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COMMISSION IMPLEMENTING REGULATION (EU) …/...

of XXX

on the performance of analytical methods for residues of pharmacologically active substances used in food-producing animals and on the interpretation of results as well as on the methods to be used for sampling and repealing Decisions 2002/657/EC and 98/179/EC

(Text with EEA relevance)

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(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 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 product, 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) Regulation (EU) 2017/625 lays down rules for the performance of official controls and other official activities by the competent authorities of the Member States for verifying compliance with Union legislation inter alia in the area of food safety at all stages of production, processing and distribution. It provides for specific rules on official controls in relation to substances whose use may result in residues in food and feed and sets general requirements for the methods to be used for sampling, laboratory analyses and tests during official controls and other official activities.

(2) Commission Decision 2002/657/EC([2]) sets requirements for the performance of analytical methods and the interpretation of results of analyses of certain substances and residues thereof in live animals and animal products and Commission Decision

[1] OJ L 95, 7.4.2017, p. 1.

[2] Commission Decision 2002/657/EC of 14 August 2002 implementing Council Directive 96/23/EC concerning the performance of analytical methods and the interpretation of results (OJ L 221, 17.8.2002, p. 8).

98/179/EC([3]) lays down detailed rules on official sampling for the monitoring of certain substances and residues thereof in live animals and animal products. Both Decisions were adopted on the basis of Directive 96/23/EC, which was repealed by Regulation (EU) 2017/625. In view of new scientific developments, those rules should be updated and they should be integrated into the framework for official controls defined by Regulation (EU) 2017/625.

(3) Decisions 98/179/EC and 2002/657/EC should therefore be repealed and replaced by this Regulation.

(4) In accordance with Regulation (EC) No 1831/2003 of the European Parliament and of the Council([4]) coccidiostats and histomonostats can be used as feed additives, therefore Commission Regulation (EC) No 152/2009([5]) laying down the methods of sampling and analysis for the official control of feed should be applied to analyses of their content in feed. However, this Regulation should apply where feed are analysed as part of follow-up actions in cases of suspected or established non-compliance with Union rules applicable to the use or residues of pharmacologically active substances authorised in veterinary medicinal products or as feed additives or with Union rules applicable to the use or residues of prohibited or unauthorised pharmacologically active substances.

(5) In order to ensure continuity in the performance of official controls and other official activities on residues of pharmacologically active substances and in order to avoid that all methods need to be re-validated at once, methods which have been validated before the date of entry into force of this Regulation may remain in use for a limited period, subject to the requirements of points 2 and 3 of Annex I to Decision 2002/657/EC, instead of the requirements laid down in Chapters 1 and 2 of Annex I to this Regulation.

(6) In order to have uniform requirements for all analytical methods, it is appropriate to set a timeline whereby all the requirements laid down in this Regulation should apply also to methods validated before the date of entry into force of this Regulation.

(7) 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

Subject matter and scope

This Regulation lays down rules concerning the methods of analysis used for sampling and for laboratory analyses in relation to residues of pharmacologically active substances in live food-producing animals, their body parts and fluids, excrements, tissues, products of animal origin,

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3 Commission Decision 98/179/EC of 23 February 1998 laying down detailed rules on official sampling for the monitoring of certain substances and residues thereof in live animals and animal products (OJ L 65, 5.3.1998, p. 31).

4 Regulation (EC) No 1831/2003 of the European Parliament and of the Council of 22 September 2003 on additives for use in animal nutrition (OJ L 268, 18.10.2003, p. 29).

5 Commission Regulation (EC) No 152/2009 of 27 January 2009 laying down the methods of sampling and analysis for the official control of feed (OJ L 54, 26.2.2009, p. 1).

animal by-products, feed and water. It also lays down rules for the interpretation of analytical results of these laboratory analyses.

This Regulation applies to official controls aimed at verifying compliance with the requirements on the presence of residues of pharmacologically active substances.

Article 2

Definitions

For the purposes of this Regulation, the definitions in Article 2 of Commission Delegated Regulation (EU) 2019/2090([6]), in Commission Regulation (EU) 2019/1871([7]), in Article 2 of Regulation (EC) No 470/2009([8]) of the European Parliament and of the Council and in Council Regulation (EEC) No 315/93([9]) shall apply.

The following definitions shall also apply:

(1) 'absolute recovery' means the yield of the final stage of an analytical process for an analyte divided by the amount of the analyte in the original sample, expressed as a percentage;

(2) 'accuracy' means the closeness of agreement between a test result and the accepted true reference value, determined by estimating trueness and precision([10]);

(3) 'alpha (α) error' means the probability that the tested sample is compliant, even though a non-compliant measurement result has been obtained (the probability of a false non-compliant result);

(4) 'analyte' means the component of a system to be analysed;

(5) 'authorised substance' means a pharmacologically active substance authorised for use in food-producing animals in accordance with Directive 2001/82/EC of the European Parliament and of the Council([11]);

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6 Commission Delegated Regulation (EU) 2019/2090 of 19 June 2019 supplementing Regulation (EU) 2017/625 of the European Parliament and Council regarding cases of suspected or established non-compliance with Union rules applicable to the use or residues of pharmacologically active substances authorised in veterinary medicinal products or as feed additives or with Union rules applicable to the use or residues of prohibited or unauthorised pharmacologically active substances (OJ L 137, 9. 12. 2019, p. 28).

7 Commission Regulation (EU) 2019/1871 of 7 November 2019 on reference points for action for non-allowed pharmacologically active substances present in food of animal origin and repealing Decision 2005/34/EC (OJ L 289, 8.11.2019, p. 41).

8 Regulation (EC) No 470/2009 of the European Parliament and of the Council of 6 May 2009 laying down Community procedures for the establishment of residue limits of pharmacologically active substances in foodstuffs of animal origin, repealing Council Regulation (EEC) No 2377/90 and amending Directive 2001/82/EC of the European Parliament and of the Council and Regulation (EC) No 726/2004 of the European Parliament and of the Council (OJ L 152, 16.6.2009, p. 11).

9 Council Regulation (EEC) No 315/93 of 8 February 1993 laying down Community procedures for contaminants in food (OJ L 037, 13.2.1993, p. 1).

10 ISO 3534-1: 2006 Statistics - Vocabulary and symbols – Part 1: General statistical terms and terms used in probability (Chapter 1).

11 Directive 2001/82/EC of the European Parliament and of the Council of 6 November 2001 on the Community code relating to veterinary medicinal products (OJ L 311, 28.11.2001, p. 1).

(6) 'beta (β) error' means the probability that the tested sample is truly non-compliant, even though a compliant measurement result has been obtained (the probability of a false compliant result);

(7) 'bias' means the difference between the estimated value of the test result and an accepted reference value;

(8) 'calibration standard' means a traceable reference for measurements that represents the quantity of substance of interest in a way that ties its value to a reference base;

(9) 'certified reference material' (CRM) means a reference material, accompanied by documentation issued by a delegated body and providing one or more specified property values with associated uncertainties and traceabilities, using valid procedures([12]);

(10) 'co-chromatography' means a technique in which an unknown substance is applied to a chromatographic support together with one or more known compounds, in the expectation that the relative behaviour of the unknown and known substances will assist in the identification of the unknown one;

(11) 'collaborative study' means analysing the same sample(s) by using the same method to determine performance characteristics of the method in different laboratories, where the study allows to calculate the random measurement error and laboratory bias for the method used;

(12) 'confirmatory method' means a method that provides full or complementary information enabling the substance to be unequivocally identified and if necessary quantified in one of the following manners:

(a) at the maximum residue level or maximum level for authorised substances;

(b) at the reference points for action (RPA) for prohibited or unauthorised substances, for which a reference point for action is established;

(c) at a concentration as low as reasonably achievable for prohibited or unauthorised substance, for which no reference point for action is established;

(13) 'coverage factor (k)' means a number which expresses the desired level of confidence and which is associated with the expanded measurement uncertainty;

(14) 'decision limit for confirmation (CCα)' means the limit at and above which it can be concluded with an error probability of α that a sample is non-compliant and the value 1 – α means statistical certainty (in percentage) that the permitted limit has been exceeded;

(15) 'detection capability of screening (CCβ)' means the smallest content of the analyte that may be detected or quantified in a sample with an error probability of β:

(a) in the case of prohibited or unauthorised pharmacologically active substances, the CCβ is the lowest concentration at which a method is able to detect or

[12] JCGM 200:2008, International vocabulary of metrology — Basic and general concepts and associated terms (VIM), Third Edition 2008: https://www.iso.org/sites/JCGM/VIM-JCGM200.htm (Chapter 5 Measurement standards (Etalons)).

quantify, with a statistical certainty of 1 – β, samples containing residues of prohibited or unauthorised substances;

(b) in the case of authorised substances, the CCβ is the concentration at which the method is able to detect concentrations below the permitted limit with a statistical certainty of 1 – β;

(16) 'fortified (spiked) sample material' means a sample enriched with a known amount of the analyte to be detected or quantified;

(17) 'inter-laboratory study' means the organisation, performance and evaluation of tests on the same sample(s) by two or more laboratories in accordance with predetermined conditions to evaluate testing performance, either as a collaborative study or a proficiency test;

(18) 'internal standard (IS)' means a substance not contained in the sample and having physico-chemical properties as similar as possible to those of the analyte to be identified or quantified;

(19) 'level of interest' means the concentration of a substance or analyte in a sample that is significant to determine its compliance with the legislation as regards:

(a) the maximum residue level or maximum level for authorised substances;

(b) reference points for action for prohibited or unauthorised substances, for which a reference point for action is established;

(c) a concentration as low as analytically achievable for prohibited or unauthorised substance, for which no reference point for action is established;

(20) 'lowest calibrated level' (LCL) means the lowest concentration on which the measuring system has been calibrated;

(21) 'matrix' means the material from which a sample is taken: animal body parts and fluids, excrements, tissues, products of animal origin, animal by-products, feed and water;

(22) 'matrix effect' means the difference in analytical response between a standard dissolved in the solvent and a matrix-matched standard (fortified post extraction and clean-up) either without a correction using an internal standard or with correction using an internal standard;

(23) 'matrix-matched standard' means a blank analyte-free matrix to which the analyte is added at a range of concentrations after sample processing;

(24) 'matrix-fortified (spiked) standard' means a blank analyte-free matrix, which prior to solvent extraction and sample processing, is spiked with the analyte at a range of concentrations;

(25) 'measurand' means the particular quantity subject to measurement;

(26) 'measurement uncertainty' 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;

(27) 'performance criteria' means requirements for a performance characteristic according to which it can be judged that the analytical method is fit for the intended use and generates reliable results;

(28) 'precision' means the closeness of agreement between independent test results obtained under stipulated conditions and is usually expressed as the standard deviation or coefficient of variation of the test result;

(29) 'qualitative method' means an analytical method, which detects or identifies a substance or a group of substances on the basis of its chemical, biological or physical properties;

(30) 'quantitative method' means an analytical method, which determines the amount or mass fraction of a substance so that it may be expressed as a numerical value of appropriate units;

(31) 'recovery' means the recovery corrected amount of an analyte divided by the fortified amount of the analyte in the matrix sample, expressed as a percentage;

(32) 'recovery correction' means the use of internal standards, the use of a matrix calibration curve as well as the use of a recovery correction factor and also a combination of these approaches;

(33) 'reference material' means a material sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process or in examination of nominal properties([13]);

(34) 'relative matrix effect' means the difference in analytical response between a standard dissolved in the solvent and a matrix-matched standard with a correction using an internal standard;

(35) 'repeatability' means precision under repeatability conditions;

(36) 'repeatability conditions' means conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time;

(37) 'reproducibility' means precision under reproducibility conditions;

(38) 'reproducibility conditions' means conditions, where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment([14]);

(39) 'ruggedness' means the susceptibility of an analytical method to changes in experimental conditions under which the method can be applied as presented or with specified minor modifications;

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[13] Codex Alimentarius Commission, Food and Agriculture Organization of the United Nations/World Health Organization, Guidelines on analytical terminology (CAC/GL 72-2009).

[14] ISO 5725-1:1994 Accuracy (trueness and precision) of measurement methods and results – Part 1: General principles and definitions (Chapter 3).

(40) 'screening method' means a method that is used for screening of a substance or class of substances at the level of interest and which are used to sift large numbers of samples for potential non-compliant results;

(41) 'screening target concentration' (STC) means the concentration lower than or equal to the CCβ at which a screening measurement categorises the sample as "Screen Positive" (potentially non-compliant) and triggers a confirmatory testing;

(42) 'selectivity' means the ability of a method to distinguish between the analyte being measured and other substances;

(43) 'single laboratory study (in-house validation)' means an analytical study involving a single laboratory using one method to analyse the same or different test materials under different conditions over justified long time intervals;

(44) 'standard addition' means a procedure in which one part of the sample is analysed as such and known amounts of the standard analyte are added to the other test portions before analysis;

(45) 'standard analyte' means an analyte of known and certified content and purity to be used as a reference in the analysis;

(46) 'substance' means matter of constant composition characterised by the entities which compose it and by certain physical properties;

(47) 'test portion' means the quantity of material drawn from the sample on which the test or observation is carried out;

(48) 'trueness' means the closeness of agreement between the average value obtained from a large series of test results and an accepted reference value and is usually expressed as bias;

(49) 'units' means those units described in ISO 80000([15]) and Council Directive 80/181/EEC([16]);

(50) 'validation' means the demonstration by examination and the provision of effective evidence that the particular requirements of a specific intended use are fulfilled([17]), through a single laboratory study or a collaborative study;

(51) 'within-laboratory reproducibility (intermediate precision/in-house reproducibility)' means measurement precision under a set of within-laboratory conditions in a specific laboratory.

Article 3

Methods of analysis

15 ISO 80000-1:2009 Quantities and units — Part 1: General (Introduction).

16 Council Directive 80/181/EEC of 20 December 1979 on the approximation of the laws of the Member States relating to units of measurement and on the repeal of Directive 71/354/EEC, OJ L 39, 15.2.1980, p. 40).

17 ISO/IEC 17025:2017 General requirements for the competence of testing and calibration laboratories (Chapter 3).

Member States shall ensure that the samples taken in accordance with Article 34 of Regulation (EU) 2017/625 are analysed using methods that comply with the following requirements:

(1) they are documented in test instructions, preferably according to Annexes of ISO 78-2:1999 Chemistry-Layouts for standards – Part 2: Methods of chemical analysis([18]);

(2) they comply with the performance criteria and other requirements for analytical methods laid down in Chapter 1 of Annex I to this Regulation;

(3) they have been validated in accordance with the requirements laid down in Chapters 2 and 4 of Annex I to this Regulation;

(4) they allow enforcement of the reference points for action, laid down in Regulation (EU) 2019/1871, the identification of the presence of prohibited and unauthorised substances and the enforcement of maximum levels (MLs), which have been set on the basis of Regulation (EEC) No 315/93 and Commission Regulation (EC) No 124/2009([19]) and maximum residue limits (MRLs), which have been set on the basis of Regulations (EC) No 1831/2003 and No 470/2009.

Article 4

Quality control

Member States shall ensure the quality of the results of analyses performed pursuant to Regulation (EU) 2017/625, in particular by monitoring tests or calibration results in accordance with ISO/IEC 17025:2017 General requirements for the competence of testing and calibration laboratories and with the requirements for quality control during routine analysis as laid down in Chapter 3 of Annex I to this Regulation.

Article 5

Interpretation of results

(1) The result of an analysis shall be considered non-compliant where it is equal to or above the decision limit (CCα) of the confirmatory method.

(2) For authorised substances for which an MRL or ML has been established, the decision limit shall be the concentration at and above which it can be decided with a statistical certainty of numerical value 1 – α that the permitted limit has been exceeded.

(3) For unauthorised or prohibited substances or for authorised substances for which no MRL or ML has been established in a specific species or product, the decision limit shall be the lowest concentration level at which it can be decided with a statistical certainty of numerical value 1 – α that the particular analyte is present.

(4) For unauthorised or prohibited pharmacologically active substances the α error shall be 1% or lower. For all other substances, the α error shall be 5% or lower.

[18] ISO 78-2: 1999 Chemistry - Layouts for standards - Part 2: Methods of chemical analysis (Annexes).

[19] Commission Regulation (EC) No 124/2009 of 10 February 2009 setting maximum levels for the presence of coccidiostats or histomonostats in food resulting from the unavoidable carry-over of these substances in non-target feed (OJ L 40, 11.2.2009, p. 7).

Article 6

Methods for sampling

Member States shall ensure that samples are taken, handled and labelled in accordance with the detailed methods for sampling laid down in Annex II to this Regulation.

Article 7

Repeals and transitional measures

Decisions 2002/657/EC and 98/179/EC are repealed from the date of entry into force of this Regulation.

However, until [*publications office please enter date five years after the date of entry into force*], the requirements laid down in points 2 and 3 of Annex I to Decision 2002/657/EC shall continue to apply to methods, which have been validated before the date of entry into force of this Regulation.

Article 8

Entry into force

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,

For the Commission
The President
Ursula VON DER LEYEN