Guidance document for the sampling of cereals for mycotoxins

IMPORTANT NOTE

This document is an evolving document which will be updated to take account of the experience gained with the application of this document or of new information provided.

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I. Scope

To elaborate the document providing guidance for sampling lots where the sampling provisions as provided for in Regulation (EC) 401/2006 of 23 February 2006 laying down methods of sampling and analysis for the official control of the levels of mycotoxins in foodstuffs are practically impossible to apply, such is the case where large lots of cereals are stored in warehouses or where cereals are stored in silos (see Annex I – B.3, last indent – footnote 1)

This means that that the current guidelines are not applicable to

- → situations in which sampling provisions provided for in Regulation (EC) 2006/401 can be applied
- → sampling of lots/batches in lorries, trucks, train wagons, (of reasonable size not exceeding 500 tonnes)

However, the provisions on the sampling equipment (in particular the number of incremental samples taken per sampling point) are also applicable to the situations covered by the provisions of Commission Regulation (EC) 401/2006.

Situations to be covered

- ships (for typical sizes see further)
 - * dynamic
 - * static
- lots in warehouses
 - * dynamic
 - * static
- silo's (in particular cylindrical silo's) (large small)
 - * dynamic
 - * static
- bulk consignments in closed containers

This guidance document can also be used for the control of mycotoxins of other commodities, such as oilseeds and feed, in situations similar to those covered by this guidance document..

II. Sampling of large batches /lots - silo's

II.1 Starting points

*one sampling regime for the control of all mycotoxins

II.2. Number of incremental samples to be taken (for lots > 500 tonnes)

100 incremental samples $+\sqrt{\text{metric}}$ tonnes, this means for a consignment of 10 000 tonnes: 200 incremental samples, 20 kg sample

II.3. Sampling equipment

There are many different types of sampling equipment or devices. The most suitable equipment should be chosen taken into account the product to be sample, the quantity required and the containers to be used.

Examples of equipment (non-exhaustive) to sample flowing grain and static lots are provided for in Annex to this document. The source of information for the examples of sampling equipment is EN-ISO 24333-2009.

II.4. Sampling equipment and incremental samples

When sampling with a sampling probe with several apertures/openings, the cereals collected by a single aperture/opening can be considered as one incremental sample on the condition that in each aperture/opening the minimum quantity of incremental sample size is collected (100 g in the case of cereals, 200 g in the case of oilseeds). Also a maximum of 1 incremental sample per 0.5 m length of sampling probe can be accepted

These provisions on the sampling equipment are also applicable to the situations covered by the provisions of Commission Regulation (EC) 401/2006.

Examples:

Sampling probe of 2 m with 4 apertures/openings, collected quantity 100-150 g per aperture/opening = 4 incremental samples

Sampling probe of 6 m with 4 apertures/openings, collected quantity 250 g per aperture/opening = 4 incremental samples

Sampling probe of 3 m with 8 apertures/openings, collected quantity 100-150 g per aperture/opening = 6 incremental samples

Sampling probe of 2 m with 1 aperture/opening, collected quantity 200 g = 1 incremental sample.

Vacuum sampling probe of 9 meters length, sample taken over the complete length: 18 incremental samples

^{*}one sampling regime for the control of all cereals

II.5 Sizes of bulk shipments and large batches in storage

Bulk shipments are transported in vessels – the size of the vessels can vary from 500 t to cape size vessels of more than 90.000 t. The holds also vary as depending on the constitution of the vessel itself.

Some examples of transport by ship:

- Panamax for about 60.000 metric tonnes in 7 up to 9 holds
- Handy-Max for about 35/45.000 metric tonnes in 5 up to 7 holds
- Handy for about 15/25.000 metric tonnes in 3 to 5 holds
- Coaster for about 2/5.000 metric tonnes in 2 to 3 holds
- River barges for 500 up to about 2.500 metric tonnes in 1 to 2 holds.

Consequently one hold can be from 2 up to 10/12 meters deep.

Rice is generally imported in 20 ton containers: length 6,10 m, height 2,60 m, width 2,20 m (max 24t)

Storage:

Storage facilities (warehouses and silos) vary also, as they depend from the material they are destined to – there are differences of storage facilities between grains and flour because it depends from the flowing of the material stored.

- Flat-(horizontal) warehouses have a size of 15/20 meters x 40 or 60 or 80 meters length or even more and cereals are stored 4 meters (but very often more) high.
- Vertical silos (metallic or concrete) can have storage capacity between 500 to 20.000 tonnes per cell with 4 to 50 cells in the same building and the height can go from 10 to 50 meters.

Rice

The size of the silo destined to rice can range hugely depending on the different companies i.e. from 20 tonnes to 1000 tonnes.

II.6. General principles when sampling large batches

In case the way of transport or storage of a batch does not enable to take incremental samples across the whole batch, sampling of such batches should preferably be done when the product/batch is in flow or in case it is feasible, the lot to be sampled should be moved to another silo, ... to enable sampling across the batch.

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

II.7. Sampling of batches transported by ship

II.7,1, Sampling of batches transported by ship by dynamic sampling

The sampling of large batches in ships is carried out while the cereals are preferably done in flow (dynamic sampling).

The sampling has to be done per hold (entity that can physically be separated). However to keep the balance of the ship, holds are emptied partly one after the other so that the initial physical separation does no longer exist after transfer into silo's. So sampling can be performed in function of the initial physical separation or in function of the separation after transfer into storage.

The unloading of a ship of cereals can take several days: 50.000 tonnes with an unload capacity of 100 to 750 tonnes an hour can take 65 hours (3 days) to 500 hours (20 days). Even if the sample is taken automatically, the presence of an inspector is necessary. Therefore it is not (always) feasible or appropriate (from resources and cost point of view) for an inspector to have to be present during the whole operation of unloading. Therefore sampling is allowed to be undertaken of part of the portion to be sampled and the result is considered representative for the whole portion to be sampled, on the condition that the quantity of the sampled part is at least 10 % of the portion to be sampled. If the operator questions the representativity of the sampling then the operator must enable at his cost to sample the whole batch.

Example

Portion to be sampled: batch of 10.000 tonnes

Unloading speed is 500 tonnes an hour: total unloading time = 20 hours

Inspector decides to sample only part of the batch (at least 10 %). He decides to sample 1000 tonnes which means that the sampling time is 2 hours.

Possibility 1

The number of incremental samples is determined taking into account the size of the whole batch to be sampled.

The sample must consist of 200 (100 + $\sqrt{10000}$ = 200) incremental samples of 100 grams, resulting in a bulk sample of 20 kg.

This means that an incremental sample must be taken every 36 sec. or every 5 tonnes

Possibility 2

The number of incremental samples is determined taking into account the size of the sampled part of the whole batch. The weight of the aggregate sample has to be determined in function of the whole batch to be sampled.

The sample must consist of 132 (100 + $\sqrt{1000}$ = 132) incremental samples of 150 grams, resulting in a bulk sample of 20 kg.

This means that an incremental sample must be taken every 54 sec. or every 7.5 tonnes

II.7.2. Sampling of batches transported by ship by static sampling

In case the sampling is done in a static way the same procedure as foreseen for silo's accessible from above has to be applied (see point II.9.1) this means:

The minimum length of the sampling probe to be used for the sampling o ships in a static way is 2 meter

There has to be a representative sampling of the accessible part (from above) of the consignment/hold performed and the result of such sampling is considered to be valid for the whole batch in the ship/hold

The analytical result of this sample is decisive to determine the compliance/non compliance of the whole batch/hold

In case of non-compliance, and in case representativity of such sampling is questioned by the operator \rightarrow operator has the possibility to request to the authorities <u>on his costs</u> an <u>official</u> representative sampling of the whole consignment in the ship \rightarrow involving a movement of the whole lot from the ship into a storage facility.

For examples see point II.8.

II.8. Sampling of large batches stored in warehouses

See point II.6

The minimum length of the sampling probe to be used for the sampling of static batch/consignment stored in warehouse is 2 meter

There has to be a representative sampling of the accessible part of the consignment performed and the result of such sampling is considered to be valid for the whole batch

The analytical result of this sample is decisive to determine the compliance/non compliance of the whole batch

In case of non-compliance, and in case representativity of such sampling is questioned by the operator \rightarrow operator has the possibility to request to the authorities on his costs an official representative sampling of the whole consignment stored in the warehouse \rightarrow involving a movement of the whole static lot.

Example 1:

Cereals stored at warehouse 30 m large -50 m deep -4 m high =6000 m³ = about 4500 tonnes accessible from 1 side (30 meter side)

Possibility to sample with sampling probe of 2m: $30 \text{ m} \times 2 \text{ m} \times 4 \text{m} = 240 \text{ m}^3 = \text{about } 180 \text{ tonnes}$

 $100 + \sqrt{4500} = 167$ incremental samples of 100 grams – resulting in 16.7 kg sample – considered to be representative for the whole 4500 tonnes batch (if the sampling probe has 4 apertures/openings: the batch has to be sampled at 42 sampling points representatively located along the accessible side)

Possibility to sample with sampling probe (with engine) of 5 meter long: $30 \text{ m} \times 5 \text{ m} \times 4 \text{ m} = 600 \text{ m}^3 = \text{about } 450 \text{ tonnes}$

 $100 + \sqrt{4500} = 167$ incremental samples of 100 grams – resulting in 16.7 kg sample – considered to be representative for the whole 4500 tonnes batch (if the sampling probe has 10 apertures/openings: the batch has to be sampled at 17 sampling points representatively located along the accessible side)

Example 2:

Cereals stored at warehouse 30 m large -50 m deep -4 m high =6000 m³ = about 4500 tonnes accessible from 4 sides (160 meter side)

Possibility to sample with sampling probe of 2m: $160 \text{ m} \times 2 \text{ m} \times 4 \text{m} = 960 \text{ m}^3 = \text{about } 720 \text{ topnes}$

 $100 + \sqrt{4500} = 167$ incremental samples of 100 grams – resulting in 16.7 kg sample – considered to be representative for the whole 4500 tonnes batch (if the sampling probe has 4 apertures/openings: the batch has to be sampled at 42 sampling points, representatively located along the 4 sides)

Possibility to sample with sampling probe (with engine) of 5 meter long: $160 \text{ m} \times 5 \text{ m} \times 4 \text{ m} = 3200 \text{ m}^3 = \text{about } 2400 \text{ tonnes}$

 $100 + \sqrt{4500} = 167$ incremental samples of 100 grams – resulting in 16.7 kg sample – considered to be representative for the whole 4500 tonnes batch (if the sampling probe has 10 apertures/openings: the batch has to be sampled at 17 sampling points, representatively located along the 4 sides)

II. 9. Sampling of silo's

II. 9.1. Sampling of silo's (easily) accessible from above

See point II.6

The **minimum length** of the sampling probe to be used for the sampling of static batch/consignment stored in silo is 2 meter

There has to be a representative sampling of the accessible part of the consignment performed and the result of such sampling is considered to be valid for the whole batch

The analytical result of this sample is decisive to determine the compliance/non compliance of the whole batch

In case of non-compliance, and in case representativity of such sampling is questioned by the operator \rightarrow operator has the possibility to request to the authorities on his costs an official representative sampling of the whole consignment stored in silo \rightarrow involving a movement of the whole lot from one silo into another silo.

For examples see point II.8.

II. 9.2. Sampling of silo's not accessible from above (closed cylindrical silo's)

See point II.6

II. 9.2.1. Sampling of silo's not accessible from above (closed cylindrical silo's) with size >>100 tonnes

Cereals are stored in such silo's cannot be sampled in a static way and therefore in case the cereals 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 has to inform the inspector when the silo will be unloaded in order to enable sampling when cereals are in flow.

II. 9.2.2. Sampling of silo's not accessible from above (closed cylindrical silo's) with reasonable size

Sampling procedure involves the release into a recipient of a quantity of 50 to 100 kg and to take the sample in a representative way from this 50-100 kg

Example for a silo of 25 tonnes:

Silo of 25 tonnes / 50 - 100 kg to be released in recipient / sample taken from this 50 - 100 kg // size of aggregate sample relates to whole consignment (25 tonnes) \rightarrow means 10 kg sample – number of incremental samples relate to quantity released: means 5 incremental samples \rightarrow 5 incremental samples of 2 kg.

The analytical result of this sample is decisive to determine the compliance/non compliance of the whole batch

In case of non-compliance, and in case representativity of such sampling is questioned by the operator \rightarrow operator has the possibility to request to the authorities on his costs an official representative sampling of the whole consignment stored in silo \rightarrow involving a movement of the whole lot from one silo into another silo.

II.10 Sampling of bulk consignments in closed containers

Can only be sampled when unloaded. Is in many cases not possible at point of import and therefore in case such containers are to be sampled the sampling must take place during unloading at point of destination.

III. Annex

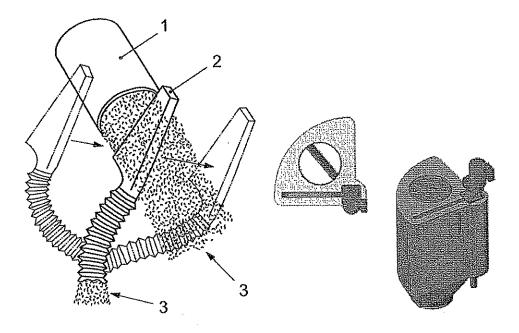
Annex . (informative)

Examples of mechanical sampling devices used on flowing grain

This annex describes the general types of mechanical sampling devices used on flowing grain, and provides illustrations of examples of such devices.

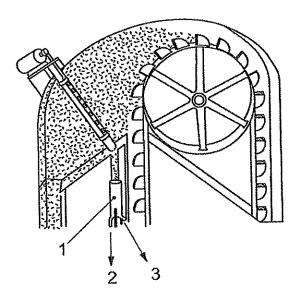
A.1 Crosscut sampling devices

Crosscut sampling devices allow a complete cross-section of a freely falling flow of grains to be taken. They may be open-nozzle sampling devices (see Figure A.1), tubular sampling devices with adjustable apertures (see Figure A.2) or tubular sampling devices with a worm screw (see Figure A.3).



- 1 nozzle
- 2 sampling device
- 3 grain

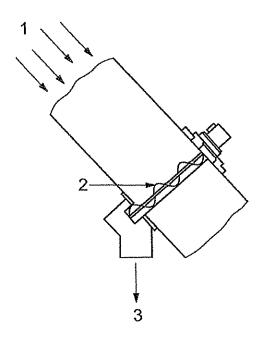
Figure A.1 — Open-nozzle crosscut sampling device, ensuring intermittent, repeated sampling



Key

- 1 sample divider
- 2 sample flow
- 3 return of excess grains into system

Figure A.2 — Tubular crosscut sampling device with adjustable apertures

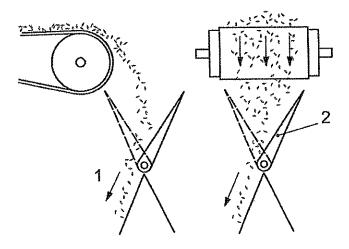


- 1 grain flow
- 2 worm screw
- 3 sample flow

Figure A.3 — Tubular sampling device with worm screw

A.2 Full-flow diverter-type sampling devices

In this type of sampling device, a flap or shutter intermittently diverts the flow of grain (see Figure A.4).



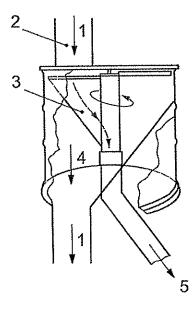
Key

- 1 sample flow
- 2 flap or shutter

Figure A.4 — Full-flow diverter-type sampling device

A.3 Rotating cup sampling devices

The freely falling flow of grain is intermittently sampled by a cup which rotates around a central vertical axis (see Figure A.5).

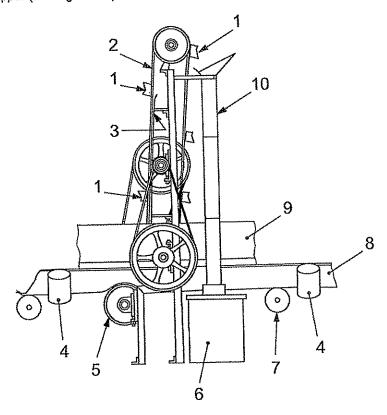


- 1 grain flow
- 2 vertical chute
- 4 flow
- 5 sample flow
- 3 rotating cup

Figure A.5 — Rotating cup sampling device

A.4 Bucket elevator sampling devices

This type of sampling device samples grain from a moving belt or conveyor. Buckets travelling in a continuous loop take samples over the entire width of the grain flow because the configuration of the lateral rollers concentrates the grain on the belt. Once the buckets have pivoted around the upper roller, the samples are delivered into the hopper (see Figure A.6).



Key

- 1 sampling bucket(s)
- 2 sampling bucket belt
- 3 belt guide
- 4 balance weight
- 5 special roller
- 6 samples container
- 7 conveyor roller
- 8 carrier belt
- 9 safety panel
- 10 hopper

NOTE As they are elevated, the buckets sample the grain from a belt or conveyor and, once they have pivoted around the upper roller, deliver the samples into the hopper.

Figure A.6 — Bucket elevator sampling device

Annex (informative)

Examples of instruments used to sample static products and instruments used to divide samples

This annex gives examples of instruments used to sample static products and instruments used to divide samples.

B.1 Cereal sampling instruments

- B.1.1 Instruments used to sample static bulk products in tote bags and rigid containers
- B.1.1.1 Manual concentric tapered sampling probes
- B.1.1.1.1 Open or closed shaft: with one or several apertures. See Figures B.1 and B.2.



Figure B.1 — Open shaft with single aperture



Figure B.2 — Open shaft with several apertures or closed shaft with compartments and several apertures

B.1.1.1.2 Open shaft with sequentially staggered apertures: several apertures. See Figure B.3.



Figure B.3 — Open shaft with several sequentially staggered apertures

- B.1.1.2 Gravity-type sampling probes with extension rods and T-shaped handles
- B.1.1.2.1 Gravity-type sampling probe: concentric. See Figure B.4.



Figure B.4 — Concentric gravity-type tapered probe head

B.1.1.2.2 Gravity-type sampling probe: cup-type. See Figure B.5.



Figure B.5 — Cup-type (head represented in open position)

- B.1.1.3 Mechanical sampling devices
- B.1.1.3.1 There are three main categories of mechanical sampling devices (see B.1.1.3.2 to B.1.1.3.4).
- B.1.1.3.2 Gravity-type sampling device. See Figure B.6.

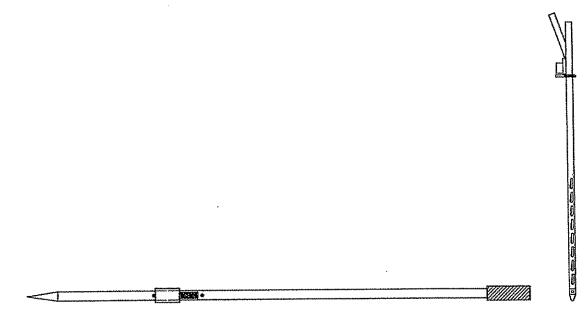
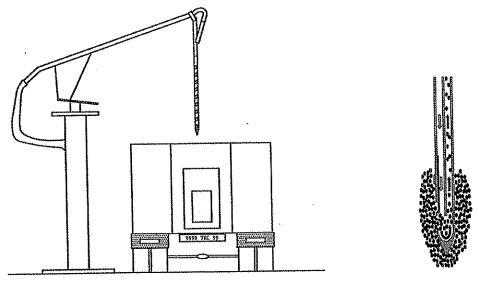


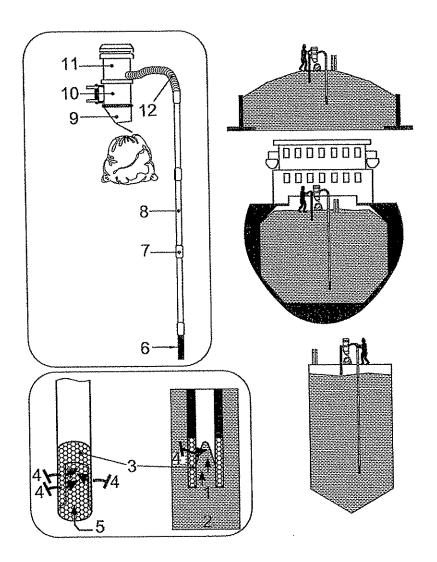
Figure B.6 — Gravity-type sampling device

B.1.1.3.3 Suction sampling device (sometimes called "vacuum sampling device"). See Figure B.7.



a) Example of sampling from a lorry

Figure B.7 (continued)



b) Example of sampling at depth over 2 m (ships, bulk tankers, etc.)

- 1 sample
- 2 grain mass
- 3 porous head unit
- 4 ai
- 5 grain sample
- 6 sampling head unit
- 7 coupling
- 8 extra sampling length
- 9 automatic discharge of collected sample
- 10 tank for collected sample
- 11 vacuum chamber
- 12 duct

Figure B.7 — Suction (or "vacuum") sampling device

B.1.1.3.4 Pneumatic sampling device (not represented)

B.1.2 Instruments used to take samples from sacks or bags including bulk sacks

B.1.2.1 Tapered sampling probes for sacks

Minimum diameter: 17 mm; aperture: 40 mm x 15 mm. See Figure B.8.

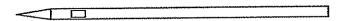


Figure B.8 — Tapered sampling probe for sacks

B.1.2.2 "Walking stick"-type sampling probe

Concentric tubes, minimum diameter: 20 mm:

- a) Open shaft: with one or several apertures;
- b) With compartments: with one or several apertures (see Figure B.9).



Figure B.9 — "Walking stick"-type, concentric sampling probe for sacks: open shaft with several compartments

B.1.2.3 Cone-shaped sampling device

See Figure B.10.

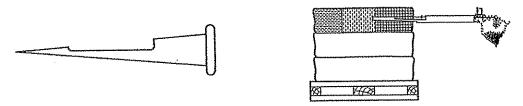


Figure B.10 — Cone-shaped sampling device

B.1.2.4 Gravity-type sampling probes with extension rods and T-shaped handles for open-topped sacks

See Figures B.4 and B.5.

B.1.2.5 Archimedes' screw sampling probe

Generally a small, portable, electric sampling probe. See Figure B.11.

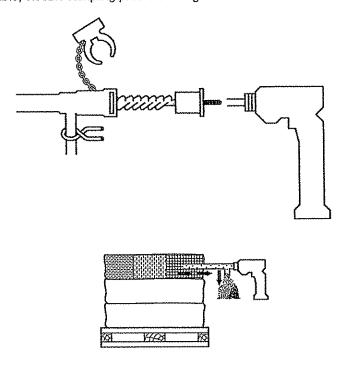


Figure B.11 — Archimedes' screw sampling probe (portable)

B.2 Instruments used to sample milled products, excluding products in granular form

B.2.1 Instruments used to sample static bulk products

B.2.1.1 Identical to those used to sample cereals (B.1.1), except for mechanical sampling devices.

Only two types of mechanical sampling device are suitable for the sampling of milled products (see B.2.1.2 and B.2.1.3). In general, pneumatic sampling devices are not suitable for this usage.

B.2.1.2 Electromechanical Archimedes' screw sampling probe (Figure B.12).

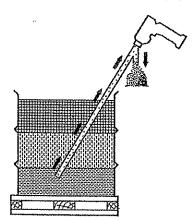


Figure B.12 — Electromechanical Archimedes' screw sampling probe

B.2.1.3 Gravity-type mechanical sampling device (Figure B.6).

B.2.2 Instruments used to take samples from sacks and bags

Identical to those used to sample cereals (B.1.2).

Guide to instruments suitable for sampling cereals and cereal products (STATIC SHAPLIN)

Reference to Figures in Annex		
Cereals in the form of grain	Milled and other cereal products	
B.1, B.2, B.3, B.4, B.5, B.6, B.7	B.12	
B.1, B.2, B.3, B.4, B.5, B.6, B.7	B.12	
B.1, B.2, B.3, B.4, B.5, B.6, B.7	B.12	
B.4, B.5, B.8, B.9, B.10, B.11	B.8, B.9, B.10, B.11, B.12	
	Cereals in the form of grain B.1, B.2, B.3, B.4, B.5, B.6, B.7 B.1, B.2, B.3, B.4, B.5, B.6, B.7 B.1, B.2, B.3, B.4, B.5, B.6, B.7	