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Working document on the summing up of LOQs in case of complex residue definitions.

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This document has been conceived as a working document of the Commission Services, which was elaborated in co-operation with the Member States. It does not intend to produce legally binding effects and by its nature does not prejudice any measure taken by a Member State within the implementation prerogatives under Regulation 1107/2009, nor any case law developed with regard to these provisions. This document also does not preclude the possibility that the European Court of Justice may give one or another provision direct effect in Member States.

Working document on the summing up of LOQs in case of complex residue definitions.

Introduction

Complex residue definitions are residue definitions (RD) that contain more than one component. In cases where for such residue definition, individual components are measured separately, different Member States used so far different approaches for reporting the LOQ value for the analysis result for the total residue definition (resLOQ value) to EFSA using the Standard Sample Description (SSD) format¹. This document fixes a general approach to avoid such divergences in future. The general recommendations on reporting results in accordance with the EFSA guidance on reporting data on pesticide residues in food and feed are applicable. The purpose of this document is to explain the new provisions for reporting the results for complex residue definitions where the individual components of the complex residue definition are measured separately, in particular for reporting the LOQs for the individual components and the full complex legal residue definitions.

The provisions described in this document for summing up of LOQs are not relevant for residue definitions that are analysed with common moiety methods (e.g. dithiocarbamates expressed as CS₂, including maneb, mancozeb, metiram, propineb, thiram and ziram), since in this case only one analyte is measured. The document equally does not apply to other methods where the components are not measured separately (e.g. salts).

Approach used for deriving MRL proposals

For MRL setting in case of complex residue definitions, where residues are present at levels below the LOQ, EFSA follows the OECD Guidance². Based on supervised field trials the LOQs of all individually measured components for which residues are \leq LOQ, are added up for deriving the input values to calculate the MRL, using the OECD calculator. The currently used practice for MRL setting is not affected by this working document.

Harmonised approach for reporting results of pesticide monitoring analysis

In collaboration with the EURLs and MS, EFSA will elaborate a list of the components that can be determined individually. For all these compounds a specific paramCode will be assigned. Also hierarchical links will be established identifying which components belong to which residue definition. However, the choice will be left up to the Member States to decide on which analytical method to use e.g. a common moiety method or a method analysing all components of the residue definition separately.

¹ More details on the data elements used to report the results of pesticide monitoring results can be found in the EFSA guidance document on Reporting data on pesticide residues in food and feed according to Regulation (EC) No 396/2005 (2014 data collection), available <http://www.efsa.europa.eu/en/efsajournal/pub/4195>.

² Guidance on crop field trials ENV/JM/MONO(2011)50 of 29 Sept 2011.

1) Sensitivity check of the method for MRLs set at the LOQ:

In order to decide if the analytical methods used for pesticide monitoring in accordance with Article 28 of Regulation (EC) No 396/2005 is sufficiently sensitive to identify samples containing residues that exceed the legal limits, a sensitivity check needs to be performed by the laboratories. Each laboratory should therefore determine analytically the individual LOQs of all components that are measured separately. It has to be verified whether the applied method is sensitive enough by checking whether the sum of the individual components' LOQs obtained is below or equal to the MRL set at the LOQ. In this check the molecular weight conversion factor needs to be introduced; this conversion factor is needed to recalculate the LOQ of the individual component to the compound for which the MRL is expressed (e.g. azocyclotin, expressed as cyhexatin).

Example: Residue definition containing 5 components; MRL is set at the LOQ of 0.1* mg/kg for the sum of the 5 components, expressed as the first component.

The individual LOQs for the 5 components are:

LOQ c1 = 0.01 mg/kg, CF₁=1 (CF to recalculate the LOQ for c1 to the compound on which the MRL is expressed)

LOQ c2 = 0.01 mg/kg, CF₂ = 1.5

LOQ c3 = 0.02 mg/kg, CF₃ =0.8

LOQ c4 = 0.02 mg/kg, CF₄ = 1

LOQ c5 = 0.01 mg/kg, CF₅= 1

Sensitivity check for LOQ for the legal RD:

$LOQ(\text{legal RD}) = LOQ_{c1} * CF_1 + LOQ_{c2} * CF_2 + LOQ_{c3} * CF_3 + LOQ_{c4} * CF_4 + LOQ_{c5} * CF_5 = 0.071$
mg/kg

As $LOQ(\text{legal RD}) \leq MRL (0.1^* \text{ mg/kg}) \Rightarrow$ sensitivity check OK.

2) Reporting of results:

All individual components, as far as they are measured separately, need to be reported separately together with their individual LOQ. In practice, there will be two cases:

Case 2a) None of the components of the complex residue definition occurred in a measurable concentration (all below LOQ):

For the LOQ of the total residue definition (resLOQ), Member States can use a default value of 99999, indicating that the overall LOQ was not calculated or they can report the result of the sensitivity check (LOQ (legal RD)). In case the 99999 code was used, EFSA will calculate the overall resLOQ using the algorithm described in the sensitivity check. In Table 1 the relevant data elements of the SSD are described.

Table 1: Example how to report results in SSD for a complex residue definition where all the components of the complex residue definition were found to be below the LOQ

Element name	Element label	Element value (code description)
paramCode	Parameter code	Valid paramCode for the respective legal residue definition (paramCode (legal RD) (the MatrixTool, Table A can be used to verify the validity of the paramCode); in addition the paramCodes for the individual components of the complex residue definition as far as they were analysed (paramCode for c1, c2, c3, c4, c5)
paramText	Parameter text	Not mandatory (see Section 5.6 of 2014 EFSA guidance document on the use of the SSD)
paramType	Type of parameter	For paramCode (legal RD): P005A if sample was analysed for all components of the complex RD P004A if sample was not analysed for all components of the complex RD For paramCode for c1, c2, c3, c4 and c5: P002A
resLOQ	Result LOQ	For paramCode (legal RD): 99999 or the result of the sensitivity check; For paramCode for c1, c2, c3, c4 and c5: LOQ expressed for the individual components (not recalculated to the compound for which the MRL is expressed).
resVal	Result value	For paramCode (legal RD) and for paramCode for c1, c2, c3, c4 and c5: leave the field blank
resType	Type of result	For paramCode (legal RD) and paramCode for c1, c2,c3, c4 and c5: LOQ or BIN (if the sample was analysed with a screening method)
resEvaluation	Evaluation of the result	For paramCode (legal RD): J002A, provided the sensitivity test was successful/satisfactory For paramCode for c1, c2, c3, c4 and c5: J029A
resComm	Comment of the result	Free text, where considered necessary (not mandatory)

Case 2b) At least one of the components of a complex residue definition occurred in a concentration \geq LOQ

As regards the data element resLOQ the same provisions as described in Case 2a are applicable. In the data element resVal the numerical value of the residue concentration measured for the legal residue definition has to be reported. For complex residue definitions, the results of the quantified residues (all individual component's residues present in concentrations at or above the individual component's LOQ) are summed up (res Val). This value will be used to be compared with the MRL. In addition, the residue concentrations for the individual components have to be reported. The detailed description of the data elements can be found in Table 2.

Table 2: Example how to report results in SSD for a complex residue definition where at least one of the components of the complex residue definition was quantified at or above the LOQ

Element name	Element label	Element value (code description)
paramCode	Parameter code	Valid paramCode for legal RD (the MatrixTool, Table A can be used to verify the validity of the paramCode); in addition paramCodes for the individual components of the complex residue definition as far as they were analysed (paramCode for c1, c2, c3, c4, c5)
paramText	Parameter text	Not mandatory (see Section 5.6 of 2014 EFSA guidance document on the use of the SSD)
paramType	Type of	For paramCode (legal RD):P005A if sample was analysed for all components of the

Element name	Element label	Element value (code description)
	parameter	complex RD; P004A if sample was not analysed for all components of the complex RD For paramCode for c1, c2, c3, c4 and c5: P002A
resLOQ	Result LOQ	For paramCode (legal RD): 99999 or the result of the sensitivity check For paramCode for c1, c2, c3, c4, c5: LOQ expressed for the individual components (not recalculated to the compound for which the MRL is expressed).
resVal	Result value	For paramCode (legal RD): report the sum of the detected components (C_{total}), (corrected for molecular weight) in addition report the results of the detected components (paramCode for c1, c2, c3, c4, c5) (not corrected for molecular weight).
resType	Type of result	For paramCode (legal RD): VAL; For paramCode for c1, c2, c3, c4 and/or c5: VAL or LOQ as appropriate
resEvaluation	Evaluation of the result	For paramCode (legal RD): J002A, J003A, J031A or J029A, as appropriate; For paramCode for c1, c2, c3, c4 and c5: J029A
resComm	Comment of the result	Free text, if considered necessary (not mandatory)

In case during the reporting of the results for certain individual components of the complex legal residue it becomes apparent that no paramCode has been assigned yet, the Member States should report the resVal and resLOQ for the full residue definition only. In such case the numerical value for the resLOQ of the sum result need to be reported. In addition, the data providers are invited to notify EFSA the need to allocate a new paramCode for the component of the complex residue definition for which a paramCode is missing.

Application date:

This document was taken note of during the PAFF Committee on pesticides residues of 30/11 – 01/12 2015. In order to allow the development of the necessary database on the components that are measured separately, the molecular weight conversion factors, the assignment of the new paramCodes needed and the implementation of the necessary changes in the SSD Guidance document, this working document will apply for monitoring data generated as from 1 January 2017 onwards.

Examples for reporting analysis results

MRL for Aldicarb (sum of aldicarb, its sulfoxide and its sulfone, expressed as aldicarb) on oranges: 0.02* mg/kg

Aldicarb resLOQ=0.006 mg/kg, CF_{MW}=1

Aldicarb sulfone resLOQ= 0.006 mg/kg, CF_{MW}=0.85

Aldicarb sulfoxide resLOQ= 0.006 mg/kg, CF_{MW}= 0.92

Sensitivity check (to be checked by the lab): sum of individual LOQs (taking into account CF) of 0.017 mg/kg ≤ MRL 0.02*mg/kg ⇒ OK

Example 1: Full legal residue definition was analysed and all components of the RD were below the LOQ

<Param Code> RF-0020-001-PPP Aldicarb (sum of aldicarb, its sulfoxide and its sulfone, expressed as aldicarb)

<Param Type> P005A (sample analysed according to full residue definition)

resLOQ = 99999 (example of a code that could be introduced as a reference to the LOQs of the individual metabolites) or 0.017

resType = LOQ

resVal = blank

<Param Code> RF-0020-002 (aldicarb)

<Param Type> P002A (part of a sum)

resLOQ = 0.006 mg/kg

resType = LOQ

resVal = blank

<Param Code> RF-0020-003 (aldicarb-sulfoxide)

<Param Type> P002A (part of a sum)

resLOQ = 0.006 mg/kg

resType = LOQ

resVal = blank

<Param Code> RF-0020-004 (aldicarb-sulfone)

<Param Type> P002A (part of a sum)

resLOQ = 0.006 mg/kg

resType = LOQ

resVal = blank

Example 2: Full legal residue definition was analysed and only one component was quantified above the LOQ

<Param Code> RF-0020-001-PPP Aldicarb (sum of aldicarb, its sulfoxide and its sulfone, expressed as alidicarb)

<Param Type> P005A (sample analysed according to full residue definition)

resLOQ = 99999 (example of a code that could be introduced as a reference to the LOQs of the individual metabolites) or 0.017

resType = VAL

resVal = 0.0085 mg/kg

<Param Code> RF-0020-002 (aldicarb)

<Param Type> P002A (part of a sum)

resLOQ = 0.006 mg/kg

resType = LOQ

resVal = blank

<Param Code> RF-0020-003 (aldicarb-sulfoxide)

<Param Type> P002A (part of a sum)

resLOQ = 0.006 mg/kg

resType = LOQ

resVal = blank

<Param Code> RF-0020-004 (aldicarb-sulfone)

<Param Type> P002A (part of a sum)

resLOQ = 0.006 mg/kg

resType = VAL

resVal = 0.01 mg/kg

Example 3: a screening method where only 1 component was analysed and was not detected³

<Param Code> RF-0020-001-PPP Aldicarb (sum of aldicarb, its sulfoxide and its sulfone, expressed as alidicarb)

<Param Type> P004A (sample analysed according to only a part of the residue definition)

resLOQ = 99999 (example of a code that could be introduced as a reference to the LOQs of the individual metabolites)

resType = BIN

resVal = blank

<Param Code> RF-0020-002 (aldicarb)

<Param Type> P002A (part of a sum)

resLOQ = 0.006 mg/kg

resType = BIN

resVal = blank

The components that were not analysed (param Code RF-0020-003 (aldicarb-sulfoxide) and param Code RF-0020-004 (aldicarb-sulfone)) should not be reported.

Remark: Please be aware that residue definitions and the respective PARAM codes of the SSD may change in future. This example only serves to illustrate the principle. For further examples and guidance on how reporting should be done, reference is made to 'Use of the EFSA Standard Sample Description for the reporting of data on the control of pesticide residues in food and feed according to Regulation (EC) No 396/2005 (EFSA Journal 2014; 12(1): 3545)' or the most recent version of this document.

³ In case a screening method was used and the marker component was detected, a confirmatory analysis needs to be carried out in agreement with the full legal residue definition and with a quantitative method.