COMMISSION IMPLEMENTING REGULATION (EU) 2022/1428

of 24 August 2022

laying down methods of sampling and analysis for the control of perfluoroalkyl substances in certain foodstuffs

(Text with EEA relevance)

THE EUROPEAN COMMISSION,

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

Having regard to Regulation (EU) 2017/625 of the European Parliament and of the Council of 15 March 2017 on official controls and other official activities performed to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection products, amending Regulations (EC) No 999/2001, (EC) No 396/2005, (EC) No 1069/2009, (EC) No 1107/2009, (EU) No 1151/2012, (EU) No 652/2014, (EU) 2016/429 and (EU) 2016/2031 of the European Parliament and of the Council, Council Regulations (EC) No 1/2005 and (EC) No 1099/2009 and Council Directives 98/58/EC, 1999/74/EC, 2007/43/EC, 2008/119/EC and 2008/120/EC, and repealing Regulations (EC) No 854/2004 and (EC) No 882/2004 of the European Parliament and of the Council, Council Directives 89/608/EEC, 89/662/EEC, 90/425/EEC, 91/496/EEC, 96/23/EC, 96/93/EC and 97/78/EC and Council Decision 92/438/EEC (Official Controls Regulation) ([1]), and in particular Article 34(6) thereof,

Whereas:

(1) Commission Regulation (EC) No 1881/2006 ([2]) sets out maximum levels for perfluoroalkyl substances (PFASs) in certain foodstuffs and Commission Recommendation (EU) 2022/1431 ([3]) lists indicative levels beyond which the Commission recommends that Member States investigate the causes of PFASs contamination in foodstuffs with high concentrations of PFASs. In order to ensure the reliability and consistency of official controls on the maximum levels for PFASs in certain foods, detailed requirements should be set for the methods used for sampling and for laboratory analyses.

(2) The measures provided for in this Regulation are in accordance with the opinion of the Standing Committee on Plants, Animals, Food and Feed,

HAS ADOPTED THIS REGULATION:

Article 1

For the purposes of this Regulation, the definitions and abbreviations set out in this article shall apply:

(1) 'lot' means an identifiable quantity of food delivered at one time and determined by the competent authority to have common characteristics, such as origin, variety, species, catch area, type of packing, packer, consignor or markings;

(2) 'sublot' means a physically separated and identifiable part of a large lot, designated to apply the sampling method;

(3) 'incremental sample' means a quantity of material taken from a single place in the lot or sublot;

(4) 'aggregate sample' means the combined total of all the incremental samples taken from the lot or sublot;

(5) 'laboratory sample' means a representative part or quantity of the aggregate sample intended for the laboratory;

(6) 'comparable size or weight' means a difference in size or weight that does not exceed 50 %;

([1]) OJ L 95, 7.4.2017, p. 1.
([2]) Commission Regulation (EC) No 1881/2006 of 19 December 2006 setting maximum levels for certain contaminants in foodstuffs (OJ L 364, 20.12.2006, p. 5).
([3]) Commission Recommendation (EU) 2022/1431 of 24 August 2022 on the monitoring of perfluoroalkyl substances in food (see page 105 of this Official Journal).

(7) 'precision' means the closeness of agreement between independent test results obtained under stipulated conditions. Precision is expressed as the standard deviation or coefficient of variation of the test results;

(8) 'within laboratory reproducibility or intermediate precision (RSD_R)' means precision under a set of within-laboratory conditions in a specific laboratory;

(9) 'limit of quantification ('LOQ')' means the lowest content of the analyte which can be measured with reasonable statistical certainty, i.e. the lowest concentration or mass of the analyte that has been validated with acceptable accuracy by applying the complete analytical method and identification criteria;

(10) 'combined standard measurement uncertainty ('u')' means a non-negative parameter associated with the result of measurement, which characterises the dispersion of values that could reasonably be attributed to the measurand based on the information used. It is obtained using the individual standard measurement uncertainties associated with the input quantities in a measurement model;

(11) 'expanded measurement uncertainty ('U')' means the value which is obtained using a coverage factor of 2 which gives a level of confidence of approximately 95 % (U = 2u);

(12) 'trueness' means the closeness of agreement between the average value obtained from a large series of test results and an accepted reference value. This value can be estimated from regular analysis of certified reference materials, fortification experiments or participation in inter-laboratory studies and is expressed as apparent bias.

Article 2

Sample preparation and analyses for the official control of the levels of PFASs in foodstuffs for which maximum levels have been established by Regulation (EC) No 1881/2006 shall be carried out in accordance with the methods set out in the Annex to this Regulation.

Article 3

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

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

Done at Brussels, 24 August 2022.

For the Commission
The President
Ursula VON DER LEYEN

ANNEX

PART A

SAMPLING METHODS

A.1. GENERAL PROVISIONS

A.1.1. **Material to be sampled**

Each lot or sublot which is to be examined shall be sampled separately.

A.1.2. **Incremental samples**

As far as possible, incremental samples shall be taken at various places distributed throughout the lot or sublot. Departure from such a procedure shall be recorded in the record provided for under point A.1.6.

A.1.3. **Preparation of the aggregate sample**

The aggregate sample shall be made up by combining the incremental samples. It shall be at least 1 kilogram or 1 litre unless not practical, e.g. when a single package has been sampled or when the product has a very high commercial value.

A.1.4. **Replicate samples**

In case replicate samples for enforcement, defence and reference purposes are taken, those replicate samples shall be taken from the homogenised aggregate sample, unless such procedure conflicts with a Member State's rules as regard the rights of the food business operator.

A.1.5. **Precautions**

In the course of sampling and the preparation of the samples, precautions shall be taken to avoid any changes, which would affect the content of PFASs, adversely affect the analytical determination or make the aggregate samples unrepresentative.

The person responsible for sampling shall take the following precautions:

(a) do not wear clothing or gloves that contain fluoropolymer linings or that are treated with PFASs to improve water and stain repellence;

(b) do not use PFASs containing moisturizers, cosmetics, hand cream, sunscreens and related products at the sampling day.

Materials used during sampling, sample storage and sample transmission shall be free of PFASs. The sample shall not come into contact with any materials such as cutting boards, sampling containers and linings of caps of sampling containers made of polytetrafluorethylene (PTFE or Teflon), polyvinylidene fluoride (PVDF) or other fluoropolymers. Contact with other PFASs containing materials shall be avoided.

A.1.6. **Sealing and labelling of samples**

Each sample shall be sealed at the place of sampling and identified in accordance with national rules.

A record shall be kept of each sampling, permitting each lot to be identified unambiguously and giving the date and place of sampling together with any additional information likely to be of assistance to the interpretation of the result.

A.1.7. **Packaging and transmission of samples**

Each sample shall be placed in a container which is clean, inert, made of polypropylene, polyethylene or other PFAS-free material, and apt to preserve the integrity of the sample and to offer adequate protection against contamination, loss of analytes by adsorption to the internal wall of the container and damage in transit. The use of glass containers is not permitted. All necessary precautions shall be taken to avoid any change in composition of the sample which might arise during transportation or storage.

A.2. SAMPLING PLANS

A.2.1. **Division of lots into sublots**

Large lots shall be divided into sublots on condition that the sublot can be separated physically. For products traded in large bulk consignments (e.g. vegetable oils) Table 1 shall apply. For other products, Table 2 shall apply. Taking into account that the weight of the lot is not always an exact multiple of the weight of the sublots, the weight of the sublot may exceed the mentioned weight by a maximum of 20 %.

Table 1

Subdivision of lots into sublots for products traded in bulk consignments

Lot weight (ton)	Weight or number of sublots
≥ 1 500	500 tonnes
> 300 and < 1 500	3 sublots
≥ 100 and ≤ 300	100 tonnes
< 100	—

Table 2

Subdivision of lots into sublots for products not traded in bulk consignments

Lot weight (ton)	Weight or number of sublots
≥ 15	15-30 tonnes
< 15	—

A.2.2. **Number of incremental samples**

The minimum number of incremental samples to be taken from the lot or sublot shall be as given in Tables 3 and 4.

In the case of bulk liquid products, the lot or sublot shall be thoroughly mixed in so far as possible and in so far it does not affect the quality of the product, by either manual or mechanical means immediately prior to sampling. In this case, a homogeneous distribution of contaminants shall be assumed within a given lot or sublot. In such case the number of incremental samples from a lot or sublot to form the aggregate sample shall be three.

Where the lot or sublot consists of individual packages or units, the number of packages or units (incremental samples) to be taken to form the aggregate sample shall be in accordance with Table 4.

The incremental samples shall be of similar weight/volume. The weight/volume of an incremental sample shall be at least 100 grams or 100 millilitres, resulting in an aggregate sample of at least about 1 kilogram or 1 litre. Where this is not possible, the provisions of A.2.6 shall apply.

Table 3

Minimum number of incremental samples to be taken from the lot or sublot of food, where the lot does not consist of individual packages or units of food

Weight or volume of lot/sublot (in kilogram or litre)	Minimum number of incremental samples to be taken
< 50	3
≥ 50 and ≤ 500	5
> 500	10

Table 4

Number of packages or units (incremental samples) to be taken to form the aggregate sample where the lot or sublot consists of individual packages or units of food

Number of packages or units in the lot/sublot	Number of packages or units to be taken
≤ 25	at least 1 package or unit
26-100	about 5 %, at least 2 packages or units
> 100	about 5 %, at maximum 10 packages or units

A.2.3. **Specific provisions for the sampling of lots containing whole fish of comparable size or weight**

The number of incremental samples to be taken from the lot is set out in Table 3. The aggregate sample uniting all incremental samples shall be at least 1 kilogram (see point A.1.3).

Where the lot to be sampled contains small fish (individual fish weighing < 1 kilogram), the whole fish is taken as incremental sample to form the aggregate sample. Where the resulting aggregate sample weighs more than 3 kilogram, the incremental samples may consist of the middle parts of the fish, weighing each at least 100 grams, forming the aggregate sample. The whole part to which the maximum level is applicable, is used for homogenisation of the sample.

The middle part of the fish is where the centre of gravity is. This is located in most cases at the dorsal fin (in case the fish has a dorsal fin) or halfway between the gill opening and the anus.

Where the lot to be sampled contains larger fish (individual fish weighing ≥ 1 kilogram), the incremental sample consists of the middle part of the fish. Each incremental sample weighs at least 100 grams. For fish of intermediate size (≥ 1 kilogram and < 6 kilogram), the incremental sample is taken as a slice of the fish from backbone to belly in the middle part of the fish.

For very large fish (≥ 6 kilogram), the incremental sample is taken from the right side (frontal view) dorso-lateral muscle meat in the middle part of the fish. Where the taking of such a piece of the middle part of the fish would result in a significant economic damage, the taking of three incremental samples of at least 350 grams each may be considered as being sufficient independent of the size of the lot or alternatively three incremental samples of at least 350 grams each from an equal part (175 grams) of the muscle meat close to the tail part and the muscle meat close to the head part of each fish may be considered as being sufficient independent of the size of the lot.

A.2.4. **Specific provisions for sampling of lots of fish containing whole fish of different size or weight**

The provisions of point A.2.3 shall apply.

Where a size or weight class/category is predominant (about 80 % or more of the lot), the sample is taken from fish with the predominant size or weight. This sample is to be considered as being representative for the whole lot.

Where no particular size or weight class/category predominates, then it shall be ensured that the fish selected for the sample are representative for the lot. Specific guidance for such cases is provided in 'Guidance on sampling of whole fishes of different size and/or weight' ([1])

A.2.5. **Specific provisions for the sampling of terrestrial animals**

For meat and offal of porcine, bovine, ovine, caprine and equine animals, a sample of 1 kilogram shall be taken from at least one animal. Where it is not possible to take a sample of 1 kilogram from at least one animal, equal sample quantities shall be taken from more than one animal to obtain a sample quantity of 1 kilogram.

([1]) https://ec.europa.eu/food/system/files/2022-05/cs_contaminants_sampling_guid-samp-fishes.pdf

For poultry meat, equal quantities shall be sampled from at least three animals in order to obtain an aggregate sample of 1 kilogram. For poultry offal, equal quantities shall be sampled from at least three animals in order to obtain an aggregate sample of 300 gram.

For meat and offal of farmed game animals and wild terrestrial animals, a sample of 300 gram shall be taken from at least one animal. Where it is not possible to a take a sample of 300 gram from at least one animal, equal sample quantities shall be taken from more than one animal to obtain a sample quantity of 300 gram.

A.2.6. **Alternative sampling methods**

Where it is not possible to carry out the sampling according the method set out in point A.2 because of the unacceptable commercial consequences (e.g. because of packaging forms, damage to the lot) or where it is practically impossible, an alternative method of sampling may be applied provided that it is sufficiently representative for the sampled lot or sublot and is fully documented. This shall be recorded in the record provided for in point A.1.6.

A.2.7. **Sampling at retail stage**

Sampling of foodstuffs at the retail stage shall be done where possible in accordance with the sampling provisions set out in point A.2. Where this is not possible, an alternative method of sampling at retail stage may be used, provided that it ensures sufficient representativeness for the sampled lot or sublot.

PART B

SAMPLE PREPARATION AND ANALYSIS

B.1. **Laboratory quality standards**

The principles as described in the EURL Guidance Document on Analytical Parameters for the Determination of Per- and Polyfluoroalkyl Substances in Food and Feed ([2]) shall be followed.

B.2. **Sample preparation**

B.2.1. **General requirements**

The basic requirement is to obtain a representative and homogeneous laboratory sample without introducing secondary contamination.

The complete aggregate sample, which is received by the laboratory, shall be finely ground, where relevant, and thoroughly mixed using a process that has been demonstrated to achieve complete homogenisation.

For products other than fish, all of the sample material received by the laboratory, to which the maximum level is applicable, shall be homogenised and used for the preparation of the laboratory sample.

For fish, all of the sample material received by the laboratory, to which the maximum level is applicable, shall be homogenised. From the homogenised aggregate sample, a representative part or quantity shall be used for the preparation of the laboratory sample.

Compliance with maximum levels laid down in Regulation (EC) No 1881/2006 shall be established on the basis of the levels determined in the laboratory samples.

B.2.2. **Specific sample preparation procedures and precautions**

The analyst shall ensure that samples do not become contaminated during sample preparation by following the precautions described in A.1.5. Furthermore, wherever possible, the apparatus and equipment coming into contact with the sample shall not contain PFASs and shall be replaced by e.g. stainless steel, high density polyethylene (HDPE) or polypropylene parts. These shall be cleaned with PFASs-free water or PFASs-free solvents and detergents.

([2]) https://ec.europa.eu/food/system/files/2022-05/cs_contaminants_sampling_guid-doc-analyt-para_0.pdf

Reagents and other equipment used for analysis and sampling shall be controlled to avoid possible introduction or loss of PFASs.

A reagent blank analysis shall be performed by carrying out the entire analytical procedure in the same manner as the test sample. When preparing reagent blanks, water may be used in place of the matrix. The levels in the reagent blanks shall be monitored in each sequence of samples.

B.3. **Methods of analysis: specific performance requirements**

Laboratories may select any validated method of analysis for the respective matrix provided that the selected method meets the specific performance criteria set out in Table 5.

Fully validated methods (i.e. methods validated by a collaborative trial for the respective matrix) shall be used or, where this is not possible, other validated methods (e.g. in-house validated methods for the respective matrix), provided that they fulfil the performance criteria set out in Table 5.

Where possible, the validation of in-house validated methods shall include the use of a certified reference material and/or participation in inter-laboratory studies.

Table 5

Parameter	Criterion
Applicability	Foods specified in Regulation (EC) No 1881/2006
Selectivity	Analytical methods shall demonstrate the ability to reliably and consistently separate the analytes of interest from other co-extracted and possibly interfering compounds that may be present.
Within-laboratory reproducibility (intermediate precision)(RSD_R)	≤ 20 %
Trueness	-20 % to +20 %
LOQ	The LOQ for PFOS, PFOA, PFNA and PFHxS each ≤ the maximum level for the respective individual PFAS. Compliance with this requirement entails that no LOQ shall be derived for the concentration of the sum of PFOS, PFOA, PFNA and PFHxS, which is calculated by summing up only the concentrations of PFOS, PFOA, PFNA and PFHxS, which were quantified at or above their respective LOQ.

PART C

REPORTING AND INTERPRETATION OF RESULTS

C.1. **REPORTING**

C.1.1. **Expression of results**

The results shall be reported as anions and be expressed in the same units and with the same number of significant figures as the maximum levels laid down in Regulation (EC) No 1881/2006. For the sum of PFOS, PFOA, PFNA and PFHxS, only the concentrations at and above the LOQ shall be taken into account to calculate the sum.

C.1.2. **Measurement uncertainty**

The analytical result shall be reported 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 % (U = 2u).

For the reporting of sum parameters and the possible comparison with legal limits, an estimation of the expanded measurement uncertainty shall also be done for these sum parameters. For PFASs, this is the case for the sum of PFOS, PFOA, PFNA and PFHxS and for total PFOS, if calculated as the sum of linear and branched PFOS.

In these cases, the calculation of the combined standard measurement uncertainty 'u' of the sum parameter is calculated as the square root of the sum of squares of the individual combined uncertainties.

The analyst shall note the 'Report on the relationship between analytical results, measurement uncertainty, recovery factors and the provisions of EU food and feed legislation' ([3]).

C.2. **INTERPRETATION OF RESULTS**

C.2.1. **Acceptance of a lot or sublot**

The lot or sublot is accepted if the analytical result of the laboratory sample does not exceed the respective maximum level as laid down in Regulation (EC) No 1881/2006, taking into account the expanded measurement uncertainty.

C.2.2. **Rejection of a lot or sublot**

The lot or sublot is rejected if the analytical result of the laboratory sample exceeds the respective maximum level as laid down in Regulation (EC) No 1881/2006, taking into account the expanded measurement uncertainty.

C.2.3. **Applicability**

The present rules on the interpretation of results shall apply for the analytical result obtained on the sample for enforcement. In case of analysis for defence or reference purposes, the national rules shall apply.

([3]) https://ec.europa.eu/food/system/files/2016-10/cs_contaminants_sampling_analysis-report_2004_en.pdf