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Developing a Hierarchical Structure for Reference Materials Traceability in the Russian Federation

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In Russia the system of reference materials of composition and properties of substances and materials (RM_s) forms one of the components of the Russian State system of measurements (SSM), the legal basis of which is the Law «On provision of Measurement Unity» [1] adopted in 1993. The Law regulates the interrelations between the State management bodies of Russia and juridical and physical persons on metrology issues.

Explanation of some terms is given in the table 1.

The general schematics of the RM system arrangement is illustrated in fig.1.

The RM system is respectively governed by "Gosstandart" of RF – the Federal level, metrology departments of Federal bodies of executive power - the Branch level, metrology departments of enterprises – the enterprise level.

According to [2], RM is a means of measurements in the form of a specified quantity of a substance or a material intended for reproducing and keeping unit sizes that describe the composition or property of this substance (material) the values of which are established as a result of metrologic certification and approved as a RM according to the adopted order. They are the basic means providing the measurement unity and are basically used for:

- metrologic certification of procedures of measurements (PM);
- graduation of measuring instrument (MI);
- certification and calibration of MI;
- control of PM uncertainty in the process of the application according to algorithms established in PM, as well as for other kinds of metrologic control;
- certification of other RM_s by the method of intercomparison (assignment of values).

RM is a specific measuring instrument; it cannot be fully referred to as measures (standards). The specific character of RM_s is expressed in the following:

- RM most commonly preserves all the basic characteristics of a substance when used to assess the values;
- In most cases RM preserves the fundamental attribute of any substance – its quantitative divisibility into parts, portions with each preserving the values certified;
- A wide variety of RM_s reproduce not a single value but a whole series of them /for instance, the contents of several components in a substance having a complex composition/. Each value characterizing the quantitative contents of the components is as a rule assigned with different values of uncertainties. It is difficult for this "measure" to determine the accuracy level and its echelon in the "vertical" hierarchy of the size unit.

Among the whole complex of goals related to the provision of the measurement unity the basic two ones are to be noted, namely:

1. reproduction of unit;
2. transfer of unit size.

To measure the concentration of a substance (content, composition) the former of the above goals involves a problem since it is not possible to produce a standard that would be similar to a mass standard, for instance. A standard cannot be a starting point of reproducing units of concentration (composition).The basis of reproducing units such as this one is apparently formed by absolute methods that are based on measurements of the main physical units that are related to concentration(composition) by the known theoretical dependence .

The rather substantial feature of the great majority of RM_s is believed to be the fact that they reproduce the values measured by indirect methods. In other words, these values cannot be established by a direct transfer of a unit size from the measuring instruments being at the top of the metrologic hierarchy. Therefore, the metrologic characteristics of RM_s are determined by the independent certification methods, i.e., formally, as is the case of unit sizes reproduced by standards.

Thus, in terms of the metrologic functions RM_s, if used for graduation of MI, certification and calibration of MI, measurements by the method of intercomparison (in other words, when the certified values of RM are used for the transfer to another MI) plays the part of measures but differs from the latter by the fact that reproduces the sizes of various parameters of substances including those that do not belong to the category of the units of physical values.

According to the State standard 8.315 [2] in Russia RM_s are categorized in terms of their adoption level and sphere of application. As for the above attributes RM_s are subdivided into the following categories:

- Inter-State ones ($ISRM_S$) used in CIS;
- State ones (SRM_S) applied within the State and provided by inter-branch deliveries;
- Branch ones (BRM_S) basically applied within a branch and provided by intrabranch deliveries;
- RM_S of enterprises (ERM_S) applied basically within a single enterprise.

The State standard of Russia "Gosstandart" is carrying on its activities relating to construction of SRM_s via the Chief center of RM_s – Urals Research Institute of Metrology (UNIIM), Ekaterinburgh (fig.1) that is in direct contact with the branch systems of RM_s.

In Minatom, RF, the RM system is governed by the Metrology department of Minatom, RF, via VNIINM that is Minatom RF Lead institution on RM_s.

Fig.2 illustrates the main stages of the RM construction. The major activities of the Lead Institution (VNIINM) to produce RM_s are presented in fig.3.

In our view, the provision of measurement unity (for instance, in nuclear material control and accounting system) is absolutely necessary to acquire the needed reliable information. Proceeding from this fact, the vertical structure of the RM system contemplating the reproduction of material (substance) parameters from the higher echelon to a lower one is more favored compared to the horizontal one. This approach is depicted in fig. 4.

As a primary RM (or RM of 1-st category) having the highest accuracy a reference material may by certified the original materials for which are high purity substances. It is more reasonable to rank it as a SRM.

The next stage is a secondary RM (or RM of 2-nd category). To establish the metrologic characteristics of this RM the coordinated standard methods of destructive analysis have to be used. Here, PM_s are favored that allow the procedure effected uncertainties to be neglected. If it is not achievable then the characteristics of the certification method have to take into account the uncertainties of the highest RM as well as those proceeding from the application of specific PM.

The composition of those RM_s approaches that of the product being controlled. Therefore at this stage it is important to study thoroughly and eliminate the factors of a sample used for an assay influencing the results of measurements of the characteristic being certified. The certification involved measurements have to be implemented by the method of intercomparison by multiple measurements of a characteristic being certified in the highest accuracy RM and in a sample being certified as well as by the schema of a single factor dispersion analysis with the simultaneous establishment of the uniformity characteristic of a material subjected to certification. The uniformity characteristic is the most important one that is able of influencing substantially the total uncertainty of the certified value of RM. These RM_s are ranked as BRM_s .

These RM_s primarily serve to transfer size values to working RM_s (the next stage) that are ranked as ERM_s (3^d category) as well as:

- to certify non-destructive assay (NDA) methods to assess the possible systematic bias;
- to calibrate (to check the calibration adequacy) of measuring instruments used for NDA;
- to control the correctness of destructive PM.

In other words, in responsible measurements capable of influencing the quality of some rather a large group of future measurements and their inclusion into the schema of transferring the sizes of the certified values via implementing the vertical hierarchic structure. When putting this schema into practice $\delta_1<\delta_2<\delta_3$, where δ_i is uncertainty of the certified value of RM. In this case it is important for the numerical values of δ_i to be in the reasonable range.

One of the most important goals of nuclear material control and accounting is to control the contents of U, Pu and their isotopes in various kinds of materials (products, waste, in storage facilities, upon shipping etc.). For the control to be reliable it is advisable to have the above schema put into practice.

Currently, there is an agreement between the Russian institutions (VNIINM, IPPM and others) and institutions of USA on development of four RM_s of the content of:

- total U;
- total Pu;
- isotopic composition of low burn-up Pu;
- isotopic composition of high burn-up Pu.

In this instance the schema given in fig 4 is feasible in several versions. One of them is illustrated in fig 5.

VNIINM is responsible for the plan of the RM construction and its subsequent implementation. NBL and LANL shall give the methodic support of the activities.

In the process of working out the plan the data were acquired and analyzed as applied to the need in the RM_s used in the methods of destructive assay. At the meetings of RF and USA experts the technical and methodic issues inherent in putting into practice the Russian plan of the RM production were agreed upon.

The list of RM_s comprises:

- Uranium RM – U_3O_8 powder of natural enrichment. Certified characteristic – mass fraction of U, %;
- RM of total Pu content – PuO_2 powder. Certified characteristic–mass fraction of Pu, %;
- RM of Pu isotopic composition - low and high burn - up PuO_2 powder.

Certified characteristic - atomic fractions of Pu-238, 239, 240, 241, 242.

1. The certification uncertainties have to be established as an assessment range at the confidence level P = 0.95.

Conclusions

1. The All-Russia RM system and the RM system of Minatom, RF, have been reviewed.
2. Schematics options have been reviewed for reproducing the composition (concentration) units from highest accuracy RM to working one.

References

1. RF Law "On Provision of Measurement Unity", 28.04.93.
2. GOST 8.315. State System of Measurement Unity Provision. Reference materials of composition and properties of substances and materials. Basic regulations.

Table 1

Explanation of some terms

Term	Explanation
1	2
1. Measurement unity . _______o _________.	A state of the art of measurements when the results are expressed in legalized units of values and the uncertainty of measurements is within the established range at the specified probability.
2. Statement of work (SOW) ___________ _______ (__)	A document coordinated by interested parties witch depicts the purpose of RM_s, the required metrologic and technical characteristics, requirements for manufacture, application, delivery, commitments of parties etc.
3. Material of RM. ________ __.	An original material in one or another aggregate condition (powder, solid body etc). Specified in SOW.
4. Certification of RM_s /Metrologic certification of RM_s). __________ __ /______________ __________ __/	Examination of RM material aimed of finding the values of metrologic characteristics of RM.
5. Metrologic characteristics of RM_s. _______________ _________________.	The certified values of RM, uncertainty of certified values and oth.
6. Certified value of RM. _____________ ________ __.	The value of the RM certified characteristics established by its certification and given in the Passport (SRM) or other documents of RM (certificate).
7. RM characteristic being certified. ___________ _________________.	The value or dependence of two values that characterize the composition or properties of a substance (material) of RM the values of which are to be established by certification of RM.
8. Metrologic expert examination (ME) _____________ __________ (__).	Examination of documents on RM developments (SOW, records of examinations, reports etc) aimed at finding out whether the taken decisions meet the established metrologic rules and norms.

State national standard of Russia (Gosstandard) Main national center of RM_s of Russia (UNIIM, Ekaterinburgh)	<u>Federal Level</u> State System of RM_s ($SSRM_s$)
Minatom, Russia Lead institution on RM_s (VNIINM) Base RM_s institutions	<u>Branch Level</u> Branch Systems of RM_s RM System of Minatom
Enterprises designated to produce RM_s	<u>Enterprise Level</u> RM System of an Enterprise

Fig. 1. General schematics RM System in Russia

1. Development of statement of work (SOW) for RM_s
2. Metrologic expert examination of SOW
3. Manufacture of RM_s
4. Certification of RMs.
    - 4.1. Investigation of RM_s.
    - 4.2. Processing results of investigation.
    - 4.3. Metrologic expert examination of results of RM investigations.
    - 4.4. Issue of RM documentation.
5. Registration.
6. Delivery of RM specimens to customers.

Fig. 2. Basic stages in RM construction

RM construction

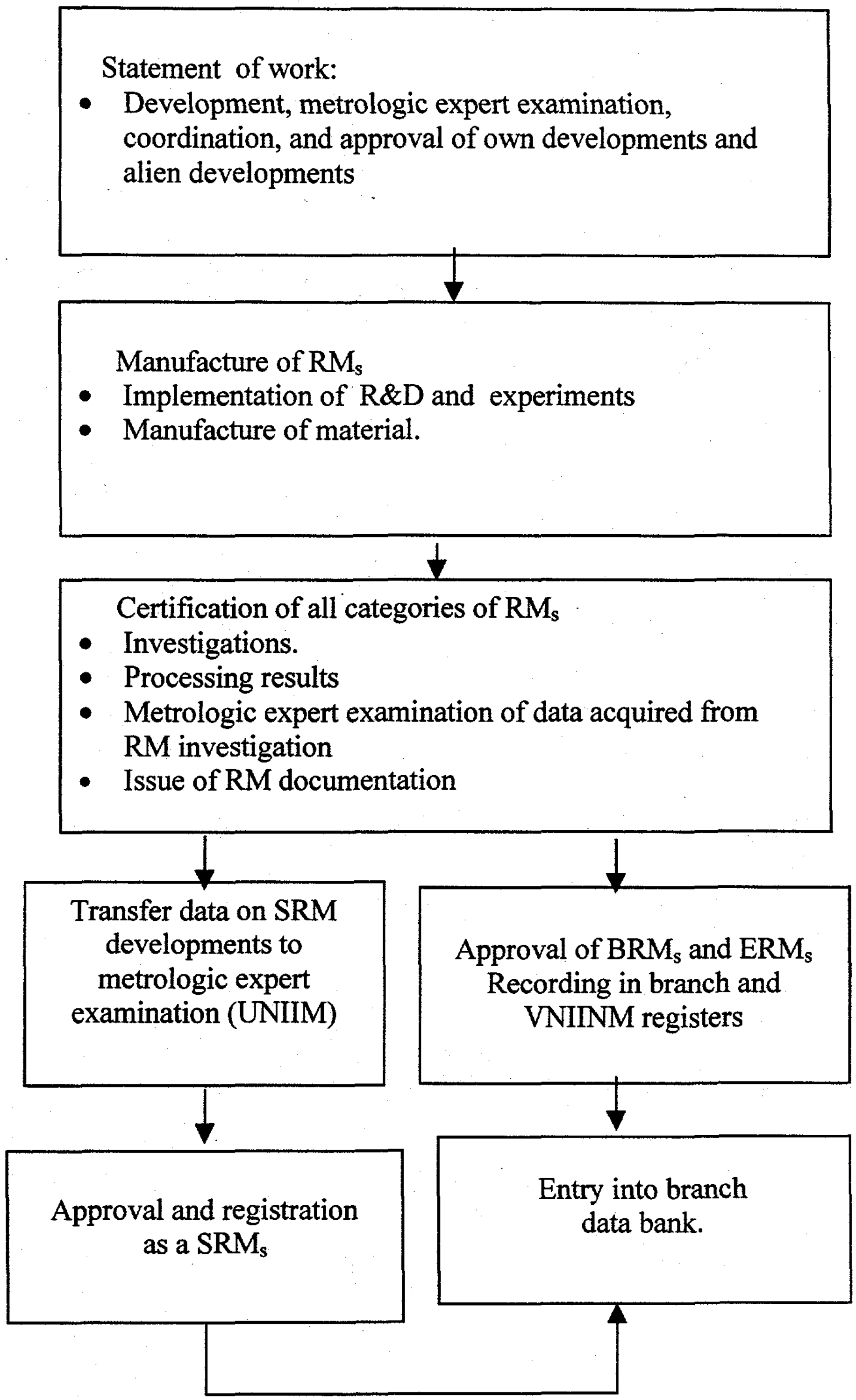
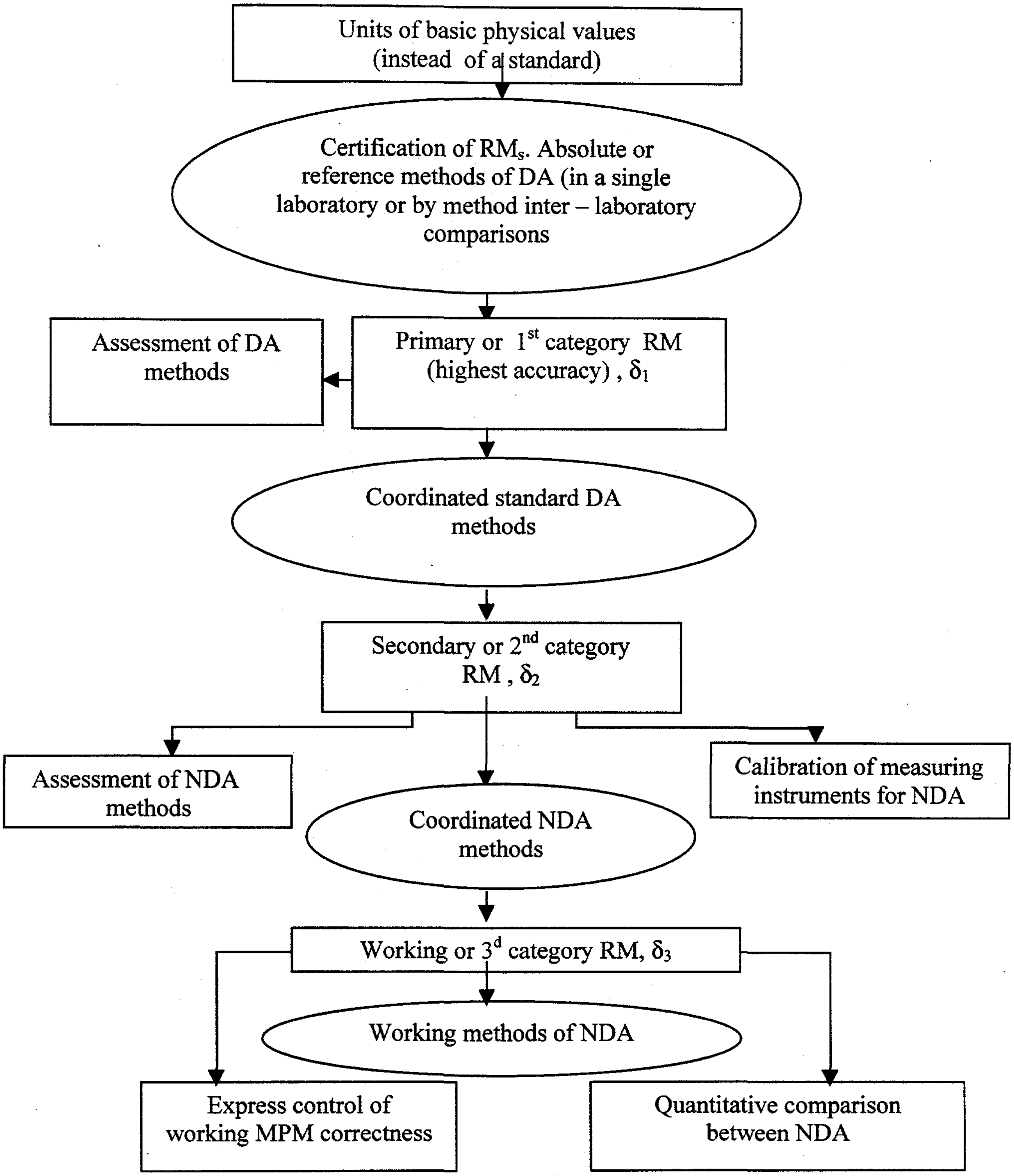


Fig. 3. Basic functions of SSC RF VNIINM in constructing RM_s



* Absolute methods: gravimetry (mass unit) – Law of constant and multiple proportions ;
Coulometry (unit of electricity and mass);
Potentiometry (unit of voltage) – Nernst law.

** Reference method – high reliability independent one; mass – spectrometry can by placed into this category.

δ_i – uncertainty of RM certification.

Fig.4. Schematic representation of reproducing units of concentration (composition) from highest accuracy RM to working RM

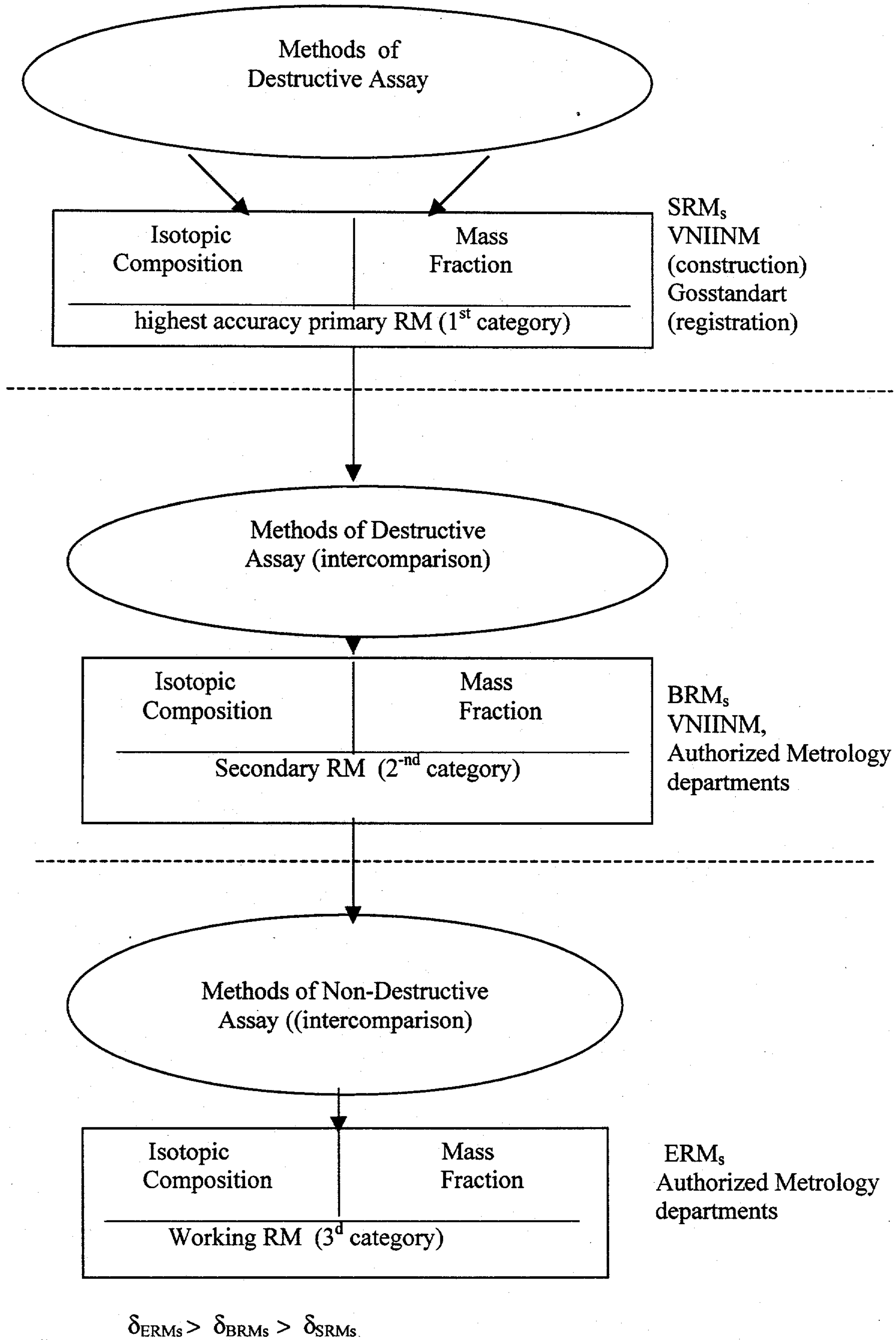


Fig. 5. Schematics options for reproducing units of composition (concentration) of RM_s of U and Pu containing materials