinion of the Standing Committee on the Food Chain and Animal Health and neither the European Parliament nor the Council has opposed them,

HAS ADOPTED THIS REGULATION:

Article 1

Regulation (EC) No 152/2009 is amended as follows:

(1) Article 1 is replaced by the following:

Article 1

Sampling for the official control of feed, in particular as regards the determination of constituents, including material which contains or consists of or is produced from genetically modified organisms (GMOs), feed additives as defined by Regulation (EC) No 1831/2003 of the European Parliament and of the Council[(5)], undesirable substances as defined by Directive 2002/32/EC of the European Parliament and of the Council[(6)] shall be carried out in accordance with the methods set out in Annex I.

The method of sampling set out in Annex I is applicable for the control of feed as regards the determination of pesticide residues as defined in Regulation (EC) No 396/2005 of the European Parliament and of the Council[(7)] and control of compliance with Regulation (EU) No 619/2011.;

(2) Annex I is replaced by the text set out in Annex I to this Regulation;

(3) Annex II is replaced by the text set out in Annex II to this Regulation.

Article 2

This Regulation shall enter into force on the twentieth day following that of its publication in the *Official Journal of the European Union*.

It shall apply from 1 January 2014.

This Regulation shall be binding in its entirety and directly applicable in all Member States.

Done at Brussels, 19 July 2013.

For the Commission

The President

José Manuel BARROSO

ANNEX I

‘ANNEX I

METHODS OF SAMPLING

1. PURPOSE AND SCOPE

Samples intended for the official control of feed shall be taken according to the methods described below. Samples thus obtained shall be considered as representative of the sampled portions.

The purpose of representative sampling is to obtain a small fraction from a lot in such a way that a determination of any particular characteristic of this fraction will represent the mean value of the characteristic of the lot. The lot shall be sampled by repeatedly taking incremental samples at various single positions in the lot. These incremental samples shall be combined by mixing to form an aggregate sample from which representative final samples shall be prepared by representative dividing.

If by a visual inspection, portions of the feed to be sampled show a difference in quality from the rest of the feed from the same lot, such portions shall be separated from the rest of the feed and treated as a separate sublot. If it is not possible to divide the feed into separate sublots, the feed shall be sampled as one lot. In such cases, mention shall be made of this fact in the sampling report.

Where a feed sampled in accordance with the provisions of this Regulation is identified as not satisfying EU requirements, is part of a lot of feed of the same class or description, it shall be presumed that all of the feed in that lot is so affected, unless following a detailed assessment there is no evidence that the rest of the lot fails to satisfy the EU requirements.

2. DEFINITIONS

— Lot (or batch)	:	an identified quantity of feed determined to have common characteristics, such as origin, variety, type of packaging, packer, consignor or labelling, and in case of a production process, a unit of production from a single plant using uniform production parameters or a number of such units, when produced in continuous order and stored together.
— Sampled portion	:	A lot or an identified part of the lot or sublot.
— Sealed sample	:	a sample sealed in such a manner as to prevent any access to the sample without breaking or removing the seal.
— Incremental sample	:	A quantity taken from one point in the sampled portion.
— Aggregate sample	:	An aggregate of incremental samples taken from the same sampled portion.
— Reduced sample	:	A part of the aggregate sample, obtained from the latter by a process of representative reduction.
— Final sample	:	A part of the reduced sample or of the homogenised aggregate sample.
— Laboratory sample	:	a sample intended for the laboratory (as received by the laboratory) and can be the final, reduced or aggregate sample.

3. GENERAL PROVISIONS

— Sampling personnel: the samples shall be taken by persons authorised for that purpose by the competent authority.

— The sample has to be sealed in such a manner as to prevent any access to the sample without breaking or removing the seal. The seal's mark should be clearly identifiable and clearly visible. Alternatively, the sample can be put in a recipient which can be closed in such a manner that it cannot be opened without irreversibly damaging the receptacle or container, avoiding the re-use of the receptacle or container.

— Identification of the sample: the sample has to be indelibly marked and must be identified in such a way that there is an unambiguous link to the sampling report.

— From each aggregate sample at least two final samples are taken: at least one for control (enforcement) and one for the feed business operator (defence). Eventually, one final sample may be taken for reference. In case the complete aggregate sample is homogenized, the final samples are taken from the homogenized aggregate sample, unless such procedure conflicts with Member States' rules as regards the right of the feed business operator.

4. APPARATUS

4.1. The sampling apparatus must be made of materials which cannot contaminate the products to be sampled. Apparatus which is intended to be used multiple times must be easy to clean to avoid any cross-contamination.

4.2. **Apparatus recommended for the sampling of solid feed**

4.2.1. *Manual sampling*

4.2.1.1. Flat-bottomed shovel with vertical sides

4.2.1.2. Sampling spear with a long split or compartments. The dimensions of the sampling spear must be appropriate to the characteristics of the sampled portion (depth of container, dimensions of sack, etc.) and to the particle size of the feed.

In case the sampling spear has several apertures, in order to ensure that the sample is taken at the different locations alongside the spear, the apertures should be separated by compartments or sequentially staggered apertures.

4.2.2. *Mechanical sampling*

Appropriate mechanical apparatus may be used for the sampling of moving feed. Appropriate means that at least the whole section of the flow is sampled.

Sampling of feed in motion (at high flow rates) can be performed by automatic samplers.

4.2.3. *Divider*

If possible and appropriate, apparatus designed to divide the sample into approximately equal parts should be used for the preparation of reduced samples in a representative way.

5. QUANTITATIVE REQUIREMENTS AS REGARDS NUMBER OF INCREMENTAL SAMPLES

— The quantitative requirements in points 5.1 and 5.2 as regards the number of incremental samples are applicable for sampled portion sizes up to a maximum of 500 tonnes and which can be sampled in a representative way. The sampling procedure described is equally valid for quantities larger than prescribed maximum sampled portion size provided that the maximum number of incremental samples given in the tables below is ignored, the number of incremental samples being determined by the

square-root formula given in the appropriate part of the procedure (see point 5.3) and the minimum aggregate sample size increased proportionally. This does not prevent a large lot being divided into smaller sublots and each sublot sampled in accordance with the procedure described in points 5.1 and 5.2.

— The size of the sampled portion must be such that each of its constituent parts can be sampled.

— For very large lots or sublots (> 500 tonnes) and for lots which are transported or stored in such a way that sampling cannot be done in accordance with the sampling procedure provided for in points 5.1 and 5.2 of this chapter, the sampling procedure as provided for in point 5.3 is to be applied.

— In case the feed business operator is required by legislation to comply with this Regulation within the frame of a mandatory monitoring system, the feed business operator may deviate from the quantitative requirements as provided for in this chapter to take into account operational characteristics on the condition that the feed business operator has demonstrated to the satisfaction of the competent authority the equivalence of the sampling procedure as regards representativeness and after authorisation from the competent authority.

— In exceptional cases, if it is not possible to carry out the method of sampling set out as regards the quantitative requirements because of the unacceptable commercial damage to the lot (because of packaging forms, means of transport, way of storage etc.) an alternative method of sampling may be applied provided that it is as representative as possible and is fully described and documented.

5.1. **Quantitative requirements as regards incremental samples in relation to the control of substances or products uniformly distributed throughout the feed**

5.1.1. *Loose solid feed*

Size of sampled portion	**Minimum number of incremental samples**
≤ 2,5 tonnes	7
> 2,5 tonnes	√ 20 times the number of tonnes making up the sampled portion[a], up to 40 incremental samples

a Where the number obtained is a fraction, it shall be rounded up to the next whole number.

5.1.2. *Loose liquid feed*

Size of sampled portion	**Minimum number of incremental samples**
≤ 2,5 tonnes or ≤ 2 500 litres	4[a]
> 2,5 tonnes or > 2 500 litres	7[a]

a In case it is not possible to make the liquid homogeneous, the number of incremental samples has to be increased.

5.1.3. *Packaged feed*

Feed (solid and liquid) can be packaged in bags, sacks, cans, barrels etc. which are referred to in the table as units. Large units (≥ 500 kg or litres) have to be sampled in accordance with the provisions foreseen for loose feed (see points 5.1.1 and 5.1.2).

Size of sampled portion	**Minimum number of units from which (at least) one incremental sample has to be taken**[a]
1 to 20 units	1 unit[b]
21 to 150 units	3 units[b]
151 to 400 units	5 units[b]
> 400 units	¼ of the √ number of units making up the sampled portion[c], up to 40 units

a In the case where opening of an unit might affect the analysis (e.g. perishable wet feeds) an incremental sample shall be the unopened unit.

b For units whose contents do not exceed 1 kg or one litre, an incremental sample shall be the contents of one original unit.

c Where the number obtained is a fraction, it shall be rounded up to the next whole number.

5.1.4. *Feed blocks and mineral licks*

Minimum one block or lick to be sampled per sampled portion of 25 units, up to a maximum of four blocks or licks.

For blocks or licks weighing not more than 1 kg each, an incremental sample shall be the contents of one block or one lick.

5.1.5. *Roughages/forage*

Size of sampled portion	**Minimum number of incremental samples**[a]
≤ 5 tonnes	5
> 5 tonnes	√ 5 times the number of tonnes making up the sampled portion[b], up to 40 incremental samples

a It is acknowledged that in certain situations (e.g. silages) it is not possible to take the required incremental samples, without causing unacceptable damage to the lot. An alternative method of sampling may be applied in such situations and a guidance for sampling such lots will be elaborated before the entry into application of this Regulation.

b Where the number obtained is a fraction, it shall be rounded up to the next whole number.

5.2. **Quantitative requirements as regards incremental samples in relation to the control of constituents or substances likely to be distributed non-uniformly in feed**

These quantitative requirements as regards incremental samples are to be used in the following situations:

— control of aflatoxins, rye ergot, other mycotoxins and harmful botanical impurities in feed materials;

— control of cross contamination by a constituent, including GM material, or substance for which non-uniform distribution is expected in feed materials.

In case the control authority has strong suspicion that such a non-uniform distribution occurs also in case of cross contamination by a constituent or substance in a compound feed, the quantitative requirements as provided for in the table below can be applied.

Size of sampled portion	**Minimum number of incremental samples**
< 80 tonnes	See quantitative requirements under point 5.1. The number of incremental samples to be taken has to be multiplied by 2,5.
≥ 80 tonnes	100

5.3. **Quantitative requirements as regards the incremental samples in the case of very large lots**

In the case of large sampled portions (sampled portions > 500 tonnes), the number of incremental samples to be taken = 40 incremental samples + √ tonnes in relation to the control of substances or products uniformly distributed throughout the feed or 100 incremental samples + √ tonnes in relation to the control of constituents or substances likely to be distributed non-uniformly in feed materials.

6. QUANTITATIVE REQUIREMENTS AS REGARDS AGGREGATE SAMPLE

A single aggregate sample per sampled portion is required.		
	Nature of feed	**Minimum size of aggregate sample[ab]**
6.1.	Loose feed	4 kg
6.2.	Packaged feed:	4 kg[c]
6.3.	Liquid or semi-liquid feed:	4 litres
6.4.	Feed blocks or mineral licks:	
6.4.1.	each weighing more than 1 kg	4 kg
6.4.2.	each weighing not more than 1 kg	weight of four original blocks or licks
6.5.	Roughage/forage	4 kg[d]

a In case the sampled feed is of high value, a smaller quantity of aggregate sample can be taken on the condition this is described and documented in the sampling report.

b In accordance with the provisions of Commission Regulation (EU) No 619/2011 of 24 June 2011 laying down the methods of sampling and analysis for the official control of feed as regards presence of genetically modified material for which an authorisation procedure is pending or the authorisation of which has expired (OJ L 166, 25.6.2011, p. 9), the aggregate sample for the control of the presence of genetically modified material must contain at least 35 000 seeds/grains. This means that for maize the size of the aggregate sample must be at least 10,5 kg and for soybean 7 kg. For other seeds and grains such as barley, millet, oat, rice, rye, wheat and rapeseed, the aggregate sample size of 4 kg corresponds to more than 35 000 seeds.

c In case of packaged feed, it may also not be possible to achieve the size of 4 kg for the aggregate sample depending of the size of the individual units.

d	In case it concerns roughage or forage with a low specific gravity (e.g. hay, straw), the aggregate sample should have a minimum size of 1 kg.

7. QUANTITATIVE REQUIREMENTS AS REGARDS FINAL SAMPLES

Final samples

Analysis of at least one final sample is required. The amount in the final sample for analysis shall be not less than the following:

Solid feed	500 g[abc]
Liquid or semi-liquid feed	500 ml[a]

a	In accordance with the provisions of Regulation (EU) No 619/2011, the final sample for the control of the presence of genetically modified material must contain at least 10 000 seeds/grains. This means that for maize the size of the final sample must be at least 3 000 g and for soybean 2 000 g. For other seeds and grains such as barley, millet, oat, rice, rye, wheat and rapeseed, the final sample size of 500 g corresponds to more for 10 000.
b	In case the size of the aggregate sample is significantly less than 4 kg or litre (see footnotes point (6), also a smaller quantity of final sample can be taken on the condition this is described and documented in the sampling report.
c	In case of sampling pulses, cereal grains and tree nuts for the determination of pesticide residues, the minimum size of the final sample shall be 1 kg in accordance with the provisions of Commission Directive 2002/63/EC (OJ L 187, 16.7.2002, p. 30).

8. METHOD OF SAMPLING FOR VERY LARGE LOTS OR LOTS STORED OR TRANSPORTED IN A WAY WHEREBY SAMPLING THROUGHOUT THE LOT IS NOT FEASIBLE

8.1. **General principles**

In case the way of transport or storage of a lot does not enable to take incremental samples throughout the whole lot, sampling of such lots should preferably be done when the lot is in flow.

In the case of large warehouses destined to store feed, operators should be encouraged to install equipment in the warehouse enabling (automatic) sampling across the whole stored lot.

In case of applying the sampling procedures as provided for in this chapter 8, the feed business operator or his representative is informed of the sampling procedure. In case this sampling procedure is questioned by the feed business operator or his representative, the feed business operator or his representative shall enable the competent authority to sample throughout the whole lot at his/her cost.

8.2. **Large lots transported by ship**

8.2.1. *Dynamic sampling of large lots transported by ship*

The sampling of large lots in ships is preferably carried out while the product is in flow (dynamic sampling).

The sampling is to be done per hold (entity that can physically be separated). Holds are however emptied partly one after the other so that the initial physical separation does no longer exist after transfer into storage facilities. Sampling can therefore be performed in function of the initial physical separation or in function of the separation after transfer into the storage facilities.

The unloading of a ship can last for several days. Normally, sampling has to be performed at regular intervals during the whole duration of unloading. It is however not always feasible or appropriate for an official inspector to be present for sampling during the whole operation of

unloading. Therefore sampling is allowed to be undertaken of part (sampled portion) of the whole lot. The number of incremental samples is determined by taking into account the size of the sampled portion.

In the case of sampling a part of a lot of feed of the same class or description and that part of the lot has been identified as not satisfying EU requirements, it shall be presumed that all of the feed in that lot is so affected, unless following a detailed assessment there is no evidence that the rest of the lot fails to satisfy the EU requirements.

Even if the official sample is taken automatically, the presence of an inspector is necessary. However in case the automatic sampling is done with preset parameters which cannot be changed during the sampling and the incremental samples are collected in a sealed receptacle, preventing any possible fraud, then the presence of an inspector is only required at the beginning of the sampling, every time the receptacle of the samples needs to be changed and at the end of the sampling.

8.2.2. *Sampling of lots transported by ship by static sampling*

In case the sampling is done in a static way the same procedure as foreseen for storage facilities (silos) accessible from above has to be applied (see point 8.4.1).

The sampling has to be performed on the accessible part (from above) of the lot/hold. The number of incremental samples is determined by taking into account the size of the sampled portion. In the case of sampling a part of a lot of feed of the same class or description and that part of the lot has been identified as not satisfying EU requirements, it shall be presumed that all of the feed in that lot is so affected, unless following a detailed assessment there is no evidence that the rest of the lot fails to satisfy the EU requirements.

8.3. **Sampling of large lots stored in warehouses**

The sampling has to be performed on the accessible part of the lot. The number of incremental samples is determined by taking into account the size of the sampled portion. In the case of sampling a part of a lot of feed of the same class or description and that part of the lot has been identified as not satisfying EU requirements, it shall be presumed that all of the feed in that lot is so affected, unless following a detailed assessment there is no evidence that the rest of the lot fails to satisfy the EU requirements.

8.4. **Sampling of storage facilities (silos)**

8.4.1. *Sampling of silos (easily) accessible from above*

The sampling has to be performed on the accessible part of the lot. The number of incremental samples is determined by taking into account the size of the sampled portion. In the case of sampling a part of a lot of feed of the same class or description and that part of the lot has been identified as not satisfying EU requirements, it shall be presumed that all of the feed in that lot is so affected, unless following a detailed assessment there is no evidence that the rest of the lot fails to satisfy the EU requirements.

8.4.2. *Sampling of silos not accessible from above (closed silos)*

8.4.2.1. *Silos not accessible from above (closed silos) with size > 100 tonnes*

Feed stored in such silos cannot be sampled in a static way. Therefore in case the feed in the silo has to be sampled and there is no possibility to move the consignment, the agreement has to be made with the operator that he or she has to inform the inspector about when the silo will be unloaded in order to enable sampling when the feed is in flow.

8.4.2.2. *Silos not accessible from above (closed silos) with size < 100 tonnes*

Sampling procedure involves the release into a receptacle of a quantity of 50 to 100 kg and taking the sample from it. The size of the aggregate sample corresponds to the whole lot and the number of incremental samples relate to the quantity of the silo released in a receptacle for sampling. In the case of sampling a part of a lot of feed of the same class or description and that part of the lot has been identified as not satisfying EU requirements, it shall be presumed that all of the feed in that lot is so affected, unless following a detailed assessment there is no evidence that the rest of the lot fails to satisfy the EU requirements.

8.5. **Sampling of loose feed in large closed containers**

Such lots can often only be sampled when unloaded. It is in certain cases not possible to unload at the point of import or control and therefore the sampling should take place when such containers are unloaded.

9. INSTRUCTIONS FOR TAKING, PREPARING AND PACKAGING THE SAMPLES

9.1. **General**

The samples must be taken and prepared without unnecessary delay bearing in mind the precautions necessary to ensure that the product is neither changed nor contaminated. Instruments and also surfaces and containers intended to receive samples must be clean and dry.

9.2. **Incremental samples**

Incremental samples must be taken at random throughout the whole sampled portion and they must be of approximately equal sizes.

The incremental sample size is at least 100 grams or 25 grams in case of roughage or forage with low specific gravity.

In case that in accordance with the rules for the sampling procedure established in point 8 less than 40 incremental samples have to be taken, the size of the incremental samples shall be determined in function of the required size of the aggregate sample to be achieved (see point (6).

In case of sampling of small lots of packaged feed where according to the quantitative requirements a limited number of incremental samples have to be taken, an incremental sample shall be the contents of one original unit whose contents do not exceed 1 kg or one litre.

In case of sampling of packaged feed composed of small units (e.g. < 250 g), the size of the incremental sample depends on the size of the unit.

9.2.1. *Loose feed*

Where appropriate, sampling may be carried out when the sampled portion is being moved (loading or unloading).

9.2.2. *Packaged feed*

Having selected the required number of units for sampling as indicated in chapter 5, part of the contents of each unit shall be removed using a spear or shovel. Where necessary, the samples shall be taken after emptying the units separately.

9.2.3. *Homogeneous or homogenisable liquid or semi-liquid feed*

Having selected the required number of units for sampling as indicated in chapter 5, the contents shall be homogenised if necessary and an amount taken from each unit.

The incremental samples may be taken when the contents are being discharged.

9.2.4. *Non-homogenisable, liquid or semi-liquid feed*

Having selected the required number of units for sampling as indicated in chapter 5, samples shall be taken from different levels.

Samples may also be taken when the contents are being discharged but the first fractions shall be discarded.

In either case the total volume taken must not be less than 10 litres.

9.2.5. *Feed blocks and mineral licks*

Having selected the required number of blocks or licks for sampling as indicated in chapter 5, a part of each block or lick can be taken. In case of suspicion of a non-homogeneous block or lick, the whole block or lick can be taken as sample.

For blocks or licks weighing not more than 1 kg each, an incremental sample shall be the contents of one block or one lick.

9.3. **Preparation of aggregate samples**

The incremental samples shall be mixed to form a single aggregate sample.

9.4. **Preparation of final samples**

The material in the aggregate sample shall be carefully mixed[(8)].

— Each sample shall be put into an appropriate container/receptacle. All necessary precautions shall be taken to avoid any change of composition of the sample, contamination or adulteration which might arise during transportation or storage.

— In case of the control of constituents or substances uniformly distributed throughout the feed, the aggregate sample can be representatively reduced to at least 2,0 kg or 2,0 litres (reduced sample)[(9)] preferably either by using a mechanical or automatic divider. For the control of the presence of pesticide residues in pulses, cereal grains and tree nuts, the minimum size of the reduced sample shall be 3 kg. In case the nature of the feed does not allow using a divider or the divider is not available, then the sample can be reduced by the quartering method. From the reduced samples the final samples (for control, defence and reference) shall then be prepared of approximately the same amount and conforming to the quantitative requirements of chapter 7. In case of the control of constituents, including genetically modified material, or substances likely to be distributed non-uniformly in feed materials, the aggregate sample shall be:

  — completely homogenized and divided afterwards into final samples or

  — reduced to at least 2 kg or 2 litres[(10)] by using a mechanical or automatic divider. Only in the case that the nature of the feed does not allow for using a divider, the sample can, if necessary, be reduced by quartering method. For the control of the presence of genetically modified material in the frame of Regulation (EU) No 619/2011, the reduced sample must contain at least 35 000 seeds/grains to enable to obtain the final samples for enforcement, defence and reference of at least 10 000 seeds grain (see footnote (**) in chapter 6 and footnote (*) in chapter 7).

9.5. **Packaging of samples**

The containers or packages shall be sealed and labelled in such a manner that they cannot be opened without damaging the seal. The total label must be incorporated in the seal.

9.6. **Sending of samples to the laboratory**

The sample shall be sent without unnecessary delay to the designated analytical laboratory, together with the information necessary for the analyst.

10. SAMPLING RECORD

A record must be kept of each sample, permitting each sampled portion and its size to be identified unambiguously.

The record shall also mention any deviation of the sampling procedure as provided for in this Regulation.

Besides making the record available to the official control laboratory, the record shall be made available to the feed business operator and/or the laboratory designated by the feed business operator.

ANNEX II

ANNEX II

GENERAL PROVISIONS ON METHODS OF ANALYSIS FOR FEED

A. PREPARATION OF SAMPLES FOR ANALYSIS

1. **Purpose**

The procedures described below concern the preparation for analysis of samples, sent to the control laboratories after sampling in accordance with the provisions laid down in Annex I.

The laboratory samples must be prepared in such a way that the amounts weighed out, as provided for in the methods of analysis, are homogeneous and representative of the final samples.

2. **Precautions to be taken**

The sample preparation procedure to be followed is dependent on the methods of analysis to be used and the constituents or substances to be controlled. It is therefore of major importance that it is ensured that the followed sample preparation procedure is appropriate for the used method of analysis and for constituents or substances to be controlled.

All the necessary operations must be performed in such a way as to avoid as far as possible contamination of the sample and changes of its composition.

Grinding, mixing and sieving shall be carried out without delay with minimal exposure of the sample to the air and light. Mills and grinders likely to appreciably heat the sample shall not be used.

Manual grinding is recommended for feed which are particularly sensitive to heat. Care shall also be taken to ensure that the apparatus itself is not a source of contamination.

If the preparation cannot be carried out without significant changes in the moisture content of the sample, determine the moisture content before and after preparation according to the method laid down in Part A of Annex III.

3. **Procedure**

3.1. *General procedure*

The test aliquot is taken from the final sample. Coning and quartering is not recommended because this might provide test aliquots with high splitting error.

3.1.1. *Feed which can be ground as such*

— Mix the sieved final sample and collect it in a suitable clean, dry container fitted with an air-tight stopper. Mix again in order to ensure full homogenisation, immediately before weighing out the amount for analysis (test aliquot).

3.1.2. *Feed which can be ground after drying*

— Unless otherwise specified in the methods of analysis, dry the final sample to bring its moisture content down to a level of 8 to 12 %, according to the preliminary drying procedure described under point 4.3 of the method of determination of moisture mentioned in Part A of Annex III). Then proceed as indicated in section 3.1.1.

3.1.3. *Liquid or semi-liquid feed*

— Collect the final sample in a suitable clean, dry container, fitted with an air-tight stopper. Mix thoroughly in order to ensure full homogenisation immediately before weighing out the amount for analysis (test aliquot).

3.1.4. *Other feed*

— Final samples which cannot be prepared according to one of the above procedures shall be treated by any other procedure which ensures that the amounts weighed out for the analysis (test aliquot) are homogeneous and representative of the final samples.

3.2. *Specific procedure in case of examination by visual inspection or by microscopy or in cases where the whole aggregate sample is homogenised*

— In case of an examination by visual inspection (without making use of microscope), the whole laboratory sample is used for examination.

— In case of a microscopic examination, the laboratory may reduce the aggregate sample, or further reduce the reduced sample. The final samples for defence and eventually reference purposes are taken following a procedure equivalent to the procedure followed for the final sample for enforcement.

— In case the whole aggregate sample is homogenized, the final samples are taken from the homogenized aggregate sample.

4. **Storage of samples**

Samples must be stored at a temperature that will not alter their composition. Samples intended for the analysis of vitamins or substances which are particularly sensitive to light shall be stored in such conditions that the sample is not adversely affected by light.

B. PROVISIONS RELATING TO REAGENTS AND APPARATUS USED IN METHODS OF ANALYSIS

1. Unless otherwise specified in the methods of analysis, all analytical reagents must be analytically pure (a.p.). When trace analysis is carried out, the purity of the reagents must be checked by a blank test. Depending upon the results obtained, further purification of the reagents may be required.

2. Any operation involving preparation of solutions, dilution, rinsing or washing, mentioned in the methods of analysis without indication as to the nature of the solvent or diluent employed, implies that water must be used. As a general rule, water shall be demineralised or distilled. In particular cases, which are indicated in the methods of analysis, it must be submitted to special procedures of purification.

3. In view of the equipment normally found in control laboratories, only those instruments and apparatus which are special or require specific usage are referred to in the methods of analysis. They must be clean, especially when very small amounts of substances have to be determined.

C. APPLICATION OF METHODS OF ANALYSIS AND EXPRESSION OF THE RESULTS

1. **Extraction procedure**

Several methods determine a specific extraction procedure. As a general rule, other extraction procedures than the procedure referred to in the method can be applied on the condition that the used extraction procedure has been proven to have the equivalent extraction efficiency for the matrix analysed as the procedure mentioned in the method.

2. **Clean-up procedure**

Several methods determine a specific clean-up procedure. As a general rule, other clean-up procedures than the procedure referred to in the method can be applied on the condition that the used clean-up procedure has been proven to result in equivalent analytical results for the matrix analysed as the procedure mentioned in the method.

3. **Number of determinations**

In case of the analysis of undesirable substances, if the result of the first determination is significantly (> 50 %) lower than the specification to be controlled, no additional determinations are necessary, on the condition that the appropriate quality procedures are applied. In other cases a duplicate analysis (second determination) is necessary to exclude the possibility of internal cross-contamination or an accidental mix-up of samples. The mean of the two determinations, taking into account the measurement uncertainty is used for verification of compliance.

In case of the control of the declared content of a substance or ingredient, if the result of the first determination confirms the declared content, i.e. the analytical result falls within the acceptable range of variation of the declared content, no additional determinations are necessary, on the condition that the appropriate quality procedures are applied. In other cases a duplicate analysis (second determination) is necessary to exclude the possibility of internal cross-contamination or an accidental mix-up of samples. The mean of the two determinations, taking into account the measurement uncertainty is used for verification of compliance.

In some cases this acceptable range of variation is defined in legislation such as in Regulation (EC) No 767/2009 of the European Parliament and of the Council of 13 July 2009 on the placing on the market and use of feed, amending European Parliament and Council Regulation (EC) No 1831/2003 and repealing Council Directive 79/373/EEC, Commission Directive 80/511/EEC, Council Directives 82/471/EEC, 83/228/EEC, 93/74/EEC, 93/113/EC and 96/25/EC and Commission Decision 2004/217/EC[(11)].

4. **Reporting of the method of analysis used**

The analysis report shall mention the method of analysis used.

5. Reporting of the analytical result

The analytical result shall be expressed in the manner laid down in the method of analysis to an appropriate number of significant figures and shall be corrected, if necessary, to the moisture content of the final sample prior to preparation.

6. Measurement uncertainty and recovery rate in case of analysis of undesirable substances

As regards undesirable substances within the meaning of Directive 2002/32/EC, a product intended for animal feed shall be considered as non-compliant with the established maximum content, if the analytical result, relative to a feed with a moisture content of 12 %, is deemed to exceed the maximum content taking into account expanded measurement uncertainty and correction for recovery. In order to assess compliance, the analysed concentration is used after being corrected for recovery and after deduction of the expanded measurement uncertainty. This procedure is only applicable in cases where the method of analysis enables the estimation of measurement uncertainty and correction for recovery (e.g. not possible in case of microscopic analysis).

The analytical result shall be reported as follows (in so far the used method of analysis enables to estimate the measurement uncertainty and recovery rate):

(a) corrected for recovery, the level of recovery being indicated. The correction for recovery is not necessary in case the recovery rate is between 90-110 %.

(b) as ‘x +/– U’, whereby x is the analytical result and U is the expanded measurement uncertainty, using a coverage factor of 2 which gives a level of confidence of approximately 95 %.

However, if the result of the analysis is significantly (> 50 %) lower than the specification to be controlled, and on the condition that the appropriate quality procedures are applied and the analysis serves only the purpose of checking compliance with legal provisions, the analytical result might be reported without correction for recovery and the reporting of the recovery rate and measurement uncertainty might be omitted in these cases.

(**1**) OJ L 165, 30.4.2004, p. 1.

(**2**) OJ L 54, 26.2.2009, p. 1.

(**3**) OJ L 187, 16.7.2002, p. 30.

(**4**) OJ L 166, 25.6.2011, p. 9.

(**5**) OJ L 268, 18.10.2003, p. 29.

(**6**) OJ L 140, 30.5.2002, p. 10.

(**7**) OJ L 70, 16.3.2005, p. 1.';

(**8**) Any lumps shall be broken up (if necessary by separating them out and returning them to the sample).

(**9**) Except in the case of roughage or forage with low specific gravity.

(**10**) Except in the case of roughage or forage with low specific gravity.'

(**11**) OJ L 229, 1.9.2009, p. 1.

Status:
Point in time view as at 31/12/2020.

Changes to legislation:
There are currently no known outstanding effects for the Commission Regulation (EU) No 691/2013.