OECD/OCDE **506**

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# OECD GUIDELINE FOR THE TESTING OF CHEMICALS

## Stability of Pesticide Residues in Stored Commodities

### INTRODUCTION

1. When compiling magnitude of residue (MOR) data from crops, crop products and products of animal origin it is essential to ensure that the residues of all components of the residue definitions (both risk assessment and enforcement) of a MOR sample remain accurately quantifiable from the time of sampling /harvest to analysis. If MOR samples are not analysed as quickly as possible after collection, chemical changes to the components of the residue may take place which may lead to inaccurate results. In cases where it is not possible to analyse MOR samples immediately after they have been taken, the MOR sample should be stored under suitable sub zero ºC conditions until analysis. In these cases the effect of storage conditions on the stability of residues should be investigated. Key references for this guideline are listed in paragraph 38(i) through 38(v) inclusive.

### PURPOSE

2. The aim of these studies is to demonstrate the time period for which stability has been shown in representative commodities from crops, by extrapolation to processed fractions derived from crops, and products of animal origin. It is then necessary for applicants to ensure that MOR samples are analysed within the shortest period for which stability has been shown in representative commodities. The MOR studies include, but are not limited to, the following: Crop field trials; Residues in limited field rotational crops; Livestock feeding studies; and Processing studies.

### GENERAL CONSIDERATIONS

3. In most MOR studies, MOR samples are stored for a period of time prior to analysis. During this storage period residues of the pesticide and/or its metabolites included in the residue definitions may decline due to processes such as volatilization or enzymatic degradation. Therefore, in order to be certain that the level of residues that were present in MOR samples at the time of their collection are the same at the time of analysis, controlled studies are needed to assess the effect storage has on residue levels in MOR samples. In other words, applicants need to demonstrate that pesticide residues are stable during frozen storage of the MOR sample to be analysed or show the degree to which residues decline in that period of time.

4. If MOR samples are always analysed within 30 days of their storage in frozen conditions, applicants can omit conducting a freezer storage stability study provided justification is given e.g. basic physical chemical properties data show residues are not volatile or labile. Normally, MOR samples should be frozen within 24 hours of sampling or harvest. However, where this is not the case, the period of ambient or cooled storage should be considered in the planning of the freezer storage stability study.

5. Under normal circumstances, it is accepted that concurrent residue freezer storage stability studies will not be required for all commodities since data from one commodity may be representative of

other related commodities (see commodity categories in Annex 1). Provided that the pesticide residues are demonstrated to be stable in the commodities of interest, a freezer storage stability study run in a separate freezer commencing on a different date will be acceptable if the storage conditions (especially temperature and matrix form i.e. whole versus homogenized) are the same as those in the corresponding MOR study.

6. For pesticides whose residues are known or suspected to be unstable or volatile (including fumigants), consideration should be given to undertaking a freezer storage stability study with all individual components of the residue definitions in advance of the MOR studies to determine proper storage conditions and maximum storage times before treated MOR samples are placed into storage. Otherwise, concurrent studies may be required to ensure that the data on the components of the residue definitions from the MOR studies are acceptable.

## TEST PROCEDURE

### Introduction

7. Freezer storage stability studies should include sufficient starting material and should have a sufficiently high concentration of residue to allow for any observed decline during storage to be quantified. Samples could either be from crops (or animals) that have been treated with pesticides in the field i.e. incurred residues, or from the spiking of control (untreated) commodities with known amounts of each component of the residue definitions. Freshly spiked control samples of the stored commodities should be analysed at each of the time points when aged/stored commodities are removed from frozen storage for analysis, to permit differentiation of procedural losses from those due to storage conditions and duration. Whilst the control samples used for procedural recovery determinations will be the same commodity, they need not be the same batch of test material as was spiked and stored. In the case of incurred residues, samples must be analysed within as short a time period as possible after harvest, to quantify the time zero residue levels.

### Test Substance

8. Generally, it is anticipated that the formulation will not play a significant role in the stability of the residue components in the harvested crop under frozen storage conditions. However, in the unlikely event that this is not the case, the applicant should supply a rationale for the validity of the freezer storage stability results. If the freezer storage stability study uses incurred residues, then it should be established that all components of the residue definitions are present in the MOR samples and at sufficient levels to allow any decline to be observed.

9. If test substances are added to untreated commodities in the laboratory, it is usually the active substance and/or relevant identified metabolites that are added. Where the residue definitions contain more than one component studies need to be designed to demonstrate stability of each component. Consequently, the use of mixed spiking solutions is not recommended as it could mask potential transformations from one compound to another. Therefore, the freezer storage stability study should be conducted with separate samples of each commodity under investigation spiked with the individual components of the residue definitions.

### Analytical Methods

10. Freezer storage stability commodities should be analysed using the same validated analytical procedure that was employed in the corresponding MOR studies. However, if a different method of analysis is used, then it should be fully validated as for the analytical method used in the MOR studies.

11. Common moiety methods are unable to measure the stability of each of the individual components of the residue definitions. In addition, a common moiety method in some instances may also detect compounds containing the moiety but which are not included in the residue definitions. This can further mask the decline of residues, which may or may not occur. Consequently, common moiety methods should not normally be used in freezer storage stability studies. However, in exceptional circumstances and with justification, common moiety methods may be used. For example, when the MOR studies have used a common moiety method to quantify the residues, then this method may be used for the determination of the stability of residues during storage.

**Spiking levels**

12. Samples should be spiked at 10x the limit of quantitation (LOQ) of the method for each analyte in order to adequately determine the stability of the residues under storage conditions. This will make it less likely that highly variable recoveries would prevent the determination of the stability of the residues. These are model studies and should be optimised for the demonstrated range of the analytical method. Spiking procedures should be undertaken in the same way as the spiking of the samples in the validation of the analytical methods e.g. for the recovery data. Where this is not possible, then a full rationale/ justification for the applicability of the data should be provided.

13. In instances where no detectable residues are found in field treated commodities, or residue levels are close to the analytical method's LOQ, spiked control commodities should be employed in the freezer storage stability studies rather than incurred residues.

**Sample form**

14. It is preferred that the form of the commodity e.g. homogenate, coarse chop, whole commodity, extract, in a freezer storage stability study should be, as far as possible, the same as that in the corresponding MOR studies. In some cases the freezer storage stability study may need to reflect storage of more than one of the above forms. For example, if MOR trial samples are stored as homogenates for several months, extracted, and then these extracts stored for several weeks prior to final analysis, the freezer storage stability commodities should be handled in the same manner.

15. In some cases MOR samples are stored in a whole state, while the freezer storage stability samples are kept as homogenates in order to ensure that the samples can be spiked uniformly. Provided the residues are found to be stable, such studies will normally be accepted since the use of an homogenate in the freezer storage stability study is likely to represent a worse case versus the use of a whole commodity.

16. If the form of the commodities stored in the freezer stability storage stability study is not as extracts and so does not reflect the storage of analytical extracts of the MOR samples prior to final analysis, the whole study need not be repeated. It would be acceptable to spike extracts of untreated samples, hold them in storage for the same time and under the same conditions as the corresponding extracts in the MOR samples, and then analyse them to determine the stability of residues in the extract. To avoid this additional study, applicants are advised to routinely include the storage of extracts in their freezer storage stability studies unless their standard laboratory practice is to analyse extracts on the same day as they are obtained. Information on the stability of residues in extracts could also be obtained from other studies, e.g. method validation or metabolism studies.

**Storage conditions**

17. It is recognised that MOR samples almost always require transport from the study site to the laboratory prior to placement into storage until residue analysis can be performed. Efforts should be made to keep samples as cold as possible during transport, e.g. packed with dry ice, and to keep the transport

period as short as possible. The freezer storage stability study should then simulate these conditions e.g. temperature used in the laboratory for storage of MOR samples prior to their analysis. Storage temperatures should be -18 °C or lower and commodities kept in the dark to eliminate the possibility of photochemical reactions. For pesticides with known instability, options to reduce this instability in the MOR samples can include storage at lower temperatures, or frozen storage of the extracts in solvent. Furthermore, samples from the MOR studies can be stabilised by the addition of acid or base during homogenisation or by cryogenic milling. Any of these additional steps should also be employed in the freezer storage stability study.

18. Storage conditions should be constantly monitored and recorded to demonstrate maintenance of acceptable storage temperatures. If the storage conditions vary significantly, e.g. due to loss of electrical power, full details should be provided and data from various time points in the study should be considered to determine if integrity of the study has been maintained.

19. As far as possible sample containers should be of the same design and of the same inert composition as in the MOR studies. However, if this is not possible, as long as the pesticide is not volatile, studies will not be rejected solely due to the use of different containers.

**Frequency and duration of sampling**

20. It is advisable that at the beginning of the study adequate aliquots of each commodity should be stored in the freezer to enable sufficient time points to be analysed covering the storage period of the MOR samples. Additional reserve commodities are also recommended in case problems are encountered and repeat analysis is required, or if longer storage periods than anticipated are needed. In all cases, the sampling points should include zero-time to establish and check the residue levels present at the time samples are placed into storage. The minimum number of sampling times will vary depending upon the stability of the residues and the maximum length of the storage period for the MOR samples.

21. Applicants may choose to use just two sampling intervals:- time zero and for example 12 or 24 months. However, this is at the applicant's risk as a rate of decline can not be established and extrapolation beyond the window may not be possible and thus compromise the MOR studies. It is recommended where residues are considered to be stable, typical sampling intervals of 0, 1, 3, 6 and 12 months could be employed but may have to be extended if the MOR samples are stored for longer periods e.g. up to 2 years. In contrast, if relatively rapid decline of residues is suspected, sampling intervals such as 0, 2, 4, 8 and 16 weeks could be chosen. If there is no prior knowledge then the choice of intervals could be a combination of the above.

22. Duplicate samples of every commodity at each time point for all components of the residue definitions need to be analysed. However, if a significant difference (greater than 20%) exists between the results for the duplicate samples from the same time point, judgement should be applied and consideration given to analysing additional samples of the commodity from that time point. Replicate analysis of the individual samples is at the discretion of the applicant. Prior analysis of the commodity, before spiking, is advisable to reveal any interference from other substances or from contamination with the test substances and consequently, in some circumstances, the need to repeat the study.

23. The applicant should ensure that all MOR samples are analysed within the time period for which freezer storage stability for all components of the residue definitions has been demonstrated. However, where this is not the case, it does not necessarily compromise the use of the MOR data. In exceptional cases, extrapolation to time points not covered by the storage intervals may be possible where there is no observable decline. However, the extent of any extrapolation should be discussed with a regulatory authority on a case by case basis.

 

**CROP COMMODITIES TO BE ANALYSED**

24. In the case of studies involving crop commodities, the principles of extrapolation between commodities within specific commodity categories is recommended and the commodity categories are as follows: commodities with high water content; commodities with high acid content; commodities with high oil content; commodities with high protein content; and commodities with high starch content. It is recognised that some commodities can fit into more than one category, but as these are model studies, such commodities have been assigned to the most representative category.

25. If residues are shown to be stable in all commodities studied, a study on one commodity from each of the five commodity categories is acceptable. In such cases, residues in all other commodities (see Annex 1) would be assumed to be stable for the same duration of time under the same storage conditions.

26. If uses are sought in just one of the five commodity categories, then residue freezer storage stability data beyond one representative commodity in that category will be needed (with the exception of the high protein category, which has only one commodity type with respect to this guideline). A study on commodities in the corresponding category is conducted in accordance with the following:

High water content category:
If the stability of test substance in three diverse commodities in this category is confirmed, further examination with other crops that belong to this category is unnecessary.

High oil content category:
If the stability of test substance in two diverse commodities in this category is confirmed, further examination with other crops that belong to this category is unnecessary.

High protein content category:
If the stability of test substance in dry legume / pulses is confirmed, further examination with other commodities that belong to this category is unnecessary.

High starch content category:
If the stability of test substance in two diverse commodities in this category is confirmed, further examination with other commodities that belong to this category is unnecessary.

High acid content category:
If the stability of test substance in two diverse commodities in this category is confirmed, further examination with other commodities that belong to this category is unnecessary.

27. If there is no observed decline of residues across the range of the five different commodity categories then specific freezer storage stability data for processed foods will not be needed. However, if instability is shown after a certain length of storage, the applicant should ensure that any commodities (RAC or processed commodity) are analysed within the demonstrated time period for stable storage.

28. The guidance on representative commodities is directed toward a pesticide that will be applied to all commodity categories. Many pesticides are applied to only a portion of these categories. When use in two or more categories is sought, but residue freezer storage stability data have not been generated on all five categories, the necessary number of representative commodities will depend on the combination of categories and how many crops in each category are to be treated with the pesticide. It is not possible to provide guidance for all the possible combinations of commodities that might be treated. Applicants will need to use judgment as to which representative commodities to use for freezer storage stability studies. One example will be presented here. In the case of a pesticide to be applied to only tree nuts (belonging to

high oil category) and stone fruit (belonging to high water category), freezer storage stability data should be provided on at least one tree nut commodity e.g. walnut, almond and one stone fruit commodity e.g. peach, cherry. If the use of this pesticide were to be expanded later to other high water crops such as leafy vegetables and cucurbits, storage stability data would be needed on these two commodity types consistent with the need for data on three diverse commodities in this category. Thus, freezer storage stability for all commodities in the high water category would be demonstrated.

29. Where freezer storage stability studies are available for representative commodities in each of the five categories, then this will cover the crops containing more than one commodity (for example, cereal crops contain grain, straw and forage). However, if cereal use is the only use supported then data using both grain and forage (to represent the high starch and high water content aspect of the crop) should be covered.

30. Commodities used for the freezer storage stability study could also be obtained from metabolism studies using radiolabelled material. In this case, the residues, according to the residue definitions, should be determined after extraction using the extraction procedure described in the residue analytical method that was employed in the MOR studies or another validated method, coupled with an appropriate radio chemical detection technique for the analyte(s) of interest. In other words, the freezer storage stability data should not be based on simply counting total radioactivity. (NOTE: The discussion in this paragraph is not referring to the residue freezer storage stability data needed to support a metabolism study which is covered elsewhere).

**ANIMAL COMMODITIES TO BE ANALYSED**

31. In the case of studies involving animal commodities e.g. livestock feeding or dermal treatment studies, the following should be chosen depending on the animal:

- muscle – e.g. cattle and/or poultry
- liver – e.g. cattle and/or poultry
- milk
- eggs

32. If residues are shown to be stable in all animal commodities studied, a study with each of the above animal commodities is acceptable. In such cases, the residues in all other animal commodities would be assumed to be stable for the same duration of time under the same storage conditions. Applicants should ensure that all MOR samples in animal commodities are analysed within the shortest period for which stability has been shown in the above animal commodities.

**FURTHER DATA CONSIDERATIONS**

33. During freezer storage stability studies freshly spiked control samples should be analysed at each of the time points when aged/stored freezer storage stability samples are removed from storage for analysis in order to demonstrate good analytical procedural recovery at the time of analysis. This will allow appropriate interpretation with regard to possible residue decline in case analytical recoveries are variable across the different time intervals. As such, if procedural recoveries are close to 100%, but stored commodities give low recoveries, then this would suggest decline on storage. However, if recoveries for both stored commodities and procedural recoveries are similar and both low then this suggests that there may not be a decline on storage.

34. Data from the freezer storage stability study indicating a limited decline does not necessarily compromise the use of the MOR data. It is not appropriate to place a limit of decline and state whether this

is acceptable or not. Any suggested decline and significance of such will be dependent upon a number of factors which can include: the rate of decline and whether a plateau is reached (equilibrium); the risk assessment; the variability of procedural recoveries as well as how close the tested commodities in the freezer storage stability studies are to the crop used in the MOR trials. Overall, acceptability of studies will be considered on a case-by-case basis taking into account the above factors.

35. In the case of measurable decline in residues, provided sufficient data points are available to construct a suitable graph, the principle of interpolation could be applied to determine the decline at any point in time. Any decline should take into account the uncorrected residues in stored commodities and procedural recoveries. If a decline is still indicated, then the applicant should ensure that all MOR samples are analysed within the time window for which the period of stability of the residues of interest has been demonstrated in the commodity.

## CONSIDERATIONS FOR DATA REPORTING

36. Reports on freezer storage stability studies should include a detailed description of the commodities that were stored (whether raw or processed); the test compound(s); the experimental design and storage conditions e.g., freezer temperature, length of storage, type of containers, residue method(s) and instrumentation; freezer storage stability results and reporting of the data; statistical analysis; and quality control measures/precautions taken to ensure the validity of these operations, including the dates for each step above. It is important for applicants to describe how MOR samples are prepared e.g. coarsely chopped, homogenized, water or buffer added before being placed in storage.

37. The values for individual commodity recoveries, as opposed to just reporting a mean, should be reported for all commodities and procedural recoveries in cases where multiple commodities have been analysed at a given time point. Results should be presented as absolute values in mg/kg and not adjusted by recovery, as well as % of nominal spike value. Individual procedural recoveries as well as the mean should also be given for all samples including the first one at time zero. The time zero sample is the same as the initial procedural recovery. A suggested tabular format for reporting the results plus an example table with some additional observational notes can be found in Annex 2.

**Materials**

(i) Test substance

(A) If spiking of commodities was used, describe the test substance(s) (chemical/ common/ experimental/CAS names), including the determination/check of the purity of the test compounds (all components of the residue definitions) and preparation of standard solutions. Provide certificates of analysis.

(B) If incurred residue samples were used, confirm the presence and the amount of all components of the residue definitions in the sample at "zero-time" (defined as the beginning of the freezer storage stability testing). First analysis for samples with incurred residues should be carried out as soon after harvest as possible.

(C) Any and all additional information the applicant considers appropriate and relevant to provide a complete and thorough description and identification of the test substances used in freezer storage stability testing.

(ii) Test commodity

(A) Identification of the individual commodities to include crop, type, variety, botanical name where available.

(B) The development stages, general condition e.g. immature/mature, green/ripe, fresh/dry, and sizes of the commodities.

(C) Describe the sampling procedure used for all commodities prior to freezer storage stability testing and for all time intervals e.g. trimming, cleaning, or other means of residue removal, compositing, sub sampling, chopping, and extraction.

(D) For incurred samples, describe source of MOR samples, field trial identification number, control or incurred residue sample, coding and labelling information etc. These should be the same as, or cross-referenced to, the sample coding/labelling assigned at harvest.

(E) Any and all additional information the applicant considers appropriate and relevant to provide a complete and thorough description of the commodities.

**Methods**

(iii) Experimental design e.g. number of test commodities, number of test substances, number and magnitude of test levels, number of replicate samples per test compound per test level, number of sampling intervals.

(iv) Test procedures

(A) Spiking procedure, if used: Detail the manner in which the test compound(s) was/were introduced to the test substrates.

(B) Storage conditions: Temperature, lighting, container types/size, commodity form (extract/macerate/etc.), sample sizes/weight(s), duration, etc. should be provided.

(C) Sampling: Describe the sampling procedure at zero-time and at regular intervals thereafter.

(D) Dates of sample preparation (maceration/extraction/etc.), "spiking" or determining the type/amount of incurred residue (zero-time), periodic sampling intervals, end of freezer storage, and residue analyses should be provided.

(E) Methods of residue analysis: The following data/information should be included within the freezer storage stability report or the full details/report of the analytical method should be included as an appendix to the freezer storage stability report.

(1) Title/designation/date and source of the analytical method should be submitted. If the method used in the freezer storage stability study is the same as used in the MOR studies, then a cross reference to elsewhere in the submission will suffice.

(2) Discuss any deviations in reagents, procedures, instrumentation, operating parameters, etc., from the analytical methods used for residue analysis of field trial samples or processed commodities.

(3) Detail the principles and stepwise procedures including extraction, clean-up, derivatisation, determination, as well as any modifications made, chemical species determined, confirmatory techniques used, etc.

(4) Instrumentation and operating parameters such as make/model, type/specificity of detectors, columns (packing materials, size), carrier gases, flow rates, temperatures, voltage, limit of quantitation and sensitivity, calibration procedures, etc. should be provided.

(5) Reagents or procedural steps requiring special precautions to avoid safety or health hazards should be explained.

(6) Procedures for calculating residue levels and percent recoveries should be reported.

(7) Any other additional information the applicant considers appropriate and relevant to provide a thorough description of the analytical methodology and the means of calculating the residue results should be provided.

**Results/Discussion**

(v) Residue results: Raw data, details of any necessary dilutions, peak heights/areas, procedural recoveries (%), formula(e)/standard curves used, sample residue levels (mg/kg), recovery, percent decline related to zero day (if observed) vs. length of storage, appropriateness of length of freezer storage stability study, etc. should be provided.

(vi) Statistical treatments: Describe tests applied to the raw data.

(vii) Other: Any additional information the applicant considers appropriate and relevant to provide a complete and thorough description of the results should be provided.

**Conclusion**

(viii) Discuss conclusions that may be drawn regarding the stability of the test compound(s) in the test commodities as a function of storage time, and the use of the data for interpolation and extrapolation purposes.

**Certification**

(ix) Certification of authenticity by the Study Director (including signature, typed name, title, affiliation, address, telephone number, date) should be provided.

(x) GLP compliance statement by designated official (including signature, typed name, title, affiliation, address, telephone number and date) should be provided.

**Tables/Figures**

(xi) Tables of data from freezer storage stability testing and a summary table of residue levels in stored samples as a function of commodity and storage time should be submitted.

(xii) Relevant graphs, figures, flowcharts, etc. should be included.

**Appendices**

(xiii) Representative chromatograms should be provided.

(xiv) Reprints of methods and other studies cited unless physically located elsewhere in the overall data submission, in which case cross-referencing will suffice, should be submitted.

(xv) Other: Include any relevant material not fitting in any of the other sections of this report.

**LITERATURE**

(1) U.S. Environmental Protection Agency. (1996). OPPTS 860.1380 ,1996, Residue Chemistry.

(2) EU (1997).Guidance document Appendix H Storage Stability of Residues Samples 7032/VI/95 rev.5 22/7/97.

(3) United Nations Food and Agricultural Organization (FAO).(1994). Stability of Pesticide Residues in Stored Analytical Samples. 1994 draft prepared by Codex Committee on Pesticide Residues Working Group on Methods of Analysis and Sampling.

(4) United Nations Food and Agricultural Organization (FAO) (1986). Guidelines on Pesticide ResidueTrials to Provide Data for the Registration of Pesticides and the Establishment of Maximum Residue Limits - Part 1 – Crops and Crop Products.

(5) Canadian Pest Management Regulatory Agency. (1998). Regulatory Directive 98-02. Residue Chemistry Guidelines.

## ANNEX 1

**Commodity Categories for Purposes of Stability of Pesticide Residues in Stored Crop Commodities**

When choosing representative commodities for study to extrapolate to other commodities within the same category, it will be necessary to exercise judgement, e.g. it would be inappropriate to select spices or hops alone to study to be representative of a range of oil content commodities.

| Commodity Categories | Commodities included in this category | Typical representative commodities |
|---|---|---|
| High water content | Pome fruit<br>Stone fruit<br>Bulb vegetables<br>Fruiting vegetables/cucurbits<br>Brassica vegetables<br>Leafy vegetables and fresh herbs<br>Stem and stalk vegetables<br>Forage/fodder crops<br>Fresh legume vegetables<br><br>Leaves of root and tuber vegetables<br>Sugar cane<br>Fresh green tea<br>Fungi | Apples, pears<br>Apricots, cherries, peaches<br>Bulb onion<br>Tomatoes, peppers, cucumber, melon<br>Cauliflower, Brussels sprout, cabbage<br>Lettuce, spinach<br>Leek, celery, asparagus<br>Wheat and barley forage, alfalfa,<br>Fresh peas with pods, petit pois, mange tout, broad bean, runner bean, dwarf French bean<br>Sugar beet and fodder beet tops |
| High oil content | Tree nuts<br>Oilseeds<br>Olives<br>Avocados<br>Hops<br>Cacao beans<br>Coffee beans<br>Spices | Walnut, hazelnut, chestnut<br>Oilseed rape, sunflower, cotton, soybean, peanut |
| High protein content | Dry legume vegetables/Pulses | Field bean, dried broad bean, dried haricot bean (yellow, white/navy, brown, speckled) |
| High starch content | Cereal grain<br>Roots of root and tuber vegetables<br>Starchy root crops | Wheat, rye, barley and oat grain<br>Sugar beet and fodder beet roots, carrot<br>Potato, sweet potato |
| High acid content | Citrus fruit<br>Berries<br>Currants<br>Grapes<br>Kiwifruit<br>Pineapple<br>Rhubarb | Lemon, mandarin, tangerine, orange<br>Strawberry, blueberry, raspberry<br>Black currant, red currant, white currant |

**IMPORTANT NOTE:** The above list of commodities is not a comprehensive list of commodities/ matrices and other commodities may be used. Applicants should consult regulatory authorities for advice on the use of other commodities.

## ANNEX 2

**Reporting example for a spike level of 0.1 mg/kg**

| Commodity | Analyte | Storage Period (months) | Residue Level in Freezer Storage Stability Sample (mg/kg) | Residue Level in Freezer Storage Stability Sample (% of nominal spiking level) (range plus mean) | Procedural Recovery for Freshly Spiked Control Sample (%) |
|---|---|---|---|---|---|
| Wheat grain | metabolite a | 0 | 0.101 0.121 | 101, 121 (111) | 101, 121 |
| Wheat grain | metabolite a | 3 | 0.122 0.115 | 122, 115 (119) | 114, 90, 95 |
| Wheat grain | metabolite a | 6 | 0.104 0.116 | 104, 116 (110) | 99, 102, 95 |
| Wheat grain | metabolite a | 12 | 0.089 0.091 | 89, 91 (90) | 98, 100, 103 |
| Wheat grain | metabolite a | 18 | 0.080 0.083 | 80, 83 (82) | 77, 82, 78 |
| Wheat grain | metabolite a | 24 | 0.072 0.069 | 72, 69 (71)* | 75, 80, 79 |
| Apple | metabolite b | 0 | 0.103 0.096 | 103, 96 (100) | 103, 96 |
| Apple | metabolite b | 3 | 0.110 0.102 | 110, 102 (107) | 112, 98, 95 |
| Apple | metabolite b | 6 | 0.096 0.098 | 96, 98 (97) | 100, 103, 95 |
| Apple | metabolite b | 12 | 0.095 0.107 | 95, 107 (101) | 97, 62, 103 |
| Apple | metabolite b | 18 | 0.083 0.081 | 83, 81 (82) | 104, 95, 99 |
| Apple | metabolite b | 24 | 0.062 0.064 | 62, 64 (63) ** | 98, 103, 92 |

* Although the level of residue seems to have declined by nearly 30%, it is considered that the samples are sufficiently stable over 24 months frozen storage in wheat grain, as all of the procedural recoveries at the latter time-points were consistently lower, than for the earlier time-points (although it is considered that some decline in stability has been observed).

** Conversely residues of metabolite b are only regarded as sufficiently stable in apple up to a period of 18 months frozen storage.