

Use of ERM certificates and materials

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This application note describes some practical aspects associated with handling and use of certified reference materials (CRMs) in laboratories. The various values provided on a certificate, re-use of materials, applying a moisture correction and interpretation of the traceability statement are also explained.

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INTRODUCTION

Understanding the information contained in reference material certificates and correct use of these materials are necessary to get the maximum benefit from them. This application note explains the basic terms used on ERM certificates, and gives guidance on the practical handling of materials.

TERMS ON THE CERTIFICATE

Types of assigned values

Three categories of values are assigned for ERM-branded reference materials:

Certified values fulfil the highest standards for reliability. They are traceable to stated references and are accompanied by a GUM (ISO Guide 98 "Guide to the expression of uncertainty in measurement") compatible expanded uncertainty statement valid for the entire shelf life of the ERM-CRM.

Indicative values are not certified due to either a larger uncertainty than required for the intended use or insufficient variety of methods used in the characterisation. The information is therefore unsuitable for certification at the accuracy required for certified values.

Additional material information are values created during the certification exercise, which are usually the result of one method only and indicate the order of magnitude rather than an accurate value.

In summary, certified values are those values the certifying body is confident in assigning with the highest accuracy, while indicative values display higher uncertainties and/or lack a full traceability statement. This hierarchy in reliability is shown by the fact that only certified values are on the first page of the certificate. It follows that certified values are more assured than indicative values which in turn are more assured than additional material information.

Metrological traceability statement

Certified and indicative values come with a traceability statement. These statements unambiguously identify the measurand as well as the traceability of the values assigned to this measurand (see also the ERM policy on traceability on www.erm-crm.org). This information is given in one or two footnotes on the certified or

indicative values and measurands (see Figures 1 and 2). The following alternatives exist:

Measurands can be structurally defined ("rational") like for total cadmium or ochratoxin A, or procedurally defined (empirical), such as for dietary fibre, extractable cadmium or impact toughness, which are defined via specific measurement protocols.

For structurally defined measurands, ERM principles are stricter than those of ISO Guide 34 and 35 and require availability of results obtained from at least two completely independent methods or confirmation of results by primary methods of measurement by an independent method to demonstrate the absence of any method bias of assigned values.

Values assigned to the measurands can be traceable to the International System of Units (SI) or to an artefact (empirical scales). In the former case, all input factors are calibrated with standards whose values are traceable to the SI, whereas in the latter cases arbitrary standards have been used for at least one step in the calibration (e.g. World Health Organisation primary reference preparation in clinical chemistry, Vienna Standard Mean Ocean Water (VSMOW) for chemical shift).

ERM® - AD452/IFCC		
GAMMA-GLUTAMYLTRANSFERASE		
	Certified value ¹	Uncertainty ²
Catalytic concentration in reconstituted material	114.1 U/L 1.90 µkat/L	2.4 U/L 0.04 µkat/L
<small>1) This value is the unweighted mean of 12 sets of results, independently obtained from 12 laboratories. It is traceable to the IFCC primary reference method at 37 °C. The material must be reconstituted according to the specified procedure (see below). Values were converted from U/L into µkat/L by multiplication with 0.01667.</small>		

Figure 1: Traceability statement of ERM-AD452/IFCC. The measurand is procedurally defined and proper calibration of all input factors is assumed.

In earlier ERM certificates, this information was combined into one footnote (Figure 1). Since the adoption of the common ERM policy on traceability, this information is given in two footnotes, one connected to identity of the measurand itself, the other specifying the traceability of the values assigned to it.

Minimum sample intake

Every material is intrinsically heterogeneous. The minimum amount of material that is representative of the whole unit (bottle, vial etc.) is defined as minimum sample intake (Figure 2). The certified value and its uncertainty cannot be guaranteed for subsamples smaller than the minimum sample intake.

Expiry date

Producers of reference materials guarantee the integrity of the material and the validity of the certificate for a specified time (known as the shelf life), provided the material is unopened and stored under the recommended storage conditions. This does not automatically mean that the user has to discard the unused sample once the shelf life has expired, but the producer cannot guarantee stability any longer. Users can continue to use a material under their own responsibility, if they have additional evidence of stability (e.g. no changes in quality control charts, comparison with other materials), however the material certificate will not be valid.

ERM® - BC367		
RAPESEED (COLZA)		
Parameter	Certified value ¹	Uncertainty
Total glucosinolate (GSL)	99 mmol/kg	9 mmol/kg
Sulphur	10.3 g/kg	0.5 g/kg
<p>1) The certified values for both GSL and S are the unweighted mean of the means of the accepted sets (GSL and 7 for S). The values are traceable to SI.</p> <p>2) Estimated expanded uncertainty U with a coverage factor $k = 2$, corresponding to a level of confidence of 95% as defined in the Guide to the Expression of Uncertainty in Measurement (GUM), ISO, 1995. Uncertainty arising from characterisation as well as from homogeneity and stability assessment were taken into consideration.</p>		
This certificate is valid for one year after purchase.		
Sales date:		
<p>The minimum sample intake is:</p> <ul style="list-style-type: none"> - 500 mg for total glucosinolate (GSL) determination, - 20 g for sulphur calibration by XRF (preparation of discs), - 200 mg for sulphur determination after digestion. 		

Figure 2: The shelf life and minimum sample intake are marked in blue and green, respectively.

The shelf life may be extended by the producer if additional information on the stability becomes available. This, however, refers only to newly purchased samples and not to samples distributed before the extension of the original shelf life.

Instructions for use

The instructions for use give a detailed description for each material. These descriptions can refer to dry mass correction (Figure 3), reconstitution procedure, use of values, storage of the material etc. If these instructions are not followed, the assigned values are not valid.

INSTRUCTIONS FOR USE
<p>The sample can be used as it is from the bottle. Before a bottle is opened, it should be shaken manually for 5 min so that the material is re-homogenised. The correction to dry mass should be made on a separate portion of 100 mg which should be dried in an oven at 102 °C for 3-4 h until constant mass is attained. The recommended minimum sample intake is 500 mg.</p>

Figure 3: Definition of the dry mass correction for ERM-CE477. Please note the different recommended sample intakes for dry mass correction and certified measurands (in this case butyltins), reflecting different degrees of homogeneity for moisture and butyltins.

HANDLING ISSUES

Measurement method to be used

The measurement method must determine the same measurand as described in the certificate. This means that for procedurally defined measurands, the method specified on the certificate must be used. For structurally defined measurands, any method determining this measurand can be used and should give unbiased results.

All instruments must be properly calibrated to ensure that measurement results are traceable to the same reference as the certified value. For results traceable to an artefact, a standard whose value is traceable to the same artefact must be used.

Use of opened bottles

For opened units, alteration or even degradation of the material can happen which could not be accounted for during the certification process. Therefore, CRM producers cannot guarantee the assigned values of opened units. It is up to the user's judgement whether or not this material can be further used and which storage conditions or treatment are necessary. As a general guideline, materials should be stored cool, dry, in the dark and closed. Further information is often available in the certification report, which is freely available on www.erm-crm.org. In any case, materials from opened containers should be used as soon as possible after opening to minimise change.

Moisture correction

Many certified values are stated as content per dry mass of sample. As results from different methods (e.g. drying oven, Karl Fischer titration, vacuum drying oven) may differ significantly, the procedure for moisture correction must be clearly stated on the certificate (Figure 3) and this method must be

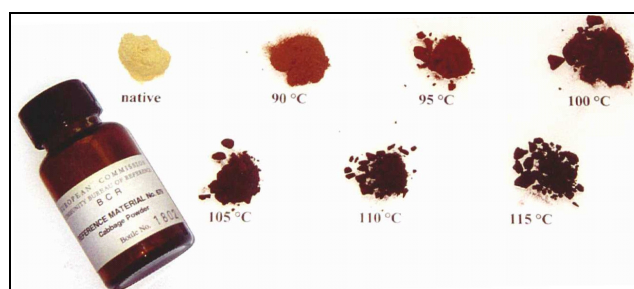


Figure 4: Effect of different drying conditions on a cabbage reference material. Specified drying condition on the certificate : 16 h at 70 °C

used. Using different conditions can significantly alter the material (Figure 4) and the results obtained. Determination of the dry mass must be performed on a separate subsample.