CODEX ALIMENTARIUS COMMISSION

Food and Agriculture Organization of the United Nations

World Health Organization

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CODEX COMMITTEE ON PESTICIDE RESIDUES

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DISCUSSION PAPER ON GUIDANCE FOR MONITORING THE PURITY AND STABILITY OF REFERENCE MATERIAL OF MULTI-CLASS PESTICIDES DURING PROLONGED STORAGE

(Prepared by the Electronic Working Group chaired by India
and co-chaired by Argentina and Iran)

Codex members and observers wishing to submit comments on the recommendations in paragraph 15 of Appendix I should do so as instructed in CL 2023/38-PR available on the Codex webpage[1]

INTRODUCTION

1. Reference materials (RMs) of pesticides are the yardsticks for the analytical measurements required by the analytical laboratories for accurate analysis of pesticide residues in food commodities and environmental samples. The purity of RMs is determined and certified by the Reference Material Producer (RMP) in accordance with international guidelines. Limited shelf life and high recurring cost are the major limiting factors of RMs to be used for qualitative and quantitative determination of pesticide residues in foods.

2. The Codex Committee on Pesticide Residues (CCPR51, 2019) considered a request related to the shelf-life of certified reference materials (CRMs) raised by some delegations as follows:

   - CRMs were used for many purposes e.g., Good Agricultural Practices (GAP), supervised field trial data, monitoring of import/export samples, etc.
   - Most of the CRMs remained stable after their expiry period mentioned in their Certificate of Analysis (CoA).
   - The limitation of the use of CRMs after the expiry date led to recurring high costs for laboratories, consideration should therefore be given to including guidance on monitoring of purity of CRMs of multi-class pesticides during prolonged storage.

3. CCPR51 agreed to request Argentina and India to prepare a discussion paper regarding monitoring of purity of CRMs of multi-class pesticides during prolonged storage for consideration at CCPR52.[2]

4. At CCPR52 (2021), India on behalf of Argentina, introduced the item and reminded CCPR of the background for the work, the work process followed in the development of the discussion paper and key issues discussed in the paper. The Delegation informed CCPR that further work was needed on this topic and the Committee agreed to establish an electronic working group (EWG) led by India to further develop the discussion paper for consideration by CCPR53.[3]

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1 Codex webpage/Circular Letters: http://www.fao.org/fao-who-codexalimentarius/resources/circular-letters/en/.
Codex webpage/CCCF/Circular Letters: https://www.fao.org/fao-who-codexalimentarius/committees/committee/related-circular-letters/tr/?committee=CCPR

2 REP19/PR51, paras. 182-184 & 186

3 REP21/PR52, paras. 198-201

5. At CCPR53 (2022), India, as Chair of the EWG and on behalf of the co-Chairs Iran and Argentina, introduced the item and recalled the request regarding limitation of the use of CRMs after the expiry date leading to high recurring costs for laboratories and trade disruption, and thus the need for harmonized guidance on monitoring of purity of RMs of multiclass pesticides during prolonged storage. Such guidance would enable the use of RMs after the expiry date when verification was performed as per the international guidance provided by Codex. Use of expired CRMs with verified purity would have economic impact by saving the purchasing cost of fresh CRMs especially by developing countries.

6. CCPR53 considered the new work proposal and noted support for the work. In response to the suggestions made by some members to consult with CCMAS, the Codex Secretariat clarified that since the proposal under discussion was specific for pesticides, the work fell within the remit of CCPR. The JMPR Secretariat commented that the use of CRMs for pesticides was important for the establishment and implementation of CXLs and achieving the goals of the Codex, i.e., protecting consumer health and facilitating trade.

7. Following the detailed deliberations on the proposed work, CCPR53 agreed to[4]:

   i. re-establish the EWG, chaired by India and co-chaired by Argentina and Iran, working in English and Spanish, to refine the discussion paper and proposal for new work taking into account comments made at the Session and submitted in writing to the Session and to build on and explain more clearly the rationale for the new work.

   ii. encourage all members and observers to participate in the EWG in particular those delegations who had made interventions during the Session, in particular, China, Japan, Singapore, Egypt and IFT to actively participate in the EWG to facilitate the consideration of and decision-making on this matter at CCPR54.

WORK PROCESS AND KEY POINTS OF DISCUSSION

8. Based on the comments received from the members and observers during CCPR53, the discussion paper was refined expanding the scope of the paper to reference materials (RMs) having known purity specified by the RMP in the certificate of analysis (CoA) and inclusion of acceptability criteria and definitions. The paper was circulated among the EWG members on the Codex online forum for comments. In the first round, comments were received from Mexico, Germany, USA, Uruguay, and Institute of Food Technologists (IFT).

9. The EWG members, in general, supported the development of the discussion paper. Most of the members emphasized on the removal of the proficiency testing for evaluation of RM purity, expansion of the acceptability criteria of RM purity to 5% and inclusion of stability and purity definitions. The discussion paper was revised and uploaded by the Chair inviting second round of comments from the EWG members and observers. In the second round, Thailand, Chile, China, United States of America (USA), Canada, and Mauritius provided their comments on the forum. As per the comments, the discussion paper has been modified including the revised proposal for new work, reference to ISO Guide 80, inclusion of quality control materials (QCMs) in definitions, and suggested changes in analytical protocol.

SUMMARY OF THE INFORMATION PRESENTED IN THE PAPER

10. The aim of this discussion paper (Appendix I) is to present the background information related to the work done on monitoring the purity and stability of RMs, summary of work done by national/international agencies, knowledge gaps, challenges, and approaches. The Proposal for new work on guidance for monitoring the stability of reference material purity of pesticides during prolonged storage is provided in Appendix II. The outline of proposed guidance for monitoring the stability of reference material purity of pesticides during prolonged storage is provided in Appendix III. The definitions of various terminologies used in the discussion paper have been included in and Annex to Appendix III. A list of members and observers of the EWG is included as Appendix IV.

4 REP22/PR53, paras. 235-242

APPENDIX I

DISCUSSION PAPER ON
GUIDANCE FOR MONITORING THE STABILITY OF
REFERENCE MATERIAL PURITY OF PESTICIDES DURING PROLONGED STORAGE
(For consideration by CCPR)

BACKGROUND

1. Reference materials (RMs) of multi-class pesticides are routinely utilized in the determination of pesticide residues, method validation, quality control and assurance, proficiency testing, inter-laboratory comparison, assigning values to specified properties, etc by the pesticide residue analysis laboratories. RMs are the yardstick for the analysis of pesticide residues in various food commodities and environmental samples.

2. According to European Reference Materials (ERM) Application Note 7, certificates of RMs have limited expiry dates. However, there exists the possibility of prolonging the validity period of the RMs, provided the users collect the requisite information related to the stability of the RMs. Their stability can be assessed by creating quality control charts, comparing the certified values of expired RMs with fresh RMs, and through satisfactory performance in proficiency testing (Linsinger, 2019).

3. Roelandts and Gladney (1998) had mentioned that expired RMs with established consensus values can be used to demonstrate repeatability in a measurement system.

4. The long-term intra-lab and inter-lab validation experiments conducted at the Pesticide Residue Laboratory (PRL) at ICAR-Indian Agricultural Research Institute, New Delhi, India to assess the purity of standards and stock solutions of 89 RMs of multi-class pesticides stored at -25°C beyond their expiry revealed that when RMs were stored in conditions better than those recommended by RMPs for longer period of 3 years, most of the RMs remained optimally stable with respect to their observed purity (Sharma et. al. 2020). The study reported that more than 96 % of the RMs remained optimally stable with respect to their observed purity even after their expiry date as per CoA.

5. The performance of the valid and expired RMs was also evaluated through 44 z-scores for 15 expired and 29 valid RMs obtained in 14 international and national PT programs conducted by the international PT sample providers namely EUPT-European Union Referral Laboratory, Spain; FAPAS-Food Analysis Performance Assessment Scheme, UK; and APLAC- Asia Pacific Laboratory Accreditation Cooperation, Australia. Inter-laboratory comparison of 6 randomly selected expired and valid RMs was tested by liquid chromatography-mass spectroscopy (LC-MS/MS) at three different ISO 17025 accredited laboratories and the average % deviation between % purity ranged from -2.35 to +0.95% (Sharma et al. 2020).

6. Dorweiler et al. (2016) conducted simulated accelerated ageing study of multi-component RM mixes of 528 pesticides and their metabolites/degradation products in solution phase at stressed temperature of 50°C, and the samples analyzed at 0, 1.5, 3, and 6- day time period which simulated 0-, 6-, 12- and 24-months storage time. Study indicated that 65% of the RMs remained stable up to 24 months, 19% exhibited borderline stability, and about 16% were significantly unstable. Thus, in the solution phase, most of the RMs retained their stability up to 24 months.

7. The long-term intra-laboratory and inter-laboratory validation experiments were also performed at the NVWA laboratory, Amsterdam, the Netherlands to assess the storage stability of standards and stock solutions of LC-pesticides (de Kok et al. PO006pdf, 2019). The stability of RM standards was assessed by continuously measuring old against new stock and calibration-mixture solutions, on a long-term basis. The study revealed that the stability of most of the pesticide reference standards was up to 15 years, and in stock solutions (in toluene or MeOH), at -18 °C was up to 10 years.

8. Proficiency testing (PT) is recommended for testing and calibration laboratories in the International Standards Organization (ISO)/International Electrotechnical Commission (IEC) 17025:2017. International standard ISO/IEC 13528:2015 is complementary to ISO/IEC 17043 providing detailed guidance on the use of statistical methods in PT. If correct/accurate results are obtained during PT programs, it may be inferred that the RMs used in the program have maintained their purity.

SUMMARY OF WORK UNDERTAKEN BY OTHER INTERNATIONAL ORGANIZATIONS

9. The requirements of the purity of RMs are highlighted in various International Organization for Standardization (ISO) standards. ISO Guide 30 (2015) relates to terms and definitions for reference materials, ISO Guide 31 (2015) provides the contents of certificates, labels and accompanying documentation of reference materials, ISO Guide 33 (2015) emphasizes on the good practice in using reference materials, and the ISO Guide 35 (2017) provides guidance for characterization and assessment of homogeneity and stability. ISO Guide 34 (2009) and ISO 17034:2016 pertain to general requirements for the competence of reference material producers. The certification of reference materials is carried out in accordance with the requirements of ISO Guides which are published by ISO/REMCO (Reference Material Committee of the International Standardization Organization) to produce and certify reference materials. The ISO/IEC 17025 measurement quality system emphasizes the use of reference materials from RMPs meeting the requirements of ISO 17034. The ISO Guide 80 (2014) provides guidance for the in-house preparation of Quality control materials (QCMs) which are a type of RM used for internal quality control purposes in a laboratory. The SANTE guidelines provides analytical quality control and method validation procedures for pesticide residues analysis in food and feed.

KNOWLEDGE GAPS AND CHALLENGES

10. Limited shelf life, diminishing purity with time, and high recurring cost are the major limiting factors of RMs to be used for determination of pesticide residues in foods. The date of expiry recommended in the certificate of analysis (CoA) limits the use of RMs after their expiry. This leads to high recurring cost of procuring new RMs by the laboratories working in the field of multi-pesticide residue analysis. The disposal of the expired RMs is also a major environmental challenge globally.

11. The non-availability of standardised analytical protocols for monitoring the stability of RM purity before and after expiry is one of the major knowledge gaps in the extended use of pesticide RMs for pesticide residue analysis beyond their expiry. There is no information available about the standardized procedures.

12. None of the international agencies or inter-governmental bodies or country has provided any guidance on the use of RMs beyond expiry.

APPROACHES

13. The standard analytical protocols need to be developed for monitoring the stability of RM purity before and after expiry. The criteria for acceptable purity of the RMs after expiry will provide guidance to the pesticide residue analysis laboratories for extended use of RMs with acceptable purity beyond their expiry date specified in the certificate of analysis (CoA).

CONCLUSIONS

14. The discussion paper provides the justification on the need for the harmonization of concepts and criteria to monitor the stability of RM purity for continued use beyond their expiry date. The use of expired RMs with verified purity in the measurement systems will not only ensure continuity of their use in the laboratories but also have economic impact by saving the purchasing cost of fresh RMs and overcome the challenges of the disposal of the expired RMs in the environment.

RECOMMENDATIONS

15. The EWG recommends CCPR54 to:

(i) consider the proposal for new work on monitoring the stability of reference material purity of pesticides during prolonged storage (Appendix II) based on the information provided in the discussion paper (Appendix I);

(ii) review the outline of the proposed new work (Appendix III) to provide general guidance for the further development of the document in the Electronic Working Group should there is agreement to proceed with the new work; and if so,

(iii) establish an EWG to prepare guidance on monitoring the stability of reference material purity of pesticides during prolonged storage based on the outline provided in Appendix III for consideration by CCPR55 (2024).

REFERENCES

CCPR51-April, 2019, Report of the 51st Session of the Codex Committee on Pesticide Residues, Haikou, P.R. China, 8 - 13 April, 2019.

CCPR53-July, 2022, Report of the 53rd Session of the Codex Committee on Pesticide Residues (Virtual),4-8 July, 2022.

de Kok, A., de Kroon, M. and Scholten, J. (PO 006 pdf, 2019). Stability of pesticides reference standards and stock solutions Part 2. LC-pesticides NVWA - Netherlands Food and Consumer Product Safety Authority, Laboratory of Food and Feed Safety-Chemistry Laboratory, National Reference Laboratory (NRL) for Pesticide Residues in Food and Feed, Wageningen, The Netherlands.

Dorweiler, K. J., Gurav, J. N., Walbridge, J. S., Ghatge, V. S., and Savant, R. H. (2016). Determination of stability from multicomponent pesticide mixes. *J. Agric. Food Chem.* 64, 6108-6124. DOI: 10.1021/acs.jafc.5b05681

ISO 13528:2015, Statistical methods for use in proficiency testing by inter-laboratory comparison, International Organization for Standardization (ISO), Geneva, 2015, pp.89

ISO Guide 30:2015, Reference materials - Selected terms and definitions, International Organization for Standardization (ISO), Geneva, 2015.

ISO Guide 31:2015, Reference materials—contents of certificates, labels and accompanying documentation, International Organization for Standardization (ISO), Geneva, Geneva, 2015.

ISO Guide 33:2015, Reference materials - Good practice in using reference materials, International Organization for Standardization (ISO), Geneva, 2015.

ISO Guide 34: 2009, General requirements for the competence of reference material producers, International Organization for Standardization (ISO), Geneva, 2009.

ISO Guide 35:2017, Reference materials—guidance for characterization and assessment of homogeneity and stability, International Organization for Standardization (ISO), Geneva, 2017.

ISO Guide 80:2014, Guidance for the in-house preparation of quality control materials (QCMs), International Organization for Standardization (ISO), Geneva, 2014.

ISO 17034:2016, General requirements for the competence of reference material producers, International Organization for Standardization (ISO), Geneva, 2016.

ISO/IEC 17025:2017, General Requirements for the Competence of Calibration and Testing Laboratories, International Organization for Standardization (ISO), Geneva, Geneva, 2017.

ISO/IEC 17043:2010, Conformity assessment - General requirements for proficiency testing, International Organization for Standardization (ISO), Geneva, 2010.

Linsinger, T. (2019). European Reference Materials Application Note 7: Prolonging the validity of reference material certificates. European Communities. pp 1-2

Roelandts, I. and Gladney, E. S. (1998). Consensus values for NIST biological and environmental standard reference materials. *Fresenius J. Anal. Chem.* 360(5):327-338.

SANTE (2022). Analytical quality control and Method validation procedures for Pesticide residues analysis in food and feed. SANTE/11312/2021, Implemented by 01/01/ 2022, European Commission Directorate General for Health and Food Safety.1-57.

Sharma, K. K., Tripathy, V., Gautam, R., Gupta, R., Tayade, A., Sharma, K., Yadav, R., Shukla, P., Devi, S., Pandey, P., Singh, G., Kalra, S., Walia, S. (2020). Monitoring of purity of CRMs of multiclass pesticides during prolonged storage before and after expiration. Accreditation Qual. Assur., 25 (10), 89-97. 10.1007/s00769-019-01411-w.

APPENDIX II

PROJECT DOCUMENT

**PROPOSAL FOR NEW WORK ON
GUIDANCE FOR MONITORING THE STABILITY OF
REFERENCE MATERIAL PURITY OF PESTICIDES DURING PROLONGED STORAGE
(For consideration by CCPR)**

1) Purpose and scope of new work

Pesticide residues in food commodities have become a worldwide agricultural trade-concern which has led to enforcement of strict pesticide regulations. Analyses of multi-class pesticides in the food chain with reliable measurement and accuracy require RMs of known chemical purity to ensure food safety, food quality and the safe environment. Purity, characteristic of a reference material, when stored under specified conditions, refers to the measurement of the quantity of a prevalent component of a substance when only that component is present. In a measurement quality system, RMs with certified purity are required to determine the amount of reference chemical in the sample.

More than 1200 pesticides are used to control the pests on different food commodities. For quantitative determination of the residues of these pesticides on food matrices, their specific RMs are required by the testing laboratories. However, limited shelf life, short expiry date, and high recurring cost of RMs act as major impediments for pesticide residue analysis.

These problems are magnified for multi-pesticide residue analysis laboratories situated in developing countries as they are required to allocate a large part of their funds to the frequent procurement of expensive RMs whose use is restricted by the date of expiry recommended in their CoAs.

Further, due to supply chain constraints, some laboratories receive RMs close to their expiration date as per the CoA. In such situations the laboratories are forced to buy new standards and prepare new stock solutions more frequently than necessary. This leads to insurmountable extra work and increased laboratory costs, especially for compounds for which stability is normally not questionable. Additionally, shipping of RMs by the suppliers to laboratories increase the acquisition time for procurement creating hurdles in sustainable pesticide residue control program. Many times, countries cannot afford frequent purchase of high-cost RMs for their pesticide residue and food safety work. Many RMs stay stable even after the date of expiry mentioned in the CoA and continue to retain their valid purity as per CoA. RMs are therefore entitled for continuous use in the laboratories even after expiry date as valid RMs as long as they meet the purity requirements.

The proposed guidelines on monitoring the stability of purity of RMs will provide guidance to the pesticide residues analysis laboratories for extended use of RMs with acceptable purity beyond their expiry date. These guidelines will be applicable to RMs of multi-class pesticides of known purity specified by the RMP in the certificate of analysis (CoA).

2) Relevance and timeliness of the work

The accurate determination of pesticide residues in food commodities is required for food safety, fixation of MRLs for pesticides, overcoming barriers due to pesticide residues in traded commodities and various other applications. RMs with specified purity are required for purity assessment and accurate qualitative and quantitative analysis (trueness and/or precision) of pesticide active ingredient(s) in technical materials and formulations, stock solutions, working solutions, and for the analysis of pesticide residues in food commodities and in soil and water samples.

Qualitative and quantitative determination of pesticide residues in food can be achieved by using high quality RMs, validated analytical protocols, comprehensive quality systems, and competent staff engaged in operating the analytical equipment. RMs that retain their purity even after their expiry period, may be continued to be used after verification of their purity as specified in the CoA.

At present no guidelines on extending the validity of RMs beyond the expiry period are provided by regulatory agencies of different countries.

The proposed work on guidance for monitoring the stability of RM purity of multi-class pesticides before and after expiry for extended use of RMs is thus relevant and timely for consideration by the Codex Committee on Pesticide Residues (CCPR).

3) Main aspects to be covered

The central objective is to use the RMs beyond their specified expiry dates for pesticide residue analysis in food and environment samples. The main aspect to be covered is to develop comprehensive harmonized guidelines which enable the laboratories to monitor the stability of RM purity of pesticides during prolonged storage (before and after expiry). If the purity of the RMs is found acceptable, their use as RMs after expiry may be continued to be allowed.

4) Assessment against the *Criteria for the Establishment of Work Priorities*

4.1 General criterion

General criterion of the proposed new work is to verify the purity of RMs as specified by RMP before and after expiration by standardized analytical protocols so that such materials that retain their purity as per the CoA even after expiry are continued to be used as valid RMs.

4.2 Criteria applicable to general subjects

4.2.1 Scope of work and establishment of priorities between the various sections of the work

The CCPR recognizes the significance of RMs in the analysis of pesticide residues in food commodities and in the soil and aquatic environment. However, the lack of data on purity of RMs during prolonged storage, and absence of SOPs for their analysis prevent their use beyond the expiry period. Scope of the work shall therefore be prioritized stepwise as (i) developing SOP for monitoring the purity of the RMs and, (ii) determining their purity at different time intervals within the expiry period, and beyond the expiry period, (iii) ascertaining whether the purity of the RMs is acceptable for its use beyond the expiry, and (iv) developing guidelines for use of RMs beyond their expiry date by laboratories.

4.2.2 Amenability of the subject of the proposal to standardization

The expiry period of the pesticide RM is determined based on their type, class, chemical structure, and storage conditions like temperature, humidity etc. Pesticides are used globally, and these guidelines would be applicable to all the laboratories with varying levels of technology. There are certain European Reference Materials (ERM) Application Notes that describe some practical aspects associated with the handling and use of RMs. The proposal is thus considered amenable to standardization.

4.2.3 Consideration of the global magnitude of the problem or issue

Since pesticides are used globally, the development of standard analytical protocols for monitoring the purity of RM of pesticides for extended use beyond their expiry is of global relevance to ensure food safety and fair-trade practices for agricultural commodities moving in international trade.

No impediment to international trade is foreseen by having an international agreed guidance on the use of verified expired RMs in pesticide residue analysis in exportable food commodities.

5. The relevance to the Codex strategic objectives

The Codex Strategic Plan 2020-2025 underpins the high priority that continues to be placed on food safety and quality by FAO and WHO and guides the Codex Alimentarius Commission in carrying out its responsibilities to fulfil the mandate of protecting consumer health and ensuring fair practices in the food trade. The use of RMs for pesticides is important for the establishment and implementation of Codex maximum residue limits (CXLs) and achieving the goals of the Codex. The development of guidance on monitoring the purity of RM of pesticides during prolonged storage is relevant to the Codex strategic objectives.

6. Information on the relation between the proposal and other existing Codex documents as well as other ongoing work

No guidance document related to monitoring the stability of purity of RMs during prolonged storage is either available or being currently considered by the Codex.

7. Identification of any requirement for and availability of expert scientific advice

No provision of scientific advice is required for the development of the guidelines.

8. Identification of any need for technical input to the standard from external bodies

For the elaboration of this document, the advice from FAO, WHO and the JMPR Secretariat will be taken as and when required. Other documents issued by international organizations such as the relevant SANTE, ISO guidelines and research reports in literature have been used as a reference to develop the guidance document.

9. The proposed timeline for completion of the new work, including the start date and the proposed date for adoption by the Commission

Subject to approval by the Codex Alimentarius Commission, the Guidelines will be considered at CCPR54 (2023) and finalized for adoption by CAC in 2026 or earlier.

APPENDIX III

PROPOSED GUIDANCE FOR MONITORING THE STABILITY OF REFERENCE MATERIAL PURITY OF PESTICIDES DURING PROLONGED STORAGE
–Outline–
(For consideration by CCPR)

OBJECTIVE

1. The purpose of these guidelines is to furnish a framework which would assist the laboratories in monitoring the stability of reference material (RM) purity of pesticides during prolonged storage and identify expired RMs with continued purity. It will enable the laboratories involved in the analysis of pesticide residues in food commodities and in the environmental samples to overcome the shortcomings associated with RMs and use them beyond their expiry dates mentioned in the Certificate of Analysis (CoA).

2. This document is applicable to solid/liquid RMs of pesticides with purity specified in the CoA issued by a reference material producer (RMP) conforming to ISO 17034. It covers the storage conditions that should be maintained, and quantitative measurements that should be performed to monitor the purity of RMs before and beyond their expiration period.

STORAGE CONDITIONS FOR REFERENCE MATERIALS

3. The storage conditions of RMs are specified by RMPs in the CoA as these are susceptible to degradation at high temperature and other environmental factors.

4. If a laboratory maintains the RMs at storage conditions better than those recommended by the RMPs (i.e., temperature lower than recommended without exposure to light and moisture), the rate of degradation of the RMs is significantly minimized. Under such conditions, the expiry date as recommended by the RMPs may be extended as appropriate for a RM by a date allowing for storage up to 10 years or as long as the certified property values mentioned in the certificate of analysis (CoA) hold good (≤ ±10%) (SANTE[1], 2022). Another study revealed the stability of pesticide reference standard up to 15 years, and in stock solution up to 10 years (de Kok et al. PO006pdf, 2019).

5. To avoid the degradation of RMs, the vials must be placed in airtight capped tube/sealed pouch and immediately stored in the refrigerator at ≤-25^{o}C (Sharma et al. 2020).

ANALYTICAL PROTOCOL FOR DETERMINING THE PURITY OF REFERENCE MATERIALS

6. At the time of procurement, the purity of the RMs to be determined under analytical conditions as mentioned in CoA by using the recommended or any appropriate analytical protocols (HPLC[2]-DAD[3]/HPLC-UV[4]/GC[5]-FID[6]/GC-ECD[7]/LC[8]-MS[9]/GC-MS and other commonly used detectors) in terms of retention time (RT in minutes) and percent peak area, by injecting a solution of suitable concentration prepared in an organic solvent.

7. The purity of the RMs during prolonged storage should be monitored periodically, preferably biannually and compared with the purity of the RM as mentioned in the CoA, before and after expiration using the same analytical protocol. The analysis should be conducted in an ISO 17025 accredited laboratory.

8. If the deviation in the purity of the RM after expiration is found within 5%, the analyte in the RM is acceptable and therefore be considered for continued use as a RM.

9. The gravimetric records should be maintained for RMs during storage.

[1] Directorate-General for Health and Food Safety, European Commission (SANTE)
[2] High-performance liquid chromatography
[3] Diode-Array Detection
[4] Ultra-violet spectroscopy
[5] Gas chromatography
[6] Flame ionization Detector
[7] Electron capture Detector
[8] Liquid chromatography
[9] Mass chromatography

ANNEX

DEFINITIONS

Reference Material (RM)

Reference material is a primary material which is sufficiently homogenous and stable with respect to one or more specified properties, and which has been established to be fit for its intended use in a measurement process.

Certificate of Analysis (CoA)

A document that provides the relevant information about certified purity, concentration date of expiry, and measurement uncertainty of an RM.

Certified Reference Material (CRM)

A certified reference material is a specific class of reference material whose property values (purity, concentration, etc.) are determined and certified in accordance with metrological principles using international best practice protocols established as per ISO guidelines. They provide a benchmark for analytical laboratories to deliver accurate and comparable results. Testing laboratories use certified reference materials to calibrate measuring instruments, evaluate test procedures and for quality control purposes.

Purity

Characteristic of a reference material, when stored under specified conditions, the measurement of the quantity of a prevalent component of a substance when only that component is present.

Reference Material Producer (RMP)

Company, organization or the agency accredited as per ISO Guide 34:2009 that produces reference materials and authorizes its property values as well as issues certificate of analysis for the reference material.

Stability

Testing of certified reference material (CRM) utilizing “isochronous” measurements based on storage design (storing CRM at different temperature ≤ 25°C for different time periods). All measurements are done at the same time and compared to long-term stability studies using reference time or reference temperature.

Proficiency Testing (PT)

Proficiency Testing is inter-laboratory comparison that enables labs to monitor the quality of their analytical results. It determines the performance of individual laboratories for specific tests or measurements and is used to evaluate laboratories’ continuing performance. An unknown sample(s) is received and analyzed by the laboratory, the lab results are returned to the PT provider, a statistical analysis of all labs results is performed, and the individual laboratory receives data on their performance compared to all other laboratories.

Quality Control Materials (QCMs)

QCMs are RMs and its principal function is to provide laboratories with an economical means of checking their routine test procedures for precision on a regular basis. QCMs are also referred as in-house reference materials, quality control samples, whose property values are sufficiently homogeneous, stable, and well established to be used for maintaining or monitoring measured processes. A QCM does not have formally assigned property values or uncertainties and are characterized only for a limited scope (a limited number of property values) and for specific laboratory applications.

APPENDIX IV

LIST OF PARTICIPANTS
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