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Good practice in reference material preparation

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Technical Report

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Good practice in reference material preparation

Bonne pratique pour la préparation des matériaux de référence

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 334, *Reference materials*.

This first edition of ISO/TR 33402 cancels and replaces ISO Guide 80:2014, which has been technically revised.

The main changes are as follows:

— this document provides guidance for the preparation of reference materials and does not include information about characterization or the assessment of homogeneity and stability;

— the scope of this document has been broadened to include all types of matrix reference materials and not only reference materials used for statistical quality control.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Introduction

Reference materials (RMs) are widely used in measurement laboratories for a variety of purposes, and it is important to ensure that the material most appropriate for a particular application is used. Certified reference materials (CRMs), i.e. those which have at least one certified value with associated uncertainty assigned by a metrologically valid procedure, are primarily used for method validation and calibrations providing metrological traceability.

While many RMs do not require characterization by metrologically valid procedures, they can be prepared to meet specific measurement requirements, including quality control. The key requirements for these RMs are sufficient homogeneity and stability, with respect to specific properties, for the intended use. Proper preparation processes can ensure the material's homogeneity and stability.

This document provides general information on key steps in material preparation of candidate matrix RMs. It is intended for laboratory staff involved in preparing and using matrix materials for specific applications. Reference material producers (RMPs) can also use it as an information source for the preparation steps of RM production.

The document includes case studies highlighting key considerations in RM preparation. Most of the case studies describe the production of matrix RMs used for statistical quality control and include information about the preparation of the materials as well as additional information about the characterization of the property values and the assessment of homogeneity and stability, as applicable.

The general requirements for the competence of reference material producers (RMPs) are outlined in ISO 17034, specifying necessary sample preparation steps. ISO 33405 covers guidance for assessing homogeneity and stability, characterization, and value assignment of property values. ISO 33403 provides guidance for the correct use of RMs. The requirements and guidance in these documents rely on the competent preparation of the candidate RM. However, preparation steps, especially for candidate matrix RMs, are intricate, and there is a lack of guidance focusing on these steps.

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Good practice in reference material preparation

1 Scope

This document gives general information on the key steps for the preparation of candidate matrix reference materials (RMs) including the material specification, sourcing and selection of bulk material, and the processing of the material, which are important steps for the production of matrix RMs.

The document provides information on the preparation of candidate RMs for laboratory staff who prepare and use matrix materials for their specific applications. This document can also be used by reference material producers (RMPs) as an information source for the preparation of the RMs that they produce.

This document also offers examples of specific case studies covering the preparation of matrix RMs in different fields of application (see Annexes A to F). These are not complete "production manuals" but highlight key considerations for the preparation steps of RMs.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO Guide 30, *Reference materials — Selected terms and definitions*

ISO/IEC Guide 99, *International vocabulary of metrology — Basic and general concepts and associated terms (VIM)*

ISO 3534-1, *Statistics — Vocabulary and symbols — Part 1: General statistical terms and terms used in probability*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO Guide 30, ISO/IEC Guide 99 and ISO 3534-1 apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Overview of preparation of candidate reference materials (RMs)

Many RMs and CRMs are produced by RMPs and are commercially available. However, laboratories conducting routine tests frequently encounter difficulties in acquiring matched matrix RMs that possess a comparable matrix and analyte content level, or even just one of these aspects. In cases where matched matrix RMs are challenging to obtain from the market, the capability to prepare samples closely matched to those used in routine tests becomes crucial.

For such RM users, the preparation of homogeneous and stable materials prior to conducting assessment of homogeneity and stability is crucial. If the preparation steps are inadequate to ensure a sufficient level of homogeneity and stability, the material will not be suitable for the intended use. Therefore, the preparation of any candidate RM requires a level of technical and organizational competence. It is acknowledged that in

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many cases the candidate RMs are prepared by technically competent staff that are knowledgeable about the materials and processes being used.

This document focusses on the steps involved with the preparation of RMs. The key steps involved in the production of a matrix RM are summarized in the flow chart in Figure 1 and are described in more detail in References [12] and [13].

The following clauses offer detailed information on material specification (Clause 5), sourcing and selection of bulk material (Clause 6) and material processing (Clause 7). More guidance on the rest of the production steps, which includes the characterization, as well as the assessment of homogeneity and stability is covered in ISO 33405. Any requirements for the production of RMs can be found in ISO 17034.

Materials can be sourced from, and processed by third parties, where they have specialized equipment and/or expertise. Materials can even be products that are commercially available and meet the user's specifications (e.g., food products available in appropriately sized units from a single production batch).

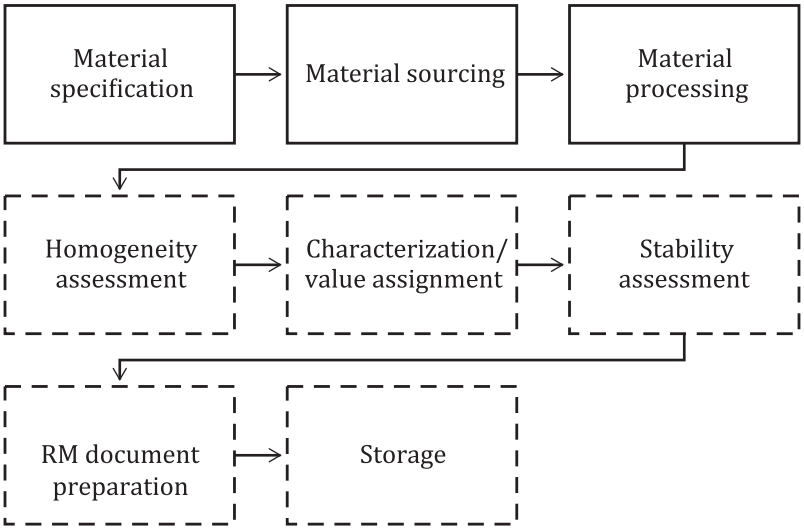

Figure 1 — Key steps in the production of RMs. This document focusses on the steps for the preparation of an RM (blocks with a solid black outline).

5 Material specification

5.1 General

The specification of a candidate matrix RM is a crucial consideration. The intended use, including the acceptable level of uncertainty for the measurement or test results, is an important consideration for both the production and use of all RMs. Key criteria include similarity to real samples and availability in appropriate quantities. In practice, the impetus for the preparation of such candidate RMs can often be the fact that adequate matrix RMs are not available. Therefore, the specific matrix and property combination for the test sample in question are likely to be used, and matching is not an issue for laboratories.

5.2 Matrix type and matching

In general terms, the uncertainties associated with a measurement result from a homogeneous sample arise from the two main stages of the measurement procedure:

— the preparation of a sample comprising digestion, extraction, clean-up, etc.;

— the measurement of the property in the prepared sample by a suitable technique.

The key criteria in the specification and selection of an RM are therefore for the material to be as similar as possible to real samples and available in appropriate quantities.

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The matrix of the RM needs to be the same or as similar as possible to the matrix of the routine test samples, so that a satisfactory result for the RM is genuinely indicative of satisfactory results for the test samples. This matrix matching requires some knowledge of the analytical procedure used on the routine samples, so that a judgment can be made as to the degree of variation of the physical/chemical properties of the sample and test matrices that can cause them to respond differently to a particular measurement procedure. For example, a freeze-dried food matrix can behave differently during analysis to a similar foodstuff with higher moisture content.

Commutability has particular significance in clinical chemistry and has been described elsewhere[14].

5.3 Properties and property values

For all RMs, the properties of a candidate matrix RM are crucial for its intended use in measuring routine test samples.

When the material is employed to verify quantitative measurement results, having a property value close to the mean value of typical test samples or values near the decision limit for the application becomes crucial. This can be verified through preliminary screening measurements on several candidate source materials to ensure the selection of the most appropriate one.

In situations where an RM is used for the statistical control of a measurement method using a quality control chart, the important characteristic of the RM is that its matrix is closely resembling that of the test sample.

For drift monitoring, the important characteristics of the RM include stability and the ability to provide a measured signal that minimizes counting statistical uncertainty. An optimal drift monitor material can have a higher concentration of the measurand compared to the test sample.

5.4 Unit size

Unit size is the amount of material that comprises a single unit of the RM. When preparing a candidate RM, the size of individual units is based on the likely use, i.e., the amount of material needed for the measurements concerned and whether the units are to contain sufficient material for a single analysis or multiple measurements.

5.5 Total bulk amount of material

An estimate is needed of the total bulk amount of candidate matrix RM that needs to be sourced. In principle, this can be estimated by considering:

— the expected number of units needed for the lifetime of the material;

— the expected number of units needed for homogeneity and stability testing and characterization (as applicable);

— the unit size;

— the preparation yield;

— the quantity of material that can readily be homogenized;

— the assumed stability of the material;

— the type and size of the storage facility.

6 Sourcing and selection of bulk material

Sourcing of bulk materials for RM production can at first seem difficult, especially in those cases where large quantities of material are needed. However, there are several options that are available including:

— leftover sample material from testing activities;

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— accurate gravimetric formulation.

Processing the bulk material can have significant cost implications for the production of RMs and simple, straightforward processing methods need to be used to ensure cost-effective RM production. The sourcing of the material usually considers the difficulty and cost implications of the processing of the material. The exact preparation procedures to be followed for a particular RM will depend on the nature of the matrix and the properties of interest.

In general, liquid matrix RMs are much easier to produce than their solid counterparts. The main reason for this is that homogeneous liquids can easily be achieved even with rudimentary equipment (e.g., large mixing containers equipped with paddles or magnetic stirrers). A liquid is easily spiked, filtered, or mixed with additives and stabilizers. The corresponding processes for solid materials, milling, grinding, mixing, and sieving are much more difficult to accomplish homogeneously, especially for quantities greater than 20 kg. These techniques require a significant investment in major capital equipment when large-scale preparation is envisaged.

When sourcing biological materials for example, for control of measurement procedures for medical laboratories, the following specific issues need to be considered:

— ethics of the retention and use of residual patients' samples for the production of RMs;

— legal liabilities of retention and use of residual patients' samples purchased for the production of RMs;

— medical laboratories creating RMs need to have a high degree of confidence in the identity of the material selected, to avoid use of misidentified organisms;

— materials sourced for RM production are screened for potential risks including health hazards, especially if the processing includes the use of contaminated sharps or has the potential for aerosol formation.

7 Material processing

7.1 General

Once the bulk material has been sourced for the candidate matrix RM, there are several processing stages that need to be carried out to ensure the candidate matrix RM has the appropriate homogeneity and stability for its intended use. Take care to ensure consistency in processing across multiple days. Some of the more common processes are described in 7.2 to 7.10.

7.2 Avoidance of contamination

For all candidate matrix RMs, it is important to prevent contamination by substances which can potentially interfere with the intended measurement process (e.g., a similar material or contamination of a blank material). Hence, all containers are carefully cleaned and dried before filling to remove possible contaminants.

In addition, consideration needs to be given to the possible interaction of bulk material with processing equipment and/or leaching of contaminants/impurities from the processing equipment parts, or the container, into the bulk material.

7.3 Drying

Removal of water makes candidate matrix RMs far easier to handle and improves both their transportation and long-term stability. Drying of soils and similar matrices is carried out at ambient or elevated temperatures, depending on the properties of interest, since the more volatile components could be partly lost at higher temperatures. Water removal also reduces the likelihood of microbial growth formation, which is a particular problem with biological materials. Freeze-drying is a technique which is useful with temperature sensitive properties or matrices.

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7.4 Milling and grinding

For solids, some form of crushing, milling, grinding and particle size reduction is often necessary to ensure uniform particle size and to improve homogeneity of the candidate matrix RM. For large quantities, these processes are slow and can take several days to complete. Take care not to introduce contamination from the apparatus during the grinding process. The health and safety aspects of grinding large quantities of particulate matter, which could have toxic components, needs to also be considered. Cryogenic grinding at –78 °C (solid CO_2) or –196 °C (liquid N_2) could be necessary for polymers, biological, oily/fatty, and thermally labile materials.

Specialised equipment can allow producing a material with a smaller particle size than laboratory samples, which can lead to changed extraction or digestion behaviours. This can result in the reference material not being representative of the real sample anymore. It is therefore important to ensure that also the particle size of the RM is representative for real samples.

7.5 Sieving

Sieving is often carried out after milling and grinding to improve the homogeneity of the candidate matrix RM. Particulate materials such as soils, ores, ashes, and ground biological materials are passed through a standard sieve to remove large particles that are above a prescribed size.

Sieving, however, changes the matrix composition. If a large fraction is removed by sieving, the analyte concentration can change, and the matrix can no longer reflect the composition of regular test samples.

7.6 Mixing and blending

When the candidate matrix RM is in solid form, it is homogenized by thorough mixing, using for example a roll-mixer, shaker, or end-over-end mixer. Such mixing is carried out after milling, grinding, and sieving.

Blending of two or more materials with sufficiently similar matrix compositions and differing property values can enable the production of RMs with a desired property value, a set of similar RMs covering a range of property values, or the production of RMs from an existing RM.

To obtain homogeneous mixtures, the materials to be mixed need to have similar densities and particle size distributions.

7.7 Filtration

Filtration of solutions before bottling removes any particulate and fibrous solids that would compromise the homogeneity of the bulk candidate matrix RM. However, some liquids cannot be filtered due to:

a) viscosity;

b) potential loss of active ingredients by adsorption to the filter;

c) the introduction of contamination. Qualification of the filter is critical to avoiding loss of active ingredients.

Typically, liquids such as waters and leachates, are filtered through a 0,45 µm filter prior to bottling or ampouling.

7.8 Stabilization

Certain analytes in the candidate matrix RM are unstable and therefore need to be stabilized at the bulk stage of the preparation procedure. Metals, for example, can precipitate out of neutral or alkaline solutions because of hydrolysis or oxidation, and adjustment of the pH of the solution to below 2 counteracts this problem. Copper at a concentration of 1 $mg \cdot l^{-1}$ has been used to counteract algal growth in aqueous solutions. Different materials can require other approaches such as addition of antioxidants, preservatives, texture stabilizers, etc.

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7.9 Sterilization

Prepared soils, sewage sludges, and biological materials can contain persistent pathogens that are potentially harmful to humans. They can also contain spores that cause fungal moulds to develop on storage, which could initiate changes in either the composition of the bulk material or the individual units. Such organisms need to be destroyed before the final units are prepared and packaged.

Before sterilizing any candidate matrix RMs, it is important to consider the impact of the proposed sterilization process on the properties of interest and/or the matrix, particularly those which degrade at elevated temperatures.

Autoclaving is an inexpensive and convenient means of sterilization that can be used for materials that are temperature resistant, for example, metals in sediments. Autoclaving can be done on the bulk material prior to final homogenization and unit preparation or on the final samples. However, it is important to ensure that the core of the material reaches 121 °C.

Irradiation can be used on the final packaged units (e.g., ampoules, bottles, or pouches). Gamma irradiation is a convenient means of sterilization at ambient temperature so changes in matrix composition are less likely than with autoclaving. Dose values need to be determined such that they are effective in removing pathogens but do not adversely affect the material by, for example, raising the temperature to unacceptable levels (e.g., chocolate). However, gamma irradiation is beyond the means of most laboratories, requiring specialist subcontractors.

Sterilization is performed after subdivision and packaging has been completed, and the material is in its final packaged form, otherwise the material will not be sterile.

7.10 Subdivision and packaging

7.10.1 General

The last steps of the material processing are subdivision and packaging. Subclauses 7.2 and 7.3 describe some of the key considerations for the subdivision process and choice of containers to ensure the RMs are sufficiently homogeneous and stable for their intended use.

Some candidate matrix RMs are used as bulk materials and do not need to be packaged into individual units. In-house reference materials are often not distributed and are therefore not always packaged into units, but subsamples are taken from the bulk prepared material as and when needed.

7.10.2 Choice of containers

For RMs to be prepared cost-effectively, one aspect that needs careful consideration is the choice of appropriate containers for the individual units. If unsuitable containers are used, the material could quickly degrade. The type of container used depends on the inherent stability of the material and the length of time it is expected to remain stable. For particularly susceptible materials, two layers of containment (e.g., a vial within a polyethylene bag) can provide additional protection against degradation and contamination.

The following examples serve to illustrate the need for careful consideration of the container and its closure.

— Materials can either lose or pick up moisture if the container is not securely closed. Glass containers with screwcaps fitted with “polycone” inserts[1)] are preferable to simple screw caps. Sealed cans, foil pouches, or septum-lined crimp-top vials offer more security.

— Oxygen sensitive materials need to be prepared and sub-sampled under an inert gas atmosphere (nitrogen or argon).

— For aqueous samples containing low concentrations of metals (e.g., mg/kg or below), glass containers are not recommended because of possible adsorption of the metals onto the walls over time. High-density polyethylene (HDPE) bottles with screwcaps are more suitable for this application but have the potential

1) Polycone liners are cone-shaped polyethylene cap liners that provide a better seal than simple wadded cap closure.

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problem of loss of water by evaporation through the bottle walls. This can be minimized by storage in a refrigerator (rather than at ambient temperature) or using fluorine-treated polyethylene bottles.

— The possibility of contamination of the RM by the leaching of impurities from the container also needs to be considered. For example, the iron content of canned foodstuff RMs could be subject to unpredictable increases on a can-by-can basis, as iron leaches from the can wall into the food matrix. Bottles (whether glass or HDPE) containing aqueous acid solutions could also give rise to leaching problems. As a rule, containers that can interact with the RM need to be carefully evaluated before use by suitable leaching trials.

— For relatively inert matrices, such as soils and other dried environmental or biological materials, screw-cap glass jars are usually satisfactory. Amber glass gives additional protection against degradation induced by light.

— RMs comprising relatively volatile components susceptible to evaporation, such as some organic solvents, will normally require a septum-lined crimp-top, glass vial, or flame-sealed glass ampoules. It is preferable for vials and ampoules to be amber to reduce the impact of light.

Some preliminary experimental work, including blank and stability studies, can be necessary to identify the most suitable container type to use for a particular RM.

If the unit size is larger than the sample size taken for a measurement, then consideration must be given to the stability of the material for reuse according to the requirements of ISO 17034. The effect of repeated opening and closing of the sample containers is also assessed if repeated use of the material is anticipated. If a material is unlikely to be stable once opened, then it is preferable to restrict the unit size to a single use portion.

Tamper evident closures need to be considered if the unit is only intended to be used once.

7.11 Subdivision procedures

Once a homogeneous bulk candidate matrix RM has been produced, the essential requirement of any subdivision process is that the homogeneity of the material is maintained. Ensure that the sub-division process itself, or the time taken to complete the subdivision of bulk material, does not re-introduce heterogeneity into the material. This could conceivably occur in several ways.

Matrices comprised of mixtures of liquids of differing volatilities (e.g., ethanol in water) can undergo selective evaporation of one component during a prolonged subdivision run, causing a rising or falling trend in property value from the first to the last units produced. Effects of this sort can be minimized by protecting the bulk material from evaporation and by completing the subdivision in as short a time as is consistent with accurate dispensing.

All liquids and solutions need to be stirred continuously while individual aliquots are being dispensed. Solutions need to be filtered before dispensing commences if particulates are likely to be present to an extent that could affect the properties of interest.

Take care with solid particulate matrices such as soils, sediments, industrial products, etc. to ensure that segregation of finer particles does not occur during subdivision. Take special care when sampling bulk material from a large drum, to ensure that there is no vertical segregation. Riffling is a process for representatively subdividing free-flowing powdered materials so that each aliquot receives similar particulate fractions. When operated effectively, riffling minimizes flow segregation and produces units with a low between-unit variation. Commercial riffling devices can be used to subdivide such materials without introducing heterogeneity. Sampling and subdivision of particulate materials are described in more detail in ISO 14488[15].

In food matrices with a high fat content (e.g., mackerel paste), there could be a tendency for the fat to separate as a discrete phase. If such effects occur, the matrix needs to be stirred continuously during dispensing and/or additives included in the matrix to slow down the separation process.

As a general principle, subdivision of a bulk material is completed as quickly as possible to minimize the opportunities for the matrix to revert to heterogeneity. Where appropriate, take steps to maintain a homogeneous bulk material during the subdivision process. It could be necessary to discard the first and/or

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