

# Guidelines for the Production of BAM Reference Materials

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## Guidelines for the Production of BAM Reference Materials

These guidelines describe the generic procedures for the production of reference materials at BAM. They implement the general provisions given in the BAM Guidelines Book (RLB-3.5).

The guidelines and additional documents dealing with generic topics in the production of reference materials are available for download on the BAM intranet site of the BAM Reference Materials Committee. Supplementary regulations and procedures for particular technical fields are specified and documented in the organisational units concerned.

### 1 BAM Reference Materials Program

#### 1.1 General

In the framework of its statutory tasks (Sprengstoffgesetz [Explosives Act] §§ 44, 45) BAM is responsible for carrying out and evaluating physical and chemical tests of substances and facilities including the provision of *reference materials* and reference procedures.

The main activities in the field of reference materials are production and distribution of *certified reference materials* (see section 1.2). In addition to these BAM produces reference materials for use in interlaboratory proficiency tests (PT materials). Selected spare PT materials are marketed as quality control materials (QC materials) (see section 1.3).

Certified reference materials are partly produced stand-alone, partly in cooperation with external partners. PT materials are normally produced stand-alone.

#### 1.2 Certified Reference Materials

Currently BAM produces certified reference materials (CRM) of various different categories as follows:

##### **Pure substances, solutions and synthetic mixtures**

*Certified properties: Purity, chemical composition*

- Inorganic pure substances
- Organic calibration standards
- Gas mixtures
- Isotopic standards

##### **Foodstuffs and related materials**

*Certified properties: Contents of trace and minor constituents*

- Organic pollutants in various matrices

##### **Environmental materials**

*Certified properties: Contents of trace and minor constituents*

- Organic pollutants in various matrices
- Inorganic pollutants in various matrices

##### **Industrial materials**

*Certified properties: Chemical composition*

- Iron and steel products (EURONORM CRM)
- Non-ferrous metals and alloys
- Special materials

## Materials for physical properties

*Certified properties: Physical properties*

- Porous materials (various porosimetry characteristics)
- Elastomeric materials (surface roughness, electrostatic properties, abrasion resistance)
- Polymer materials (molecular weight)
- Surface and layer materials
- Optical materials, X-ray films
- Fluorescence standards

A comprehensive compilation of available certified reference materials is published in the internet ([www.bam.de](http://www.bam.de) > Expert Information > Reference Materials / [www.bam.de](http://www.bam.de) > Service > Webshop) and in a printed catalogue. There, among others, contact persons are given for the various materials.

### 1.3 PT Materials / QC Materials

BAM operates a program of regular interlaboratory comparisons for proficiency testing (PT) in different fields of environmental analysis (see [www.bam.de](http://www.bam.de) > Expert Information > Interlaboratory Comparisons). Spare PT materials for inorganic soil analysis are marketed as quality control materials (QC materials). A listing of available PT / QC materials is published in the internet ([www.bam.de](http://www.bam.de) > Expert Information > Reference Materials – contact: Dr. H. Scharf, Division I.1)

## 2 General Regulations

The general regulations applicable to the production of certified reference materials (CRM) are defined in the BAM Guidelines Book (RLB), doc. RLB-3.5.

The procedures for CRM production comply with the recommendations of ISO Guide 34 and, in essence, also to those of ISO Guide 35. Concerning the treatment of stability, besides the strategy recommended by ISO Guide 35 alternative technically equivalent strategies are utilised, see section 5.4.

The information stated in certificates for reference materials complies with the recommendations of ISO Guide 31.

The quality management for CRM production complies with the requirements of ISO Guide 34.

Procedures and quality management for the production of PT materials comply with the recommendations of ISO/IEC Guide 43-1 and, in addition, are oriented on those for the production of certified reference materials.

CRM for analytical-chemical characteristics may be utilised for the presentation of BAM's measurement capability in the international data base operated by the BIPM (CMC entries – see RLB-3.7). The particular requirements for this purpose are described in section 6.6.

## 3 Schedule of CRM Projects

A CRM project according to these guidelines is a project for the production of a CRM or a series of similar CRM. The schedule of a CRM project is divided into several phases as follows:

**A – Planning and Preparation**

- Initial assessment
- Specification of requirements for the CRM
- Feasibility assessment
- Production planning
- Selection of collaborators
- Presentation to the Certification Committee
- Decision: BAM CRM or ERM
- Approval

**B – Implementation**

- Production / purchasing of the starting material
- Preparation and packaging
- Assessment of homogeneity
- Assessment of stability
- Certification measurements
- Data evaluation

**C – Documentation and Release**

- Certification report
- Certificate
- Handling by the Certification Committee
- Release of the CRM
- Labelling
- Metrological utilisation (CMC Entries)

**D – Storage and Distribution**

- Storage
- Stability monitoring
- Consultancy and distribution

The flow chart in Annex B provides a survey of the detailed schedule.

**4 Planning and Preparation****4.1 Initial Assessment**

In any decision on the production of a CRM (or a series of CRM), due account shall be given to the aspects as follows.

**Demand**

- Regulatory relevance
- Target groups (customer requests)
- Cooperations (national and international obligations)
- Market requirements

**Significance / benefit**

- Strategic significance
- Economic significance
- Relevance for standardisation and pre-normative research
- Relevance for interlaboratory comparisons
- Relevance for the validation of test procedures
- Availability of comparable reference materials

**Relevant aspects**

- Competence and experience
- Regulatory tasks
- International obligations
- Metrological integration
- Interdisciplinary projects
- Orders from industry

**Cost**

- Financial expenses
- Personnel expenses
- Expected sales

**4.2 Specification of Requirements for the CRM**

The detailed requirements for the CRM arise from the intended use. As a rule the following items are relevant:

- the matrix, i.e. the kind of material
- the property values to be certified
- the acceptable uncertainty level of the certified property values
- the application field of the CRM
- the application form of the CRM

**4.3 Feasibility Assessment**

The next step is a comprehensive assessment of the material, technical and organisational conditions for realising the CRM project. There, in addition to the requirements considered in section 4.2, the following questions have to be addressed:

- Is it possible to acquire appropriate starting material?
- Is appropriate measuring capability available at BAM?
- Should external collaborators be included?

**4.4 Production Planning**

Given a positive result of the feasibility assessment, the various steps for the technical implementation of the CRM project are planned in detail:

**Production / purchasing of the starting material**

- Producer / supplier
- Suitability check
- Acquisition
- Storage

**Preparation and packaging**

- Preparation of the candidate material
- Specification of packaging units and containers
- Homogenisation and subdivision
- Packaging and labelling

**Assessment of homogeneity**

- Does the material require homogeneity testing?
- Which sample size is relevant?
- Which precision is required?
- Who performs the test?
- How is the test performed?

**Assessment of stability**

- Does the material require stability testing?
- Which influential factors have to be addressed?
- Which storage conditions should be considered?
- Who performs the test?
- How is the test performed?

**Certification measurements**

- Specification of the certification procedure
  - (a) In-house certification using a single measuring facility
  - (b) In-house certification using several measuring facilities
  - (c) Certification by interlaboratory comparison with external collaborators
- Specification of acceptable measurement procedures (where appropriate)

**Data evaluation**

- Who performs the data evaluation?
- Which procedure for data evaluation is used?
- Which software is used?

In addition planning is required for:

**Storage and distribution**

- Storage (conditions, capacity)
- Stability monitoring
- Consultancy and distribution

**4.5 Selection of Collaborators**

If other laboratories / institutes or other divisions of BAM are involved in a CRM project, and if their contributions are significant for the quality of the CRM, then their competence must be ensured and verified.

Significant contributions could be:

- Participation in homogeneity testing
- Participation in stability testing
- Participation in certification measurements

As far as possible, partners are selected among laboratories of proven competence. Otherwise appropriate measurement comparisons must be undertaken (e.g. an interlaboratory comparison for qualification). Further advice is given in the document RLB-3.5, section 5.2.

The project leader (normally the contact person for the CRM category concerned) keeps records of applicable partners including information on competence.

**4.6 Presentation to the Certification Committee**

In the planning stage, CRM projects are presented to the Certification Committee (see RLB 3.5, Annex 1). To this end, a CRM project planning form according to RLB-3.5, Annex 3 is used. The Certification Committee registers the project and provides advice as appropriate.

For planning further CRM from a series, an abbreviated version of the project planning form with reference to a previous comprehensive description is sufficient.

#### **4.7 Decision: BAM CRM or ERM**

BAM is a founding member of the ERM initiative, where leading European reference materials producers jointly market selected CRM under the trademark „European Reference Materials (ERM)“. Further information: [www.erm-crm.org](http://www.erm-crm.org).

CRM to be marketed as ERM are subjected to a comprehensive review by the other partners (currently IRMM and LGC). Documentation (planning, certification report, certificate) for such CRM have to comply with dedicated requirements agreed by the ERM consortium, see the intranet site of the Reference Materials Committee. Contact person for ERM: Dr. J. Vogl, Division I.5.

#### **4.8 Approval**

CRM Projects are approved by the responsible head of division. Approval can be granted, subject to revocation, for all CRM projects of an organisational unit.

### **5 Implementation**

#### **5.1 Production / Purchasing of the Starting Material**

Production and purchasing of the starting material largely depend upon the type of CRM:

- matrix materials, i.e. natural or industrial materials with typical levels of relevant properties, e.g. contaminated soils with certified concentrations of heavy metals around legal limits
- synthetic materials prepared for this particular purpose, e.g. calibration solutions or gas mixtures
- pure substances with certified purity and relevant impurity levels
- hybrid and special materials, e.g. preparations of matrix materials made by spiking with pure substances

When planning to produce matrix CRM, starting materials with suitable property levels must be obtained in sufficient amounts.

The starting materials must be checked whether they are suitable for the production of the planned CRM. This check a. o. concerns

- the levels of the properties to be certified and of other relevant properties
- storage and prospective stability
- further preparation

#### **5.2 Preparation and Packaging**

The main purpose of further preparation of the starting material (e.g. drying, milling, sieving, sterilisation etc.) is to generate a homogeneous batch of stable material with property levels as required. In addition the prepared material should be similar to the typical test samples used with the test methods for whose quality assurance the CRM is intended.

Packaging includes the following steps:

- Specification of packaging units and containers
- Splitting the batch among the packaging units
- Filling into the designated containers
- Labelling

When splitting the batch, homogeneity among packaging units must be ensured. To this end, particular measures (e.g. sample splitting by „cross riffing“) may be necessary.

The requirements for the containers depend upon the type of reference material. General requirements are as follows:

- The containers must be such that the reference material is protected against adverse effects of ambient conditions (air moisture, oxygen, light etc.).
- The reference material must be inert against the inner surface of the containers.

For storing the packaged material, appropriate storage conditions have to be specified and appropriate storage capacity has to be made available. Storage conditions are derived from available information about stability-relevant factors and, where applicable, dangerous properties of the reference material according to the relevant regulations for dangerous goods.

Besides storage conditions, shipping conditions have to be specified, considering ensurance of stability and, where applicable, dangerous properties according to the relevant regulations for the transportation of dangerous goods. There the results of stability investigations (see section 5.4) have to be taken into account.

### 5.3 Assessment of Homogeneity

For a CRM it must be ensured that the certified values are valid for all packaging units. In addition the certified values must be valid for all samples (withdrawn and handled according to specifications) from a packaging unit. Accordingly a distinction is made between

- homogeneity between packaging units and
- homogeneity within packaging units.

The purpose of homogeneity assessment is to determine the level of residual inhomogeneity after preparation and packaging. In addition associated contributions to the uncertainty budget of the certified values are evaluated.

The procedure for homogeneity testing and evaluation of associated uncertainty contributions complies with the recommendations of ISO Guide 35.

ISO Guide 35 is focussed on „batch certification“ as the normal case: the prepared material is split among several packaging units, and the certified values (including uncertainty) are specified such that they are valid for all packaging units. To this end, the recommended procedure includes provisions for testing homogeneity *between packaging units* and including a designated uncertainty contribution ( $u_{bb}$ ) to account for residual (or non exclusive) inhomogeneity after preparation and packaging. In particular, it is recommended to include an uncertainty contribution corresponding to the limited sensitivity of the measuring procedure for differences between samples.

According to ISO Guide 35, an assessment of homogeneity within packaging units is not mandatory. Neither is a separate uncertainty contribution mandatory, since as a rule such inhomogeneity is already accounted for by other uncertainty contributions. Instead appropriate conditions for withdrawing and handling CRM samples (minimum sample size, provisions before withdrawing samples, provisions after withdrawing samples) must be specified as to ensure that the certified values are valid for all samples (withdrawn and handled according to specifications) from a packaging unit.

*Note:* Inhomogeneity within packaging units is only relevant for solid reference materials. Gaseous and liquid reference materials are homogeneous as a rule.

Considering the issues above, the BAM policy is as follows:

- Gaseous reference materials are exclusively produced in the single-unit mode (i.e. no splitting among several packaging units). Therefore an assessment of homogeneity is not required.

- Liquid reference materials are produced in the single-unit mode as well as in the batch mode (splitting among several packaging units, e.g. vials). For batch production homogeneity assessment and a designated uncertainty contribution ( $u_{bb}$ ) are mandatory.
- Solid reference materials (powders, chips, granules etc.) are exclusively produced in the batch mode. Therefore homogeneity assessment and a designated uncertainty contribution ( $u_{bb}$ ) are mandatory in any case.

Compact materials are special cases, requiring procedures tailored to the definition of samples for specified applications. Homogeneity testing investigates differences between property values of different samples. The differences are accounted for by an associated uncertainty contribution or an associated trend (quantitative dependence on the sampling location).

Concerning the selection of the measurement method for homogeneity testing, it should be noted that for this purpose precision is the crucial figure of merit while trueness is rather of marginal importance.

ISO Guide 35 contains general recommendations for the performance and evaluation of homogeneity tests. Guidance for the interpretation and implementation of these recommendations are given in the document „Durchführung und Auswertung von Homogenitätsuntersuchungen nach ISO Guide 35“ [Performance and evaluation of homogeneity tests according to ISO Guide 35]. Supplementary procedures – as required – are given in the QM documentation of the organisational units concerned.

#### **5.4 Assessment of Stability**

For a CRM it must be ensured that the certified values are valid until the end of the utilisation period („expiry date“) specified in the certificate. This validity applies to unopened packaging units under proper storage. After opening there is no warranty of stability.

In stability testing the temporal change of certified values is investigated over an appropriate period, if necessary in dependence on stability relevant parameters like storage temperature. As a result of measurement data evaluation,

- it is determined whether and to which extent the material exhibits instability
- the conditions for storage and shipping are specified
- a (provisional or final) expiry date is specified

The procedure for stability testing complies with the recommendations of ISO Guide 35 and partially goes considerably beyond these recommendations.

As a deviation from the recommendations of ISO Guide 35, as a rule the uncertainty of stability measurements is not accounted for by an additional uncertainty contribution ( $u_{ITS}$  /  $u_{sts}$ ) to the uncertainty of the certified values but rather in the specification of the expiry date.

As another deviation from the recommendations of ISO Guide 35, materials exhibiting significant instability are not discarded perforce. If the instability can be controlled, i.e. if the temporal change of the certified values can be predicted with appropriate accuracy over a reasonable period of time, then also such materials can be certified.

A comprehensive justification and description of the procedure is given in the document „CRM stability testing policies scrutinised“ and in the publication „Stability testing in an integrated scheme“.

Concerning stability, the following cases are differentiated:

- Stability class 1: Materials whose stability (concerning the certified property values) at the designated storage and shipping conditions over the designated period of utilisation is evident according to expert opinions.
- Stability class 2: Materials for whose stability there is no evidence, but for which no significant instability was detected.
- Stability class 3: Materials where significant instability was detected, and where the kinetics (influential factors, temporal law) is unknown.
- Stability class 4: Materials where significant instability was detected, and where the kinetics (influential factors, temporal law) is known.

For materials of stability class 1 as a rule no stability test is undertaken.

For materials of stability class 4 the certified values concerned are specified as a function of time.

Materials of stability class 2 and 3 are treated in the same manner, i.e. no principal difference is made whether or not the temporal changes observed in stability testing are significant. In any case the observed trend (temporal change) including the confidence interval concerned (single-sided, confidence level 95 %) is extrapolated over the utilisation period under consideration. The expiry date is derived from the requirement that the extrapolated deviation from a certified value must not exceed the expanded uncertainty of the certified value. As a rule a safety margin is included in the determination of the expiry date.

Stability testing includes two phases as follows:

- comprehensive stability investigation in advance of the certification measurements
- supplementary stability investigations up to the release of the CRM

In addition, stability is monitored on a regular basis (for materials of class 2 and 3 only) until the CRM is sold out.

In the comprehensive stability investigation the degradation mechanisms under consideration and the relevant influential factors (in particular storage temperature) are accounted for. When temperature effects on stability are expected, samples are stored at different temperatures, and the temperature effect is evaluated using an appropriate model (model-based evaluation).

In the supplementary stability investigations samples are investigated after extended storage in order to examine the validity of the predictions derived from the comprehensive evaluation. If necessary, provisional expiry dates are revised.

Regular stability monitoring until the CRM is sold out is treated in section 7.2.

Planning and execution of stability investigations require due care that measurements on samples stored over different time intervals are comparable. This can be achieved by:

- isochronic experimental design
- drift control and drift correction (when necessary)

*Note:* Isochronic design means that all samples stored at different conditions over partly different time intervals are jointly investigated / analysed in a measurement series under repeatability conditions (see ISO Guide 35 and the references given there).

For the significance of the stability investigation the stability and the precision of the measurement procedure are the crucial figures of merit while trueness is rather of marginal importance.

ISO Guide 35 contains general recommendations for the performance and evaluation of stability investigations. Guidance for the interpretation and implementation of these recommendations are given in the document „CRM stability testing policies scrutinised “ and in the publication „Stability testing in an integrated scheme“. Supplementary procedures – as required – are given in the QM documentation of the organisational units concerned.

## 5.5 Certification Measurements

The procedure for the organisation and execution of certification measurements complies with the recommendations of ISO Guide 35.

For the determination of certified values the following strategies may be utilised:

- (a) In-house certification using a single measuring facility
- (b) In-house certification using several measuring facilities
- (c) Certification by interlaboratory comparison with external collaborators

*Note:* Also in cases where CRM with defined property values are obtained by preparation, the certified values as a rule result from measurements (e.g. weighing). Therefore such strategies fall under category (a).

For certification measurement only validated measurement procedures are admissible. Apart from special cases, trueness is the crucial figure of merit. Further general requirements for the measurement procedures to be used in certification are as follows:

- (a) In-house certification using a single measuring facility:
  - full metrological traceability
  - complete uncertainty budget
  - successful participation in related intercomparisons with other institutes
- (b) In-house certification using several measuring facilities:
  - full metrological traceability
  - complete uncertainty budget
- (c) Certification by interlaboratory comparison with external partners:
  - full metrological traceability

Apart from certification using a single measuring facility (preferably using a „primary method of measurement“) several independent measurement methods should be used for being able to detect and compensate method biases.

In the certification of method-specific properties a standardised measurement procedure is used. In this case the property is defined by the measurement procedure, and traceability to the standard takes the place of metrological traceability. This implies that all participants must work strictly according to the standard procedure concerned.

ISO Guide 35 contains general recommendations for the design and execution of certification measurements. Supplementary procedures – as required – are given in the QM documentation of the organisational units concerned.

## 5.6 Data Evaluation

The procedure for the evaluation of certification measurements complies with the recommendations of ISO Guide 35. A key issue is GUM-compliant evaluation and statement of the uncertainty of certified values.

As a rule, certification measurements are evaluated at BAM. In exceptional cases external experts are included.

In-house certification measurements are evaluated using dedicated data evaluation models and uncertainty budgets for the individual measurements. In case of several measurement procedures this includes consolidation of the various data evaluation models and uncertainty budgets. The basis for this is the model-based approach described in the GUM.

Certification measurements by interlaboratory comparison are evaluated according to pertinent statistical methods, using appropriate computer programs [e.g. SoftCRM V. 1.2.2 (successor of the former BCR Program) and SYSTAT V. 8.0 (SPSS Inc. 1998)].

In cases of batch certification the uncertainty budget for certified values has to include a contribution of the inhomogeneity between packaging units ( $u_{bb}$ ).

ISO Guide 35 contains general recommendations for the evaluation of certification measurements. Supplementary procedures – as required – are given in the QM documentation of the organisational units concerned.

## 6 Documentation and Release

### 6.1 Certification Report

The production of a CRM has to be documented comprehensively in a certification report, addressing at least the following issues:

- Preparation and packaging including information on utilisation and storage
- Homogeneity assessment including associated uncertainty evaluation and specification of minimum sample size
- Stability assessment including determination of the expiry date and specification of storage and shipping conditions
- Certification measurements including participants, measurement methods and measurement results
- Evaluation of certification measurements including calculation of certified values and evaluation of their uncertainty
- Traceability of certified values

Annex C gives a model for structuring certification reports.

For CRM that are issued jointly with external partners (e.g. EURONORM CRM or ERM), the applicable agreements concerning the contents of certification reports have to be considered.

Certification reports are provided upon request to customers for the CRM concerned. For selected CRM the certification reports are published in the Internet.

### 6.2 Certificate

The information in the certificate complies with the recommendations of ISO Guide 31. The BAM guideline RLB-3.5 contains guidance on the preparation of certificates (Annex 2) and on CRM labelling (Annex 4).

For CRM that are issued jointly with external partners (e.g. EURONORM CRM or ERM), the applicable agreements concerning the contents and layout of certificates have to be considered.

Certificates are published in the Internet.

### **6.3 Handling by the Certification Committee**

Draft certificates and draft certification reports are submitted for comments to the Certification Committee and discussed comprehensively at a committee meeting face-to face with the CRM project leader who is invited.

In case of minor need of changes the drafts are approved subject to the agreed amendments, and release of the CRM (i.e. signing the certificate) is recommended.

In case of major need of changes a comprehensive revision of the drafts and re-submission are agreed.

In case of technical deficiencies, compromising the validity of certified values, the committee gives recommendations for further action (e.g. supplementary investigations).

*Note:* The Certification Committee can be asked for advice at any earlier time. This may help to avoid substantial supplementary investigation.

### **6.4 Release of the CRM**

The final versions of the certificate and the certification report are submitted to the head of department together with the recommendation of the Certification Committee.

The CRM is released by signing the certificate.

### **6.5 Labelling**

Mandatory data for labels are specified in the BAM Guideline RLB-3.5, Annex 2.

### **6.6 Metrological Utilisation (CMC Entries)**

Certified reference materials are important means to disseminate BAM's measurement capabilities to testing laboratories and may there be used to establish metrological traceability of test results. In the field of chemical analysis this metrological function of BAM CRM is supported by associated entries in the international CMC data base provided by the BIPM (see RLB-3.7).

CRM to be utilised in CMC entries have to comply with the following requirements:

(1) The certified properties must belong to the quantities in the scope of the CCQM (see <http://kcdb.bipm.org> – Appendix C, metrology area Amount of Substance), i.e. they must be related (directly or indirectly) to measurements of amounts of substance. For other quantities CMC entries can be obtained by agreement with the Physikalisch-Technische Bundesanstalt.

(2) The certified values must be based essentially upon BAM's own measurement capability. This means that:

- Homogeneity testing (as required) was carried out by BAM (stand-alone or in cooperation).
- Stability testing (as required) was carried out by BAM (stand-alone or in cooperation).
- The certification measurements were either carried out completely by BAM, or (in case of certification by interlaboratory comparison) BAM has participated in the certification study, and the BAM results agree with the certified values within uncertainty limits.
- The certification measurements were evaluated by BAM or according to BAM specifications.

(3) For the BAM measurement capabilities (measurement procedures) concerned, successful participation in international measurement comparisons (preferably CCQM key comparisons or pilot studies) must be demonstrated.

Applications for CMC entries are submitted to the BAM CCQM Committee. The further course of action is described in the BAM Guideline RLB-3.7 and in the QM manual of Department I (QMH-I-12.0).

## **7 Storage and Distribution**

The procedures for storage and distribution of reference materials comply with the recommendations of ISO Guide 34.

### **7.1 Storage**

Reference materials must be stored – separately from test materials and laboratory chemicals – in such way that any adverse effects on their quality as well as misuse and loss are excluded.

If particular storage conditions (e.g. cooling) were specified, compliance must be monitored and documented.

Where applicable, safety measures for occupational health and environmental protection are taken according to the relevant dangerous properties (toxic, flammable, explosive, radioactive etc.).

Access to rooms and facilities where CRM are stored, as well as withdrawal of CRM are regulated and documented in the divisions concerned.

### **7.2 Stability Monitoring**

For reference materials of stability class 2 and 3 (see section 5.4) long-term stability is monitored until the material is sold out. To this end, in the case of CRM from batch certification, packaging units are taken from the stock at appropriate intervals and investigated. CRM from single-unit certification require particular provisions.

If stability monitoring exhibits significant deviations from certified values, the CRM concerned is taken out of sale, and the previous customers of the CRM are notified.

If stability monitoring exhibits deviations that are larger than those predicted on the basis of the comprehensive stability investigation (see section 5.4) but still fall within the certified uncertainty limits, the following options apply:

- continued sale with a shortened utilisation period, or
- taking the CRM out of sale.

In any case the previous customers of the CRM are notified and the information is published via Internet.

If stability monitoring exhibits deviations that are substantially smaller than those predicted on the basis of the comprehensive stability investigation, the utilisation period may be extended. Also in this case the previous customers of the CRM are notified and the information is published via Internet.

### **7.3 Consultancy and Distribution**

Inquiries concerning individual reference materials are preferably answered by the leader of the CRM project concerned or by the contact person given in the Internet.

Complaints concerning a reference material are answered by the leader of the CRM project concerned or by the contact person given in the Internet, in agreement with the head of the division concerned. The respondent keeps records of any complaint.

Applicable warranty conditions are given in the Internet ([www.bam.de](http://www.bam.de) > Service > Webshop > GTC).

For shipping of reference materials the dangerous properties according to relevant regulations for the transportation of dangerous goods have to be taken into account.

For distribution of reference materials through a third party (authorized dealer) the applicable requirements of ISO Guide 34 (conditions for storage and shipping, customer records) have to be addressed.

### Normative Documents

- RLB-3.5, *Herstellung und Zertifizierung von Referenzmaterialien [Preparation and certification of reference materials]*
- RLB-3.7, *Wahrnehmung metrologischer Staatsaufgaben [National metrology functions]*
- ISO Guide 31, *Reference materials – Contents of certificates and labels* (Edition 2000)
- ISO Guide 34, *General requirements for the competence of reference material producers* (Edition 2009)
- ISO Guide 35, *Reference materials – General and statistical principles for certification* (Edition 2006)
- ISO/IEC Guide 43-1, *Proficiency testing by interlaboratory comparison – Part 1: Development and operation of proficiency testing schemes* (Edition 1997)

### Informative Documents

- *Durchführung und Auswertung von Homogenitätsuntersuchungen nach ISO Guide 35 [Performance and evaluation of homogeneity tests according to ISO Guide 35]* (2006, S. Noack)
- *CRM stability testing policies scrutinised* – Discussion paper for the Reference Materials Committee (2006, W. Bremser)
- W. Bremser, R. Becker, H. Kipphardt, P. Lehnik-Habrink, U. Panne, A. Töpfer, *Stability testing in an integrated scheme*, Accreditation and Quality Assurance, 2006, Vol. 11, p. 489 - 495

### Background Information

- *Referenzmaterialien in der BAM [Reference materials at the BAM]* – internal strategy document (2010, S. Recknagel, M. Hedrich, U. Panne), available from the intranet: [www.bam.de](http://www.bam.de) > Fachthemen [Expert information] > Referenzmaterialien [reference materials]
- BAM-Forschungsbericht 267 *Metrologie in der Chemie an der BAM – Aktivitäten im Rahmen der Meterkonvention [BAM Research report 267 Metrology in chemistry at the BAM – Activities in the framework of the Meter Convention]* (2004, W. Hässelbarth, Th. Steiger), available from: [www.bam.de](http://www.bam.de) > Service > Publications > BAM Research Reports (Online Version in German language)

Authors: W. Hässelbarth, Th. Steiger, Dept. I

Consultation – Task group for the implementation of ISO Guide 34: 2006-01-26, 2006-03-28

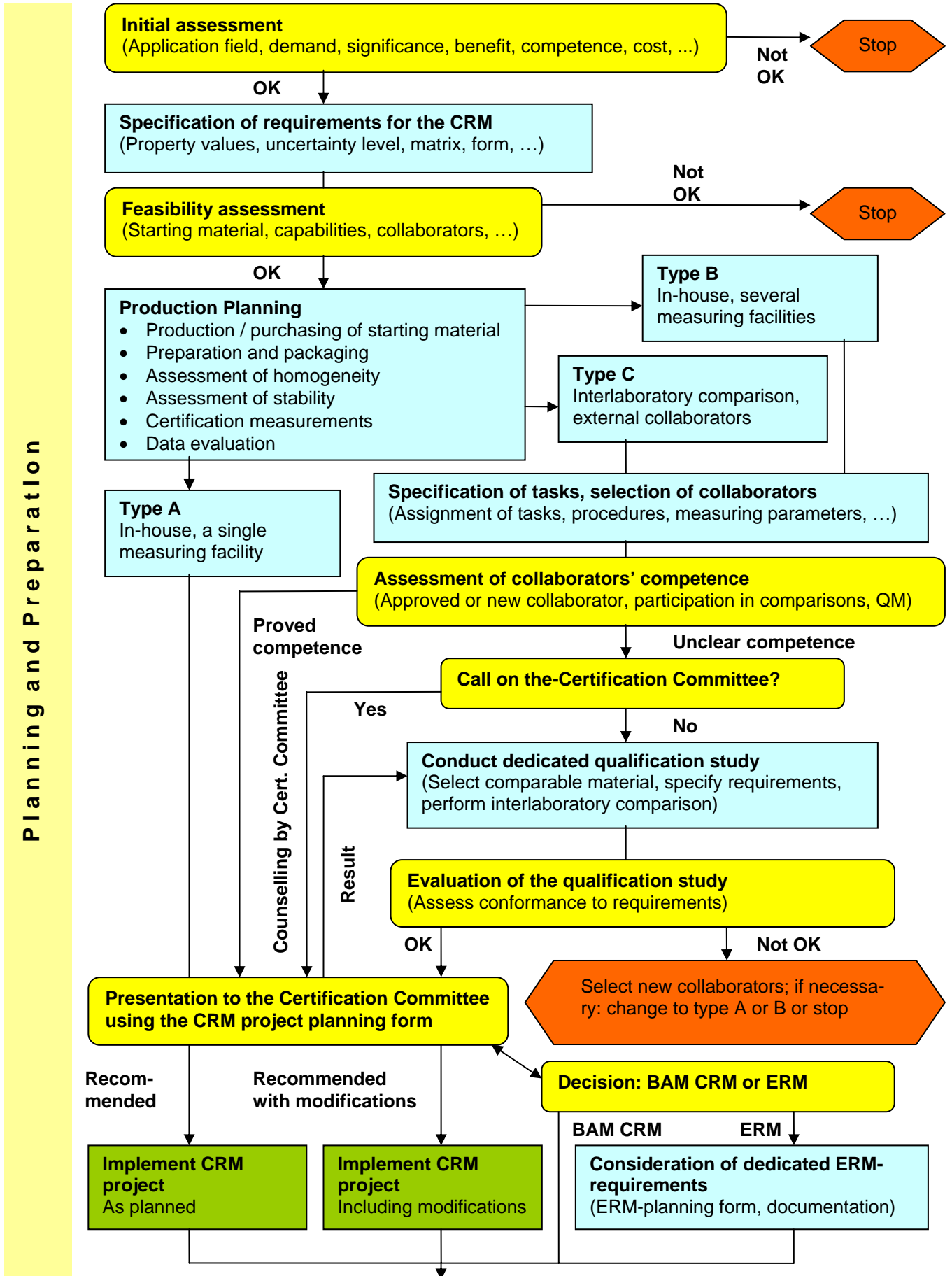
Consultation – Reference Materials Committee: 2006-02-14

Approval and release – Reference Materials Committee: 2006-06-20

**Annex A: Abbreviations and Acronyms**

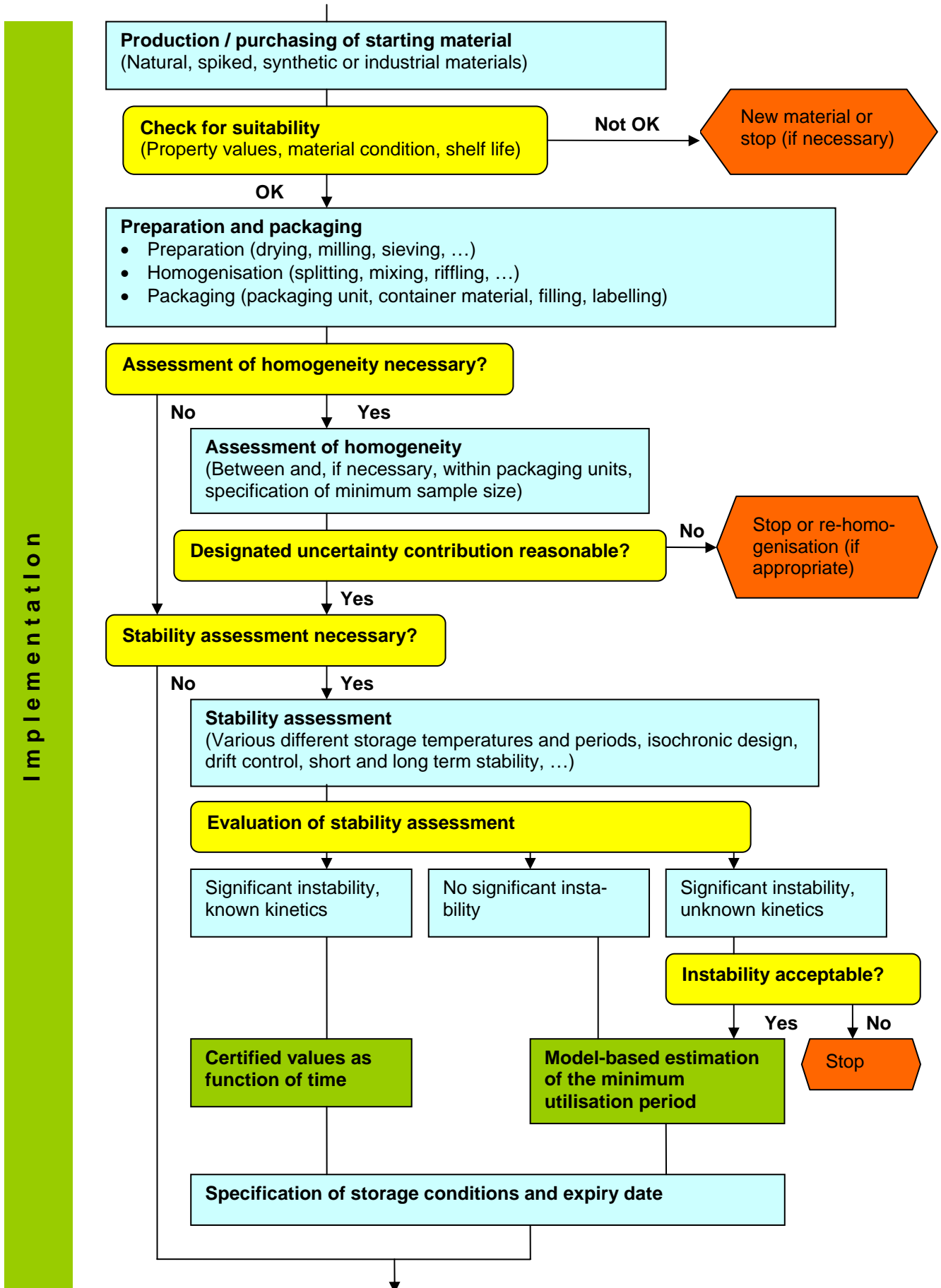
BCR	Former institution of the European Commission for the production of reference materials ( <u>B</u> ureau <u>C</u> ommunautaire de <u>R</u> éférence)
BIPM	International Office for Weights and Measures ( <u>B</u> ureau <u>I</u> nternational des <u>P</u> oids et <u>M</u> esures)
CCQM	Consultative Committee of the Meter Convention for metrology in chemistry ( <u>C</u> omité <u>C</u> onsultatif pour la <u>Q</u> uantité de <u>M</u> atière)
CIPM	International Committee for Weights and Measures ( <u>C</u> omité <u>I</u> nternational des <u>P</u> oids et <u>M</u> esures)
CIPM MRA	Multilateral agreement between the members of the Meter Convention on mutual recognition of national standards, calibration and measurement certificates of national metrology institutes ( <u>M</u> utual <u>R</u> ecognition <u>A</u> rrangement)
CMC	<u>C</u> alibration and <u>M</u> easurement <u>C</u> apability (see Appendix C of the CIPM MRA)
CRM	<u>C</u> ertified <u>r</u> eference <u>m</u> aterial
ERM	Registered trademark of the ERM Group (BAM, IRMM, LGC) ( <u>E</u> uropean <u>R</u> eference <u>M</u> aterial)
EURAMET	Association of the European metrology institutes ( <u>E</u> uropean <u>A</u> ssociation of National <u>M</u> etrology Institutes)
EURONORM	Consortium of the European Committee for Iron and Steel Standardisation
GTC	<u>G</u> eneral <u>t</u> erms and <u>c</u> onditions
GUM	<u>G</u> uide to the Expression of <u>U</u> ncertainty in <u>M</u> easurement
IEC	<u>I</u> nternational <u>E</u> lectrotechnical <u>C</u> ommission
IRMM	Research institute of the European Commission in Geel (Belgium) ( <u>I</u> nstitute for <u>R</u> eference <u>M</u> aterials and <u>M</u> easurements)
ISO	<u>I</u> nternational <u>O</u> rganization for <u>S</u> tandardization
LGC	Research institute of the United Kingdom in Teddington (formerly <u>L</u> aboratory of the <u>G</u> overnment <u>C</u> hemist)
QM	<u>Q</u> uality <u>m</u> anagement
RLB	BAM Guidelines Book ( <u>R</u> icht <u>l</u> inien <u>b</u> and)

**Annex B: Flow Chart for CRM Projects (Part I)**

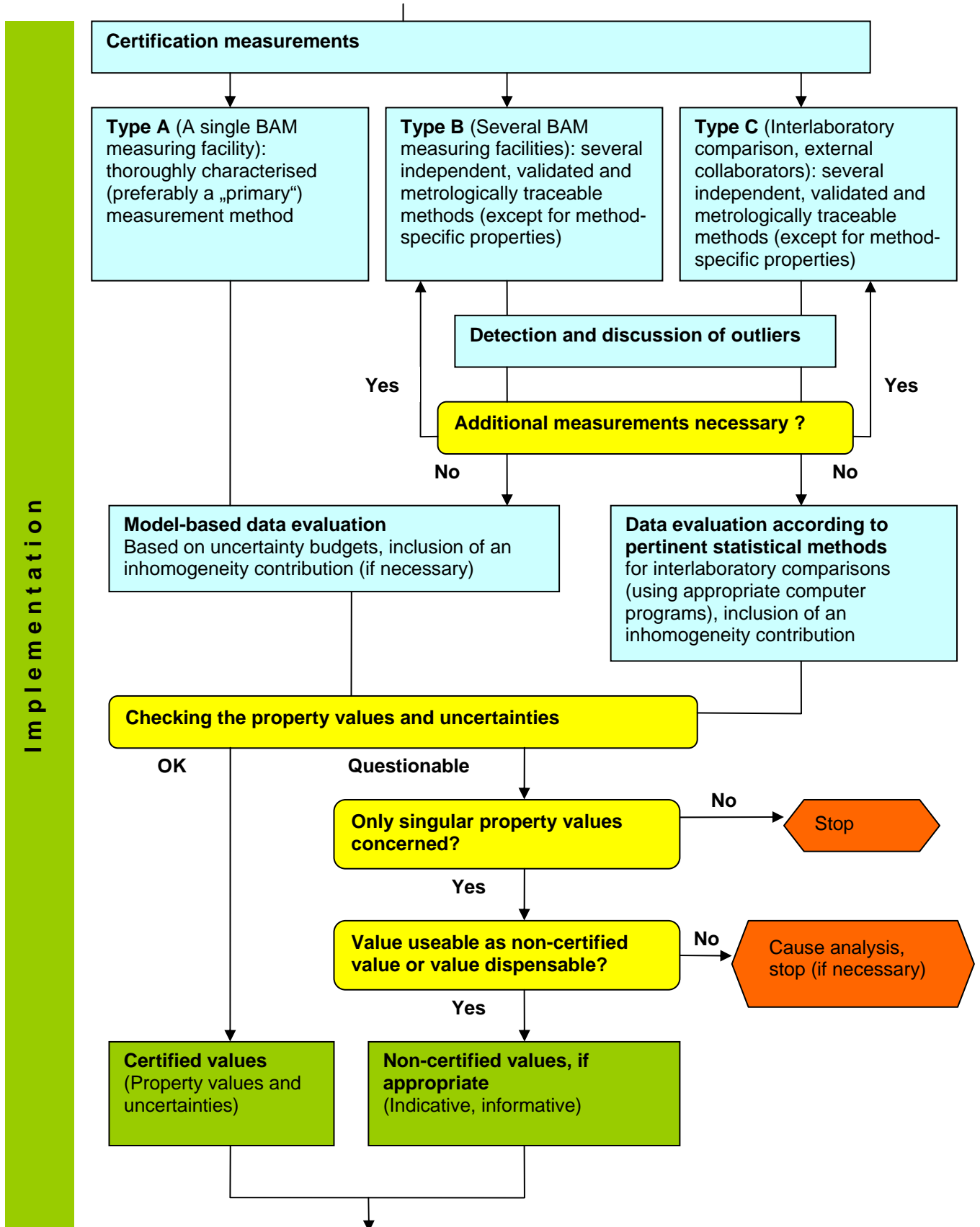


Planning and Preparation

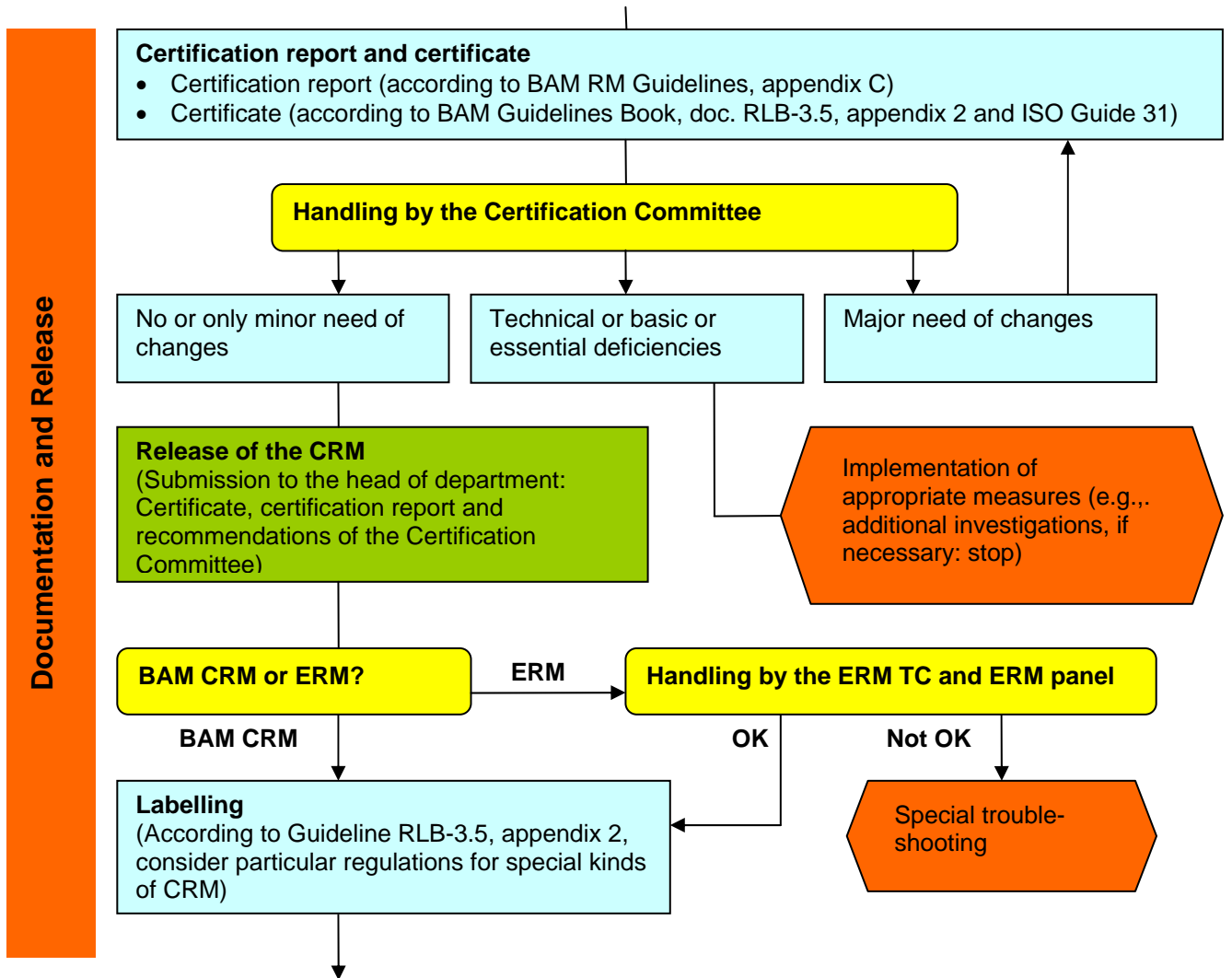
Annex B: Flow Chart for CRM Projects (Part II)



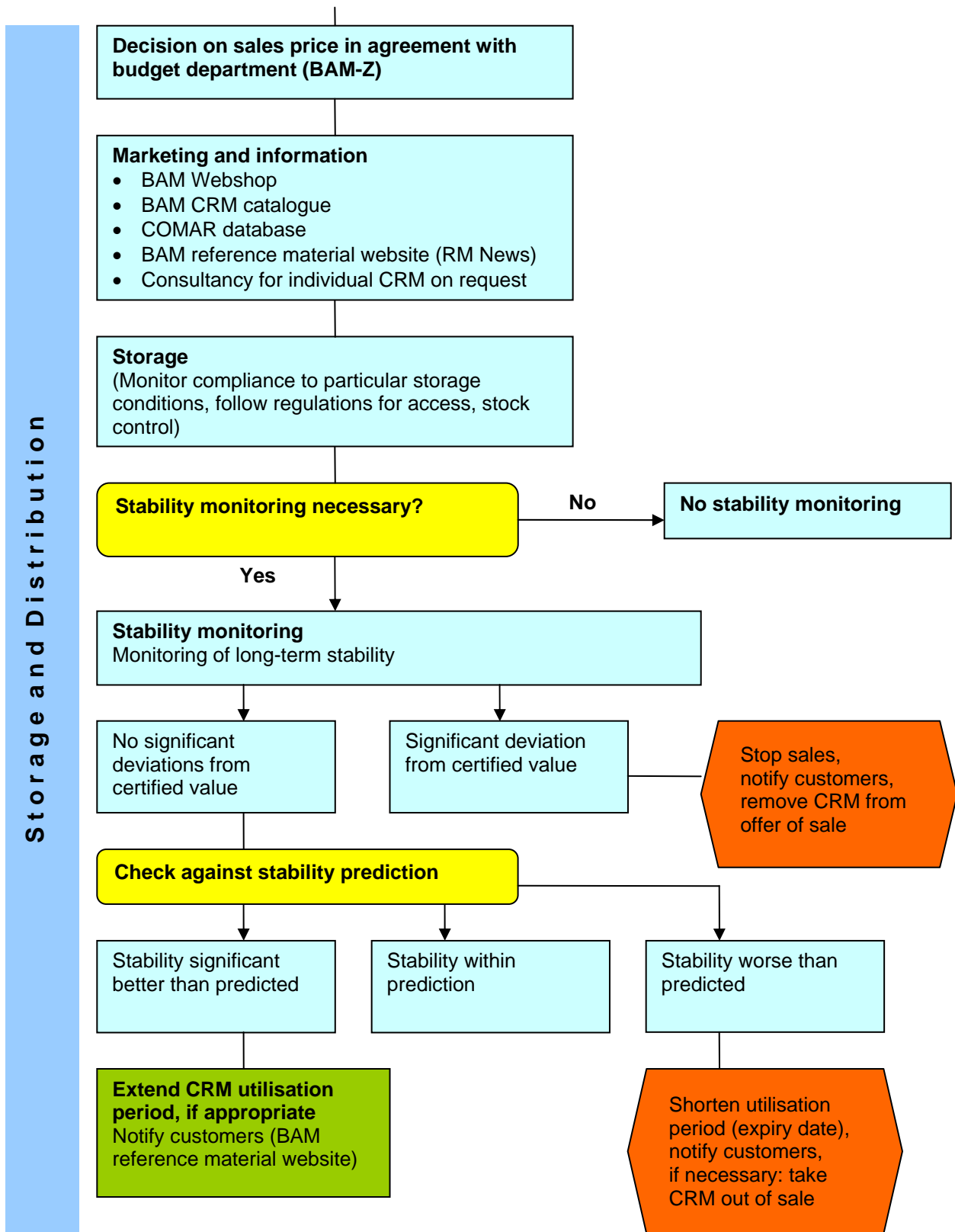
**Annex B: Flow Chart for CRM Projects (Part III)**



**Annex B: Flow Chart for CRM Projects (Part IV)**



**Annex B: Flow Chart for CRM Projects (Part V)**



## **Annex C: Model for Structuring Certification Reports**

Summary

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