

International Target Values 2000 for Measurement Uncertainties in Safeguarding Nuclear Materials

H. Aigner, R. Binner, E. Kuhn

IAEA, P. O. Box 100, A-1400 Wien, Austria

U. Blohm-Hieber

European Commission, Euratom Safeguards Office, Plateau du Kirchberg, L-2900 Luxembourg, Luxembourg

K. Mayer (ESARDA WGDA)

European Commission, JRC, Institute for Transuranium Elements, Postfach 2340, 76125 Karlsruhe, Germany

S. Guardini (ESARDA WGDA)

European Commission, JRC, Institute for Systems, Informatics and Safety, I-21020 Ispra (VA), Italy

C. Pietri (ANSI/INMM)

HiTech Consultants, 5506 Grand Ave., Western Springs, IL 60558, USA

B. Rappinger (ISO/TC85/SC5/WG1)

Westinghouse AB, BQC, SE-72163 Vasteras, Sweden

B. Mitterrand (ISO/TC85/SC5/WG3)

COGEMA La Hague, Beaumont-Hague CEDEX F-50444, France

J. Reed (ISO/TC85/SC5/WG12)

BNFL Sellafield, B229 Sellafield, Seascale, Cumbria, CA201PG, United Kingdom

O. Mafra-Guidicini (ABACC)

ABACC, Av. Rio Branco 123/515, Centro, Rio de Janeiro, RJ 20040-005, Brazil

S. Deron

Consultant, IAEA, P. O. Box 100, A-1400 Wien, Austria

Abstract

This issue of the International Target Values (ITVs) represents the fifth revision, following the first release of such tables issued in 1979 by the ESARDA/WGDA. The ITVs are uncertainties to be considered in judging the reliability of analytical techniques applied to industrial nuclear and fissile material, which are subject to safeguards verification. The tabulated values represent estimates of the "state of the practice" which ought to be achievable under routine measurement conditions. The most recent standard conventions in representing uncertainty and reliability data have been considered, while maintaining a format that allows comparison with the previous releases of the ITVs. The present report explains why target values are needed, how the concept evolved and how they relate to the operator's and inspector's measurement systems. The ITVs 2000 are intended to be used by plant operators and safeguards organizations, as a reference of the quality of measurements achievable in nuclear material accountancy, and for planning purposes. The report suggests that the ITVs can be used with benefit for statistical inferences regarding the significance of operator-inspector differences whenever valid performance values are not available.

1. Introduction

Safeguarding nuclear material involves a quantitative verification of the accountancy of fissile materials by independent measurements. The effectiveness of these verifications depends to a great extent upon the quality of the

accountancy measurements achieved by both the facility operator and the safeguards inspectorate. For this reason a typical model of Safeguards Agreements^[1,2] stipulates that:

"The Agreement should provide that the system of measurements on which the records used for the preparation of reports are based shall either conform to the latest international standards or be equivalent in quality to such standards".

Although the above requirement was directed to the facility operators, it indeed applies equally well to the safeguards inspectorates.

In the absence of relevant international standards of measurements, the International Atomic Energy Agency (IAEA) had defined in the 1970s a set of international standards of nuclear material accountancy^[3], which lists the "expected measurement accuracy associated with the closing of a material balance" at five different types of nuclear facilities. However, these values have never been reviewed despite numerous technological changes since their adoption by consensus by a group of experts designated by their Governments. Safeguards officials and evaluators but also plant measurement specialists need more current and informative references regarding the performance capabilities of measurement methods used for the determination of the volume or mass of a material, for its sampling, its elemental and isotopic assays. Such informa-

tion is needed for the various nuclear materials encountered in the nuclear fuel cycle.

The Working Group on Techniques and Standards for Destructive Analysis (WGDA) of the European Safeguards Research and Development Association (ESARDA) pioneered the way in 1979 by presenting a list of "Target Values" for the uncertainty components in destructive analytical methods^[4] to the safeguards authorities of Euratom and of IAEA. Revised estimates were prepared in collaboration and published as the 1983 Target Values^[5] after four years of extensive discussion and consultation with and within operators' laboratories and safeguards organizations. The international acceptance of the concept grew further with the next review, which involved, besides the ESARDA/WGDA and IAEA, the active participation of the members of two specialized committees of the Institute of Nuclear Materials Management (INMM). The 1987 Target Values, published as a result of this review^[6], defined, like the previous editions, the values of "random" and "systematic" error parameters to be aimed for in elemental and isotopic analyses of the most significant types of materials using common destructive analytical methods. The same groups took a new step when they agreed to define with the 1988 edition^[7] the values of the random error parameter to be met in the elemental assays as a result of sampling. Unfortunately, it was not possible at this time to include values for sampling uncertainties arising from systematic effects.

Following a 1988 recommendation of the IAEA Standing Advisory Group on Safeguards Implementation (SAGSI), the IAEA convened a Consultants Group Meeting in June 1991 to provide expert advice on international standards of measurements applicable to safeguards data. A concept of International Target Values (ITVs) was proposed on the model of the 1988 ESARDA Target Values and included estimates of the "random and systematic error" uncertainties originating from the measurements of volumes or masses of nuclear materials. The scope of ITVs was also extended to include a consideration of the non-destructive assay methods (NDA) which had won acceptance as accountancy verification tools.

Specialists from four continents took part in the discussion of the proposed concept. The ESARDA/WGDA held joint meetings with the ESARDA Working Group on NDA methods (ESARDA/WGNDA). The IAEA organized a series of Consultants Group Meetings with the participation of a representative from a large European reprocessing plant, of Brazilian and Japanese nuclear national authorities along with representatives of ESARDA, INMM, the International Organization for Standardization (ISO), the European Commission (EC) and IAEA inspectorates. The result was the publication of an IAEA Safeguards Technical Report in March 1993, titled "1993 International Target Values for Uncertainty Components in Fissile Isotope and Element Accountancy for the Effective Safeguarding of Nuclear Materials"^[8]. Articles in the ESARDA Bulletin^[9] and in the Journal of the INMM^[10] widely publicized the IAEA technical report. The report itself was translated into Japanese^[11].

International experts and panels have now reviewed the experience gained with the use of the 1993 ITVs and the progress made since 1993 in accountancy and safeguards verification measurements. These include ESAR-

DA/WGDA, ESARDA/WGNDA, the Institute for Nuclear Material Management (INMM), the Japanese Expert Group on ITV 2000, Working Groups of the International Standardization Organization (ISO) dealing with analytical measurements in nuclear fuel industry and the Brazilian/Argentinean Agency of Accountability and Control of Nuclear Materials (ABACC). This report contains the changes made in the presentation of the ITVs and in some of the target values to reflect the latest recommendations of the experts.

An effort was made to bring the nomenclature in line with the latest recommendations of ISO^[12], the National Institute of Standards and Technology (NIST)^[13] and the European Association of Chemical Measurements (EURACHEM)^[14]. A clear distinction for example is made between the meaning of the term "error" and the term "uncertainty". The ITVs 2000 indeed represent target standard uncertainties, expressing the precision achievable under stipulated conditions. These conditions typically fall in one of the two following categories: "repeatability conditions" normally encountered during the measurements done within one inspection period; or "reproducibility conditions" involving additional sources of measurement variability such as "between inspections" or "between laboratories" variations.

As in earlier publications the values listed in the present document have been derived from an evaluation of actual measurement data. Four sources of information were considered. The most relevant and complete set of measurement data still comes from the information gathered by safeguards inspectorates during the statistical evaluation of the results of the measurements reported by the facility operators and the results of independent measurements performed on the same materials by the inspectors^[15,16]. This approach will be referred to as the "top-down" approach. These data were complemented and confirmed by "bottom-up" assessments of measurement uncertainty components published by measurement specialists^[17-26] and derived according to the ISO^[12], NIST^[13] and EURACHEM^[14] guides. In addition and whenever possible, it was verified that the proposed ITVs were consistent with the results of laboratory intercomparisons^[27-33] or measurement quality evaluation programmes^[34-48]. In cases where little or no statistical data was available (particularly for sampling uncertainties), some values were defined on the basis of expert opinion.

The ITVs 2000 bear a date like the ESARDA Target Values and 1993 ITVs issued previously. This reflects the experience that the quality of measurements may improve with the development of newer methods and instruments. ITVs also reflect the current understanding of the structure of the uncertainty components in nuclear material accountancy measurements. Changes can also occur in the future as this understanding improves or varies.

As with the previous lists, the ITVs 2000 should be achievable from today forward under the conditions normally encountered in typical industrial laboratories or during actual safeguards inspections. They do not represent the measurement uncertainties, which would only be achieved under exceptional or ideal laboratory conditions, or with most recently developed methods, which have not yet found wide use for daily and routine measurements.

Significant changes in the application of instruments and techniques have taken place since the previous edition. Measurements with instruments like high level neutron coincidence counters (HLNC), K-edge X-ray absorptiometer and fluorescence analyzers (HKED) are used routinely at the plants by inspectors with great success, not only to detect partial defects but also to verify the flow and balance of nuclear materials. This has allowed to decrease strongly the fraction of items, which need to be verified by chemical analysis. The latter methods are used now mainly for verifying the quality of operators' measurement systems and the absence of small but measurable biases in the closing of the material balances. Here, improvements were also observed with the combined use of Large Size Dried Spikes (LSD) and thermal ionization mass spectrometers with multidetectors and total sample evaporation for the verification of the uranium and plutonium content in spent fuel solutions and U/Pu fuel materials by isotope dilution mass spectrometry (IDMS).

It is expected that the ITVs 2000 will continue to be a motivating goal for beginner laboratories and a reasonable reference for experienced laboratories and safeguards evaluators. With the growing acceptance of modern quality assurance concepts it is suggested that the ITVs 2000 can also constitute a good reference against which analytical laboratories would validate their measurement system.

2. Safeguards Accountancy Verification Measurements

As evident from the title of the report and the introduction, the principal application of the ITVs should be in safeguards activities. The safeguards verification data also form the major source of information on which the ITVs are based. A description of the origin of the safeguards data is therefore relevant.

Figure 1 describes the basic measurement scheme followed in safeguards measurement verifications. For each inspection, j , the inspector selects, in accordance with a random sampling plan, the items or batches of nuclear materials to be verified by an independent measurement. The inspector then compares the result of his verification measurement, Y_{ij} , to the result, X_{ij} , which the operator has obtained on the same batch or item i , and which the operator has declared to the inspectorate. The ability of the inspector to detect whether the difference d_{ij} is significantly different from zero depends upon the overall uncertainties in the results X_{ij} and Y_{ij} . Figure 1 identifies the major steps of the measurement process where uncertainties can arise, although not all steps may be relevant for every method (e.g., several of the steps may be omitted or combined under a single step for NDA methods).

Step 1 corresponds to the measurement of the volume or mass of the item or batch of material. This so-called "**bulk**" measurement, when needed, takes place in the plant area and involves a calibration procedure.

Step 2, the "**sampling**", involves removing, for the purpose of the analytical measurement, a representative portion of the material from the batch or item to be analyzed. This portion may be a complete item in the case of an NDA measurement. This step is also done in the plant area.

Step 3 concerns the precautions which must be taken in the way the sample is "**conditioned**" and packaged at

the sampling station so that all characteristics to be measured are preserved during its transport to the location or laboratory where the characteristics will be measured^[49].

Step 4, the "**shipment**", is the transport of the sample to the location where it can be measured. This is rarely a trivial operation even when the movement is very short, as in the case of an NDA measurement, which is often done practically on the spot.

Step 5, the "**treatment**", is intended to bring the sample into the most appropriate geometrical, physical and/or chemical form for the measurement. This step is skipped when a complete item is subject to an NDA measurement. The treatment of a sample taken for destructive analysis may involve a sequence of individual steps, such as subsampling, dissolution, dilution, spike or standard addition, chemical treatment or chemical separation, etc.

Step 6, represents the "**measurement**" itself. In general terms, a measurement is based on a calibration from which the parameters linking the observed signal and the measurand are determined. Typical examples are HLNC calibration curves for Pu mass determination, calibrations of Gamma Spectrometers for ²³⁵U abundance determination or the determination of the mass-discrimination correction factor for a mass-spectrometer. The standardization of a titrant solution is another example of a calibration, although it is frequently not recognized as such. Calibration functions may be as simple as a single calibration factor (actually representing a straight line through the origin), or may be complex and represented by an empirically determined calibration curve. Calibrations based on recognized references, such as certified reference materials or well-known physical constants, establish the traceability chain between the measurement result and the International System of Units (SI). Calibrations may be valid and used without modifications for long times, repeated on a daily basis or even performed with each individual measurement. Sometimes, calibration is performed in two steps: elaborate calibration exercises for determining the fundamental characteristics of the calibration function are combined with more frequently repeated "normalization" measurements to correct for short-term effects or minor deviations from the overall calibration function at the specific working range.

Step 7, the "**calculation**", consists in transforming the results of the physical or chemical measurement obtained in the preceding step into an estimate of the amount of fissile element or isotope in item or batch i . Particularly when the operator and inspector use DA, this involves estimating the total element content, by combining the result of the bulk measurement w_{ij} with the elemental concentration c_{ij} . In the case of Uranium materials, this is combined with the isotope abundances f_{ij} of the fissile isotope (²³⁵U or ²³³U) to yield a measure of the amount of fissile isotope in item or batch i , according to equations (1) and (2), respectively.

$$X_{ij} = w(O)_{ij} \cdot c(O)_{ij} \cdot f(O)_{ij} \quad (1)$$

$$Y_{ij} = w(I)_{ij} \cdot c(I)_{ij} \cdot f(I)_{ij} \quad (2)$$

Every stage of the process, starting with bulk measurements must be performed under well-controlled conditions. Hence quality control measures are imperative at every step of the process. Quality control on sampling can be

done by taking replicate samples after different mixing times or taking samples from a number of items of the same batch of bulk materials. Quality control materials or samples can be introduced at specific steps to monitor the quality of the whole process or any part of it, including the conditioning and shipment steps. Figure 1 shows an example where control materials are used independently by the operator and the inspector to check the quality of the processes following the sampling. Quality control measures should be performed in the frame of a documented quality system^[49-53].

The uncertainties in the measurements of element concentrations and isotope abundances in the ITVs refer to the combined effects of the uncertainties in Steps 3 to 6 occurring after the taking of the sample in Step 2.

Step 8, the "reporting" of the results, is purely clerical but unfortunately it can be a source of errors. Uncertainties arising from such errors are not considered in the ITVs proposed in this document. Yet it is essential that appropriate quality assurance measures be taken to avoid the occurrence of clerical errors.

When NDA is used the attention focuses most on the measurement (Step 6) as the preceding steps have usually less impact or may even be omitted. For example, bulk measurements and sampling are not needed if the NDA method allows direct measurement of the total amount of fissile element or isotope contained in a whole item or batch of nuclear material, as with various neutron counters or calorimeters.

3. Evaluation of Safeguards Accountancy Verification Measurements

The statistical terms used in this document are defined in Annex 1 in an effort to promote the understanding of the statistical concepts used here.

The safeguards inspectors examine the operator-inspector paired differences to determine whether these remain within upper and lower limits, which are commensurate with the characteristic uncertainties of the operators' and inspectors' measurement systems. For this purpose, the inspectors need to quantify the major uncertainties of the actual data collected during their verification activities.

Two categories of uncertainties play an important role in planning for inspections and in drawing inferences from inspection data: uncertainties due to repeatability effects, which are of a purely random nature, and uncertainties resulting from systematic effects within a given set of data, corresponding to an inspection period^[54,55]. These uncertainties will be designated by the symbols **u(r)** and **u(s)**, respectively:

- **random uncertainty components, u(r)**, are due to errors varying in an unpredictable way among individual items or results. Counting statistics or the repeatability of measurements within a short period of time under constant conditions are typical examples for random uncertainty sources. Simply stated, the effects of random uncertainties can be reduced by repeated measurement, sampling and analysis, but it is not possible to correct for random errors.
- **uncertainty components of a systematic character, u(s)**, are due to errors affecting an entire group of

items in the same way, like all measurement results interpreted with the same calibration curve, normalized with the same normalization experiments, or affected by the same background subtraction. But also uncertainties in the certified values of reference materials, nuclear data uncertainties or constant instrument or laboratory biases will appear to have a systematic character. The effects of uncertainties of a systematic character cannot be reduced by repetition under a fixed set of conditions encountered during a given inspection period. The cause of systematic errors may be known or unknown. If both the cause and the value of a systematic error are known, it can be corrected for, but there will still remain an uncertainty component of systematic character, which is associated with this correction.

A basic assumption is that $u(r)$ and $u(s)$ are characteristics of the type of material, its chemical and physical form and of the method of measurement. A further assumption is that the component of systematic character, $u(s)$, is constant for a given inspection period, but that it varies in a random manner from one inspection to another, for both the operator and the inspector.

Consequently, the inspectors group the data pairs originating from one inspection period, j , by material balance areas (MBA), by strata of materials of similar characteristics and by measurement methods^[56]. For a given MBA and stratum, call:

$$d_{ij} = (X_{ij} - Y_{ij}) / X_{ij} \quad (3)$$

the operator-inspector difference, d_{ij} , for item i in inspection j , with

$$\begin{aligned} i &= 1, 2, \dots, m_j \\ j &= 1, 2, \dots, K \end{aligned}$$

Note: *to simplify the presentation, relative differences are treated here. In practice, absolute differences, $(X_{ij} - Y_{ij})$, would be used when the size of the items of a given stratum vary widely.*

The assumed error model is

$$d_{ij} = d + \Delta_j + \varepsilon_{ij} \quad (4)$$

where

- d is the mean difference over the K inspections,
- Δ_j is the systematic error of the operator-inspector difference during inspection j , and
- ε_{ij} is the random error of the operator-inspector difference for item i during inspection j .

The expected values of Δ and ε are both zero (i.e., they are both centred random variables in a statistical sense). An analysis of variance components of the operator-inspector differences, d_{ij} , according to this model equation gives estimates of the variance $s^2(\varepsilon)$ of the random component and of the variance $s^2(\Delta)$ of the component due to systematic effects within the given inspection period^[56-59]. In performing this analysis of variance components, it is generally assumed that Δ_j and ε_{ij} are normally distributed and that the variances of the random error are the same for all inspections. The set of results are therefore screened for outliers prior to performing this evaluation.

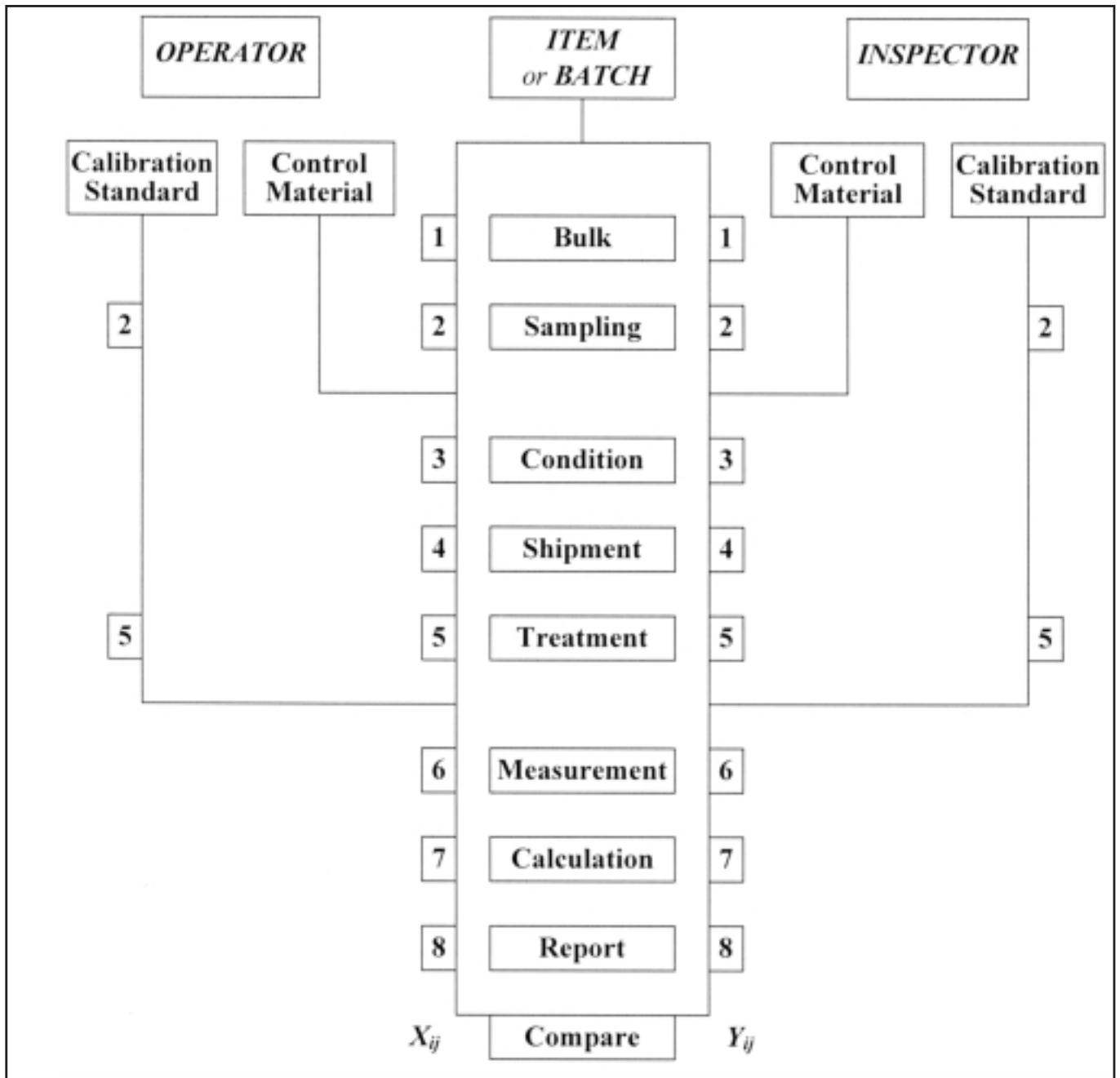


Figure 1: Accountancy and Verification Measurement Scheme for item *i* during inspection *j*

Paired comparisons of this type are done separately for bulk measurements, element concentrations and isotope abundances, as well as for the masses of fissile elements and isotopes. One obtains, for each type of measurement, an estimate of the combination of the actual uncertainty components for the operator's and inspector's measurement systems:

$$s^2(\epsilon) = u^2(r,O) + u^2(r,I) - 2r(\epsilon) u(r,O) u(r,I) \quad (5)$$

$$s^2(\Delta) = u^2(s,O) + u^2(s,I) - 2r(\Delta) u(s,O) u(s,I) \quad (6)$$

where

- $u(r,O)$ and $u(r,I)$ are the standard uncertainties due to random error components for the

operator and the inspector respectively,

is the Pearson correlation coefficient between the operator's and the inspector's random errors,

- $r(\epsilon)$

and

- $u(s,O)$ and $u(s,I)$

are the standard uncertainties due to effects of systematic character for the operator and the inspector respectively

- $r(\Delta)$

is the Pearson correlation coefficient between the operator's and inspector's systematic errors.

It can be expected that the uncertainties of operator's and inspector's data have similar magnitudes when both are obtained with similar methods. Under the assumption that the errors of the operator and of the inspector are independent from each other, i.e.

$$r(\varepsilon) = r(\Delta) = 0, \quad (7)$$

the values

$$u^2(r,O) \approx u^2(r,I) \approx s^2(\varepsilon) / 2 \quad (8)$$

and

$$u^2(s,O) \approx u^2(s,I) \approx s^2(\Delta) / 2 \quad (9)$$

provide good estimates of the standard uncertainties.

In other situations operator's DA results may be compared with much less precise and/or much less accurate inspector's results obtained for example by some NDA methods. If, for example

$$u(r,I) \gg u(r,O) \quad (10)$$

and

$$u(s,I) \gg u(s,O) \quad (11)$$

Then,

$$s(\varepsilon) \approx u(r,I) \quad (12)$$

and

$$s(\Delta) \approx u(s,I) \quad (13)$$

i.e., the total fluctuation originates practically solely from the uncertainties in the measurements of one party only, the inspector in this example. In such a case, $u(r,O)$ and $u(s,O)$ must be derived from a comparison with inspector's measurements also obtained by DA.

In the IAEA data analysis, various statistical techniques[56] are used to derive separate estimates of the operator's and inspector's uncertainty parameters based on the collection of historical operator-inspector differences. The results of these evaluations are "Performance Values" obtained for each MBA/stratum/measurement method combination. These Performance Values are generally updated once a year as more historical data becomes available for DA and NDA. A similar approach is also applied by other Safeguards organizations^[60-63].

Annex 2 describes how the Performance Values are used in planning inspections^[61-64] and in drawing inferences based on the declared values of the operator and on the measured values of the inspector. There are, however, situations where insufficient historical data is available to derive Performance Values. In these instances ITVs are used until sufficient measurement history is accumulated.

Conversely the most recent and best Performance Values may be used to justify a revision of the ITVs. The relationship between the ITVs and the Performance Values is explained in Section 6 and Figure 2.

4. Results of Laboratory Intercomparisons

Laboratory intercomparisons also offer a documented set of relevant experimental data for defining Target Values. The most useful information stems from experiments,

where the participants analyze very well characterized materials or measure well known volumes or masses of nuclear materials in industrial tanks or containers, and where their results are directly compared to the certified composition of the materials or to the certified value of the respective quantities. Permanent or periodic measurement evaluation programmes have a greater value for our present purpose than one-shot intercomparison experiments, because the participants tend to follow more closely their routine measurement procedure when the intercomparison samples are submitted sufficiently frequently.

The Institute for Reference Materials and Measurements (IRMM, Geel), the Commission d'Etablissement des Méthodes d'Analyse (CETAMA) of the Commissariat à l'Energie Atomique (CEA, France) and the New Brunswick Laboratory at Argonne (NBL, USA) administer such programmes in the area of nuclear material measurements. The discussion of the present edition of the ITVs made extensive reference to the reports published on the results obtained in the Regular European Interlaboratory Measurement Evaluation Programme (REIMEP)^[35-41], the programme of Evaluation de la Qualité des Résultats d'Analyses dans l'Industrie Nucléaire (EQRAIN)^[34,42-44], and the Safeguards Measurement Evaluation Programme (SME)^[45,46,65] run respectively by these three organizations. The calorimetry Exchange Programme of the Mound Laboratory^[47,48] and the Waste Drum Measurement Evaluation Programme of NBL are examples of too rare NDA measurement evaluation programmes. Unfortunately also, there exists still no permanent measurement evaluation programme regarding bulk measurements and the quality of sampling procedures.

Mass measurements are rather straightforward, so that actual inspection data probably provide sufficiently reliable estimates of their uncertainties. The measurement of volumes of solutions in industrial tanks using pneumatic level indicators is a more complex procedure and has been the object of several scientific experiments with international participation. The results of these experiments have been reported^[66-69] and were used in the discussion of the relevant Target Values. The uncertainties to be expected in the use of tracer techniques for volume measurements have been evaluated in the same or similar experiments^[67,70-73].

There are numerous references of interest regarding one-shot intercomparisons of the quality of elemental and isotopic assays by DA^[29-33,74-76], as well as extensive intercomparisons of non destructive measurements by gamma spectrometry^[27,28,31,39].

The evaluations of such one-shot experiments are usually much more elaborate than those of actual inspection data or those of permanent measurement evaluation programmes. They provide, therefore, a better insight into the structure of the sources of measurement uncertainties.

A frequent drawback of interlaboratory comparisons is that they too rarely involve the measurements of actual industrial materials under industrial conditions. The report of the cooperative certification of working reference materials of plutonium and uranium oxides for NDA constitute exceptions^[77-79].

The ITVs 2000 were defined to be consistent with the standard uncertainties observed in the most recent interlaboratory comparisons and measurement evaluation pro-

grammes involving the use of current technologies, with due consideration to the comments made above.

5. Results of Method Validations and Quality Control Measurements

The experimental validation of measurement methods has become a standard practice for metrological and analytical laboratories required by most quality systems. Consolidated guidelines for the standardized performance of such studies are just emerging, however^[52, 80]. This sometimes makes it difficult to compare the respective results obtained by different laboratories. The most trustworthy studies of this type are certainly those which identify the basic metrological parameters of the measurement process, estimate the contributions of the uncertainties occurring in these elementary steps, and compare the expected performance with the results of actual measurements of well-known amounts of materials^[17-26, 81-91]. When the uncertainty propagation model yields larger values than the experimental determinations of the total uncertainty, it is probable that the design of the experiments failed to include one or several sources of potential uncertainties. These cases must receive specific attention. The ESARDA/WGNDA has undertaken a comprehensive examination regarding the propagation of the uncertainties in NDA measurements for safeguards, and in general to the Quality Control and Quality Assurance aspects of NDA measurements. Workshops^[52, 53], discussed QC and QA in the whole process of NDA assay, starting with the instrument design, down to the use in laboratory conditions, in verification measurement, preparation and use of RMs, traceability, qualification and certification of the measurement results.

The reports on the developments of isotope dilution mass spectrometric assay of spent fuel solutions using Large Size Dried (LSD) Spikes^[92], metal spike^[93], internal standard^[94] and total evaporation techniques (TET)^[95] were considered with a particular interest because the analyses of spent fuel dissolver solutions at large reprocessing plants should be of the highest possible accuracy.

The EURACHEM document^[14] contains model cases for the uncertainty propagation for various types of analytical techniques. The IAEA is preparing a technical report on the propagation of uncertainties in radiochemical measurements and nuclear material analyses^[96]. The latter report includes examples dealing with uranium and plutonium assays of spent fuel solutions by isotope dilution mass spectrometry (IDMS), α -spectrometry and X-ray fluorescence analysis (XRFA). These documents describe how the elementary sources of uncertainties in each step of the assay are identified. This leads to the expression of the final result, y , as a function of the elementary parameters of the assay, x_i :

$$y = f(\{x_i\}) \quad (14)$$

The combined uncertainty^[12-14] can be derived according to equation (15) when the $\{x_i\}$ are actually independent variables:

$$u_c^2 = \sum_i c_i^2 \cdot u_i^2 \quad (15)$$

where u_c is the combined standard uncertainty for the assay result y
 u_i the standard uncertainty for parameter x_i

c_i a coefficient of sensitivity defined in equation (16) below.

$$c_i = (\delta y / \delta x_i) \quad (16)$$

Quality control measurements carried out in parallel with the assay of actual samples are a particularly relevant and convenient source of information. These measurements follow as far as possible the same process than the one applied to the samples. The control materials used in these measurements are preferably characterized or certified materials with well documented traceability to the International System (IS) of Measurements. "Type A"^[12] estimates of several standard uncertainties u_i can usually be derived from a variance component analysis of the results of the quality control measurements collected over a sufficiently long period of time. Physical data, certificates of reference materials, weights, physical standards and instruments, such as balances, provide "Type B"^[12] estimates of the other standard uncertainties. This approach is meanwhile applied at several laboratories and has yielded comparative assessments of the respective uncertainties of major nuclear analytical techniques, such as mass spectrometry, IDMS^[22, 97], radiometry (HRGS, GS, α -spectrometry), and the potentiometric titration of uranium and plutonium.

The standard uncertainties obtained from the above studies are grouped in two categories:

- The standard uncertainties of purely random character, $u(r)_i$,
- The standard uncertainties of systematic character, $u(s)_i$.

Equation (15), applied to the random uncertainties, $u(r)_i$, provides an estimate the combined standard uncertainty of the random effects, $u_c(r)$. The combined standard uncertainty of systematic character, $u_c(s)$, is calculated similarly by applying equation (15) to combine the contributions of the standard uncertainties $u(s)_i$.

Such studies establish the necessary quantification of the traceability of nuclear material analyses, and constitute an essential source of information for the selection of the ITVs 2000.

6. Meaning of International Target Values 2000 for Uncertainty Components

The International Target Values 2000 for Measurement Uncertainties (ITVs 2000) are values for uncertainties associated with a single determination result; e.g., this may be the result reported by one laboratory on one sample (independent of the analytical scheme applied internally in the laboratory), or the result of an NDA measurement performed on a single item. The ITVs 2000 take into account actual practical experiences and should be achievable today under the conditions normally encountered in typical industrial laboratories or during safeguards inspections.

The ITVs 2000 were selected on the basis of a critical discussion of the inspectorates' performance evaluations of actual historical data and their comparison with the 1993 ITVs. They are also chosen to be consistent with uncertainty assessments provided by:

- experimental validation of measurement methods and instrumentation,
- interlaboratory measurement evaluation programmes, or
- individual laboratories.

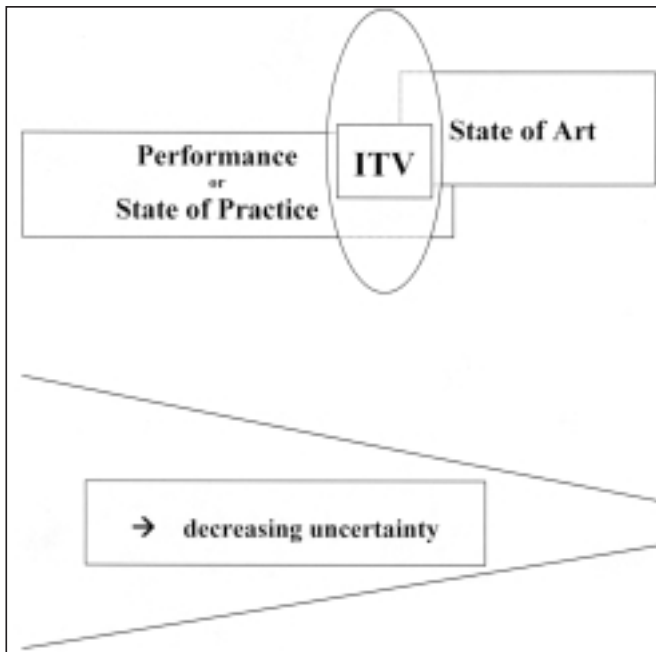


Figure 2: Conceptual Relationship between Performance, State of Art and International Target Values

The ITVs 2000 are applicable to the accountancy data collected by the inspectorates. They do not represent the ultimately achievable performance of a measurement system, which would be obtained under exceptional or ideal laboratory conditions. However, they reflect reasonably well the progress observed during the past several years in the routine performance of measurements done for the purpose of material accountancy and verification.

Figure 2 visualizes the conceptual relationship between Performance Values and ITVs. Performance Values are described by a range of values of the parameters measuring the uncertainties observed during actual industrial operations and safeguards inspections. This range is sometimes said to represent the State-of-the-Practice. The uncertainties achieved under "ideal" conditions by research laboratories or laboratories producing and certifying primary reference materials can be represented by another range of values which may be taken to illustrate the State-of-the-Art in analytical measurements. At a given time, the two ranges of values can overlap to various degrees depending upon the nature of the measurement and the spread of analytical technology advances at that time. The ITV for a given type of measurement is a single value, which has been selected to be a goal of acceptable level, achievable in practice.

The ITVs 2000 intend to take also into account all sources of measurement uncertainties, including sources which may not be apparent in Performance Values resulting from paired comparisons of operator's and inspector's measurements.

7. Structure and Content of the ITVs 2000

The presentation of the 1993 ITVs involved 16 different tables. A different format was chosen for the presentation of the ITVs 2000, which include only 7 tables.

- Table 1 provides a list of the codes used to identify the

measurement methods in Tables 2 to 7. The methods used by the IAEA are described briefly in Reference [98].

- Tables 2 to 6 list the ITVs 2000 for bulk and density measurements, sampling, the determination of element concentration, of ²³⁵U isotope abundance, and of plutonium isotope ratios, respectively.
- ITVs for total amount of fissile element or isotope are given in Table 7 for NDA techniques providing a direct measurement.
- Each table identifies separate ITVs according to the type of material and measurement method, as appropriate.
- Two parameters, **u(r)** and **u(s)**, characterize the quality, which should be aimed for in a specific measurement of a given material using a specified method at a single laboratory; **u(r)** and **u(s)** are specific subsets of the combined standard uncertainty comprising all uncertainties arising from random effects and systematic effects, respectively, according to the description in chapter 3.

These parameters should include all uncertainty components, which determine the potential difference between the measured and the true value. For example, the values specified for the element and isotope concentration measurements include all uncertainties generated in steps 3 to 6 of Figure 1 as well as the uncertainties of the calibration measurements, and the uncertainties of the reference data and materials used for the calibration.

- It has not yet been possible to propose ITVs for the term **u(s)** applicable to sampling, except in a few cases, where this parameter was found to be actually measurable. It should also be noted that random sampling errors were frequently not assessed on the basis of experimental data (due to lack of such) and are based on expert opinion and facility experience.

- The combination of the **u(r)** and **u(s)** parameters

$$u_c(t) = [u(r)^2 + u(s)^2]^{1/2} \quad (17)$$

is equivalent to the relative combined standard uncertainty of the measurement, as it is defined in the ISO^[12], NIST^[13] and EURACHEM^[14] Guides, when it is applied to the measurement of a single laboratory.

- The ITVs in Tables 2 to 7 apply to situations where the measured quantity is large enough so that the relative uncertainty of the measurement remains essentially constant for the given range of measurements.
- The **u(r)** and **u(s)** parameters of bulk measurements, sampling, element concentration and isotope abundance measurements from Tables 2 to 6 must be combined according to equations (18) and (19), in order to obtain the ITVs, **u_c(r)** and **u_c(s)**, applicable to analytical data resulting from a given combination of several measurement steps.

$$u_c(r)^2 = \sum_l u_l(r)^2 \quad (18)$$

$$u_c(s)^2 = \sum_l u_l(s)^2 \quad (19)$$

where *l* refers to an individual step of the analytical process,

and $l = 1, 2, \dots, n$

Examples of such calculations are given in Chapter 8.

Table 1: Measurement Method Codes

Method /Instrument Code	Technique
ANCC	Advanced Neutron Coincidence Counter
AWCC	Active Well Coincidence Counter
CALR	Calorimeter
COMP	Combined Product Uranium Concentration and Enrichment Assay (COMPUCEA)
DIPT	Dip Tube
EBAL	Electronic Balance
FRSC	Fuel Rod Scanner
GRAV	Gravimetry
GSMS	Gas Source Mass Spectrometry
HKED	Hybrid K-Edge/K-XRF Densitometer
HLNC	High Level Neutron Coincidence Counter
HRGS	Infield High Resolution Gamma Spectrometer
IDMS	Isotope Dilution Mass Spectrometry
INVS	Inventory Sample Coincidence Counter
KED	K-Edge Densitometer
LCBS	Load-Cell Based Weighing System
LMCA	Laboratory Multichannel Analyzer/Hi-resolution GS
LMCN	Laboratory Multichannel Analyzer, NaI-detector
PCAS	Plutonium Canister Assay System
PHON	Photon Neutron Interrogation Device
PMCG	Portable Multichannel Analyzer, GeLi-detector
PMCN	Portable Multichannel Analyzer, NaI-detector
PSMC	Plutonium Scrap Multiplicity Counter
TIMS	Thermal Ionization Mass Spectrometry
TITR	Titration
UNCL	Uranium Neutron Coincidence Collar
VTDM	Vibrating Tube Density Meter
WDAS	Waste Drum Assay System

Note: Measurement codes for NDA instruments correspond to the codes adopted in the IAEA Safeguards Manual^[39]

Table 2: Bulk & Density Measurements

Measurement	Instrument	Uncertainty Component (% rel. Std. Uncertainty)	
		u(r)	u(s)
		Mass	LCBS
	EBAL	0,05	0,05
Volume ^{1/}	DIPT	0,30	0,20
Density	DIPT	0,30	0,20
	VTDM	<0.05	<0.05

1.) Volume determinations are made on the basis of level pressure, density and temperature measurements. The volume measurement uncertainties are highly dependent on the homogeneity of the liquid, the quality of the density measurements and of the calibration equation determined in the calibration process. The volume measurements may also involve an absolute error component which has to be taken into consideration when determining the overall uncertainty of volume measurements. For accountability tanks in large-throughput facilities, uncertainties of 0.05% for u(r) and 0.1% for u(s) at full volume are achievable if: i.) A carefully designed calibration procedure has been implemented under well-controlled environmental and stable temperature conditions; and ii.) Measurements are performed on a well-characterized and homogenized liquid.

Table 3: Sampling Uncertainties for Elemental Concentration and ^{235}U Abundance

Material	Uncertainty Component (% rel. Std. Uncertainty)				Recommended Minimum Sample Size ^{5/}
	Concentration		^{235}U Abundance		
	u(r)	u(s) ^{1/}	u(r)	u(s) ^{1/}	
DUF ₆	0,10	nd	1	nd	5-10 g
HEUF ₆ & LEUF ₆ & NUF ₆	0,05	nd	0,10	nd	5-10 g
U-oxide Powder	0,20	nd	nd	nd	10-20 g
U-oxide Pellets	< 0.05 ^{2/}	< 0.05	< 0.05	< 0.05	1 pellet
U Scrap (clean) ^{3/}	1	nd	1	nd	30 g
U Scrap (dirty) ^{4/}	10	nd	10	nd	2 x 30 g
Reprocessing Input Sol.	0,30	0,20	< 0.05	nd	2 x 1 ml
U Nitrate Sol.	0,10	nd	< 0.05	nd	10 ml
Pu, U/Pu Nitrate Sol.	0,20	nd	< 0.05	nd	10 ml
Pu-oxide	0,10	nd			2 x 1 g
FBR & LWR MOX	0.70(Pu) 0.20(U)	nd	0,10	nd	2 x 1 pellet or 2 x 2 g (FBR MOX) or 2 x 5 g (LWR MOX)
MOX Scrap(clean) ^{3/}	1	nd	1	nd	2 x 5 g
MOX Scrap(dirty) ^{4/}	10	nd	10	nd	2 x 10 g
U Metal	0,05	nd	< 0.05	nd	1-5 g
HEU Alloys	0,20	nd	< 0.05	nd	5-10 g

- 1.) Missing values (nd) have not yet been defined.
- 2.) 0.20 for Gadolinium-containing pellets.
- 3.) Scrap with low impurity content and suitable for direct recycling.
- 4.) Sampling errors can vary widely depending on material heterogeneity and sample size.
- 5.) According to STR-69^[100]

Table 4: Element Concentration

Method	Material	Uncertainty Component (% rel. Std.Uncertainty)				Notes ^{1/}
		U-Conc.		Pu-Conc.		
		u(r)	u(s)	u(r)	u(s)	
GRAV	U Oxides(pure),UF ₆	0.05	0.05			2/
	Pu Oxide			0.05	0.05	2/
TITR	U Oxides,UNH,UF ₆	0.1	0.1			
	U Alloys	0.2	0.2			
	Pu Oxide, Pu Nit.			0.15	0.15	3/
	MOX, U/Pu Nit.	0.1	0.1	0.2	0.2	3/
IDMS	U & Pu Compounds					4/5/
	Hot Cell Conditions Glove Box Conditions	0.2 0.15	0.2 0.1	0.2 0.15	0.2 0.1	
KED	U in solution	0.2	0.15			6/
	Pu in solution			0.2	0.15	6/7/
	FBR MOX			0.3	0.2	6/
HKED	Spent Fuel Solution, LWR MOX	0.2	0.15	0.6	0.3	8/
COMP	U Compounds	0.2	0.15			2/6/9/
ANCC	Pu Oxide, MOX			0.2	0.2	10/
INVS	Pu Oxide, MOX			2	1.5	11/12/
	MOX Scrap			10	2.5	11/

- 1.) Concentration measurements on powders and solutions require weight change correction because of sample instability.
- 2.) Material containing non-volatile impurities < 1000 ppm
- 3.) Equivalent performance may be expected when applying coulometry
- 4.) Materials typically encountered in the nuclear fuel cycle
- 5.) Under conditions of sufficiently different isotopic compositions of spike and sample and near-optimum sample:spike ratio[83,96,97]
- 6.) Measurement time 1000 sec., adjusted for age of source when necessary
- 7.) For samples in solution with >50 g/l Pu
- 8.) 150 g/l U, 1.5 g/l Pu
- 9.) 200 g/l U
- 10.) For: 2g sample; 4 hour counting time; isotopic determination by mass spectrometry; detector efficiency > 40%
- 11.) Measurement time 300 sec.
- 12.) Isotopic determination by mass spectrometry

Table 5: ^{235}U Abundance

Method	Material	Uncertainty Component (% rel. Std. Uncertainty)		Notes
		u(r)	u(s)	
GSMS	DUF ₆ & NUF ₆	0,1	0,1	
	LEUF ₆	0,05	0,05	
	HEUF ₆	0,02	0,02	
TIMS	DU (< 0.3 wt.% ^{235}U)	0,5	0,5	
	U (0.3% < ^{235}U < 1%)	0,2	0,2	
	LEU (1% < ^{235}U < 20%)	0,1	0,1	
	HEU (> 20 wt.% ^{235}U)	0,05	0,05	
COMP	LEU Compounds	0,4	0,2	1/
LMCN ^{2/}	LEU Oxides	0,3	0,3	
	HEU Oxides	0,2	0,2	
PMCN ^{2/3/}	DUF ₆	20	15	4/
	NUF ₆	10	8	4/
	LEUF ₆	5	3	4/
	NU Oxides	5	5	
	LEU Oxides	3	2	
	NU & LEU Scrap (clean) ^{5/}	5	5	6/
	NU & LEU Scrap (dirty)	15	10	6/
	LEU Fuel Rods	2,5	1	
	LEU Fuel Assemblies	2,5	1	
	HEU Metal	0,5	0,5	7/
PMCG ^{3/}	HEU Alloys	1	1	7/
	DUF ₆	15	10	4/
	NUF ₆	8	5	4/
	LEUF ₆	4	2	4/
	LEU Oxides	3	2	
	HEU Metal	0,5	0,5	7/
	HEU Alloys	1	1	7/

1.) Measurement time 1000 sec., adjusted for age of source when necessary; see Ref. [21]

2.) For materials not containing reprocessed uranium.

3.) Measurement time 300 sec.

4.) Includes uncertainty component associated with ultrasonic thickness gauge measurement of the UF6 cylinder.

5.) Scrap with low impurity content and suitable for direct recycling.

6.) Uncertainties for scrap represent average performance observed on historical data. Material matrix heterogeneity is the main contributor to the observed uncertainties and can vary widely.

7.) Calibration against reference material certified to 0.3 % or better & uncertainties in the correction of container wall absorption of 0.5 % or less.

**Table 6: Plutonium Isotope Assay
of Pu Oxide and MOX**
(% Relative Standard Uncertainties)

Material Type	Isotope Ratio	Typical Value for Ratio (*100)	Method					
			TIMS ^{1/}		HRGS ^{2/}		LMCA ^{3/}	
			u(r)	u(s)	u(r)	u(s)	u(r)	u(s)
High-Burnup Pu	²³⁸ Pu/ ²³⁹ Pu	1,7	1,5	1	2	2	1	1
	²⁴⁰ Pu/ ²³⁹ Pu	43	0,1	0,05	1	1	0,7	0,7
	²⁴¹ Pu/ ²³⁹ Pu	13	0,2	0,2	1	1	0,7	0,7
	²⁴² Pu/ ²³⁹ Pu	8	0,2	0,3				
Low-Burnup Pu	²³⁸ Pu/ ²³⁹ Pu	0,02	10	10	10	10	5	5
	²⁴⁰ Pu/ ²³⁹ Pu	6	0,15	0,1	2	2	1,5	1,5
	²⁴¹ Pu/ ²³⁹ Pu	0,2	1	1	2	2	1	1
	²⁴² Pu/ ²³⁹ Pu	0,05	2	2				

- 1.) ²³⁸Pu/²³⁹Pu by alpha spec./TIMS combination
- 2.) Measurement time 3 x 100 sec.
- 3.) Measurement time 3 x 1000 sec.; 0.5 g Pu.

Table 7: Total Mass - ^{235}U & Pu
by Direct NDA Measurement Techniques

Instrument	Material	Uncertainty Component (% rel. Std.Dev.)				Notes
		^{235}U Mass		Pu Mass		
		u(r)	u(s)	u(r)	u(s)	
AWCC	HEU Metal, HEU Alloys	5	3			1/
	HEU Fuel Elements	3	2			1/
FRSC	U Fuel Rods	1	1			
PHON	LEU Oxides	2	1			
	LEU Scrap	4	1			
UNCL	U Fuel Assemblies	4	2			
HLNC	Pu Oxide Powder			1	0.5	2/3/
	FBR MOX (> 10% Pu)			2	0.5	2/3/
	LWR MOX (< 10 % Pu)			4	1.5	2/3/
	MOX Scrap			10	3	2/4/
	Pu Fuel Rods			1.5	1	2/3/
	MOX Fuel Rods			2	1	2/3/
	MOX Fuel Assemblies			1.5	1	2/3/
PCAS	FBR MOX			1.5	1	3/
	MOX Scrap			8	2	4/
PSMC	MOX Scrap (clean)			2.5	1	3/5/
	MOX Scrap (dirty)			8	2	4/
WDAS	MOX Waste			8	2	4/
CALR	Pu Oxide and MOX			0.4	0.4	3/6/7/

- 1.) Measurement time 600 sec.
- 2.) Measurement time 300 sec.
- 3.) Isotopic determination by mass spectrometry and alpha spectrometry.
- 4.) Uncertainties for scrap represent average performance observed on historical data. Material matrix heterogeneity is the main contributor to the observed uncertainties and can vary widely.
- 5.) Scrap with low impurity content and suitable for direct recycling
- 6.) ^{241}Am content determined by gamma spectrometry or alpha spectrometry
- 7.) Lower uncertainties are achievable for materials containing low burn up Pu

8. Use of ITVs

ITVs are considered to be achievable in routine measurements involved in the determination of the amount of nuclear materials for materials accountancy and safeguards verification purposes. They are intended to be used as a reference by plant operators, state systems and international safeguards organizations. They should, however, not be normally used in place of values based on actual measurements in estimating the statistical significance of operator-inspector differences or MUF. Analytical laboratories can find it useful to determine experimentally the actual uncertainties of their measurements, and to compare them with the corresponding values, which can be derived from the ITVs 2000.

Safeguards authorities regularly compare the performance values with the current ITVs. They will examine with the relevant authorities and laboratories means of improving the performance, in cases where the performance values are significantly higher than the ITVs, and too high to allow the IAEA to meet its detection goals[101]. When reliable performance values are not available, ITVs may be used instead to calculate sampling plans, to set reject limits and to calculate estimates of the combined uncertainties of inventories, throughputs, MUF and D's, as described in Annex 2.

Such applications of the ITVs require having a good insight of the measurement and verification systems. It is in particular important to recognize that, because of practical constraints, some measurement steps may be common to the operator and the inspector. It should also not be forgotten that the operator-inspector differences can carry errors which are not related to measurement uncertainties.

The following three examples illustrate how the tabulated ITVs can be used to calculate ITVs for combined uncertainties applicable to practical situations. Further examples are presented in Reference [102].

Example 1:

Target Values for the Determination of the Total Mass of Fissile Element on Independent Samples

Consider a situation where the operator and the inspector determine fully independently the total amount of plutonium in a batch of LWR pellets. The operator measures the plutonium concentration by titration on ten randomly

selected pellets, the inspector by IDMS on an independently selected but single pellet.

The Target Values for the combined relative standard uncertainties applicable to the determination of the total mass of plutonium by the operator are derived from the following equations, respectively for the random errors, the errors of systematic character and their combination:

$$u_c(r, O) = \sqrt{\sum_i u_i^2(r, O)/n_i(O)} \quad (20)$$

$$u_c(s, O) = \sqrt{\sum_i u_i^2(s, O)} \quad (21)$$

$$u_c(O) = \sqrt{[u_c^2(r, O) + u_c^2(s, O)]} = \sqrt{(0.0555 + 0.0425)} = 0.31\% \quad (22)$$

The above values would be used in the calculation of Target Values for the relative standard uncertainties to be expected in the inventory, throughput and MUF declared by the operator.

Similar equations are used to calculate the corresponding values applicable to inspector's measurements, $u_c(r, I)$, $u_c(s, I)$ and $u_c(I)$. The Target Value for the combined uncertainties on the total Pu mass measured by the inspector is equal to:

$$u_c(I) = \sqrt{[u_c^2(r, I) + u_c^2(s, I)]} = \sqrt{(0.5150 + 0.0125)} = 0.73\% \quad (23)$$

Its magnitude is determined essentially by the random sampling uncertainty component. This is also true for the Target Value applicable to the Operator-Inspector difference:

$$u_d = \sqrt{[u_d^2(r) + u_d^2(s)]} = \sqrt{(0.5705 + 0.0550)} = 0.79\% \quad (24)$$

Assuming that the values of Target Values, u_c 's, given in Table 8 and equations (22), (23) and (24), are effectively achieved, the 95% confidence intervals of the final results of the operator, of the inspector and of their difference, would be respectively equal to:

$$CL(O) = k u_c(O) = 2 \times 0.31 = 0.62\% \quad (25)$$

$$CL(I) = k u_c(I) = 2 \times 0.73 = 1.46\% \quad (26)$$

$$CL(d) = k u_c(d) = 2 \times 0.79 = 1.58\% \quad (27),$$

where the coverage factor k is 2.

Table 8: Target Values for Total Pu Mass with Independent Samples and DA (Example 1)

	Step	Method Instr.	n_i	ITV (% rel. Std. Dev.)			Variance Component		
				$u_i(r)$	$u_i(s)$	Table	$u_i^2(r)/n_i$	$u_i^2(s)$	
O P E R A T O R	1- Bulk	EBAL	1	0,05	0,05	2	0,0025	0,0025	
	2- Sampling Pu-Conc.		10	0,70	nd	3	0,0490		
	6- Pu-Conc.	TITR	10	0,20	0,20	4	0,0040	0,0400	
	Sum of variance components							0,0555	0,0425
	Combined Std. Uncertainties, $u_c(r,O)$ and $u_c(s,O)$, (in % rel.)							0,24	0,21
I N S P E C T O R	1- Bulk	EBAL	1	0,05	0,05	2	0,0025	0,0025	
	2- Sampling Pu-Conc.		1	0,70	nd	3	0,4900		
	6- Pu-Conc.	IDMS	1	0,15	0,10	4	0,0225	0,0100	
	Sum of variance components							0,5150	0,0125
	Combined Std. Uncertainties, $u_c(r,I)$ and $u_c(s,I)$, (in % rel.)							0,72	0,11
D I F F	Variance of Rel. Operator-Inspector Difference							0,5705	0,0550
	Standard Uncertainties of Rel. Diff., $u_d(r)$ and $u_d(s)$, (in %)							0,76	0,23

**Example 2:
Target Values for the Determination of the
Total Mass of Fissile Element on a Common
Sample.**

In situations where the inspector analyzes a subsample of a homogeneous operator's sample, the sampling errors no longer contribute to the uncertainty of the Operator-Inspector difference. An example of this situation could be

a co-operative effort to identify the existence of biases in the chemical analysis.

Apply these conditions to the first example. In this case, as shown in Table 9, the Target Value for the Operator-Inspector difference and its 95% confidence interval will be:

$$u_d = \sqrt{(0.0675 + 0.0550)} = 0.35 = \% \tag{28}$$

$$CL(d) = k u_d = 2 \times 0.35 = 0.70\% \tag{29}$$

Table 9: Target Values for Operator-Inspector Difference on Total Pu Mass, with Common Sample and DA (Example 2)

	Step	Method Instr.	n _i	ITV (% rel. Std. Dev.)			Variance Component	
				u _i (r)	u _i (s)	Table	u _i ² (r)/n _i	u _i ² (s)
O P E R A T O R	1- Bulk	EBAL	1	0,05	0,05	2	0,0025	0,0025
	2- Sampling Pu-Conc.		1	0,70	nd	3		
	6- Pu-Conc.	TITR	1	0,20	0,20	4	0,0400	0,0400
	Sum of variance components contributing to Op-In Differ.							0,0425
I N S P E C T O R	1- Bulk	EBAL	1	0,05	0,05	2	0,0025	0,0025
	2- Sampling Pu-Conc.		1	0,70	nd	3		
	6- Pu-Conc.	IDMS	1	0,15	0,10	4	0,0225	0,0100
	Sum of variance components contributing to Op-In Differ.							0,0250
D I F F	Variance components of relative difference						0,0675	0,0550
	Standard Uncertainties of Rel. Diff., u _d (r) and u _d (s), (in %)						0,26	0,23

Example 3:
Estimation of the Uncertainty of
Operator-Inspector Differences for NDA
Sampling Plan Calculations

Consider a situation where an inspector must calculate a sample size for verifying the content of LEUF₆ containers using a PMCN. The operator declarations for the material are based on DA measurements of ²³⁵U abundance and the stoichiometric value for U-concentration in UF₆. No historical inspector measurement data is available. Therefore ITVs need to be used to provide an estimate of the uncertainty which may be associated with the operator-inspector difference.

The variance components calculated from the ITVs 2000 are given in Table 10. The standard combined uncertainty associated with the operator-inspector difference in this example is equal to:

$$u_d = \sqrt{u_d^2(r) + u_d^2(s)} = \sqrt{(25.0175 + 9.0075)} = 5.83 \% \quad (30)$$

In the absence of an uncertainty estimate based on historical measurement data, the inspector would thus use the above value calculated from the ITVs for performing sample size calculations and establishing rejection limits. In this example, the relatively large uncertainty associated with the NDA measurement almost entirely determines the overall uncertainty of the operator-inspector difference.

Table 10: Target Values for Operator-Inspector difference (Example 3)

	Step	Method/Instr.	ITV (% rel. Std. Dev.)			Variance Component	
			u _d (r)	u _d (s)	Table	u _d ² (r)	u _d ² (s)
O P E R A T O R	1- Bulk	EBAL	0,05	0,05	2	0,0025	0,0025
	2- Sampling ²³⁵ U wt. %		0,1		3	0,0100	
	6- U-Conc.	Stoichiom. Val.					
	6- ²³⁵ U wt. %	GSMS	0,05	0,05	5	0,0025	0,0025
	Sum of variance components						0,0150
I N S P E C T O R	1- Bulk	EBAL	0,05	0,05	2	0,0025	0,0025
	6- U-Conc.	Stoichiom. Val.					
	6- ²³⁵ U wt. %	PMCN	5	3	5	25,0000	9,0000
	Sum of variance components						25,0025
D I F F	Variance components of relative difference					25,0175	9,0075
	Standard Uncertainties of Rel. Diff., u _d (r) and u _d (s), (in %)					5,00	3,00

9. Future Developments

It is intended to keep updating the ITV tables regularly in order to incorporate the latest relevant information. The following activities will be especially important for this purpose:

- Growing emphasis is being placed on reassessing the uncertainties of chemical measurements according to the ISO^[12], NIST^[13] and EURACHEM^[14] guides. This should be done systematically for the methods in current use. It should become a part of the process of qualification of new measurement methods and instrumentation.
- The inspectorates will continue to update actual performance evaluations.
- It is important that interlaboratory measurement evaluation programmes continue to be conducted, particularly in the area of Pu measurements. Operator and inspector laboratories should participate in such programmes. Their results should be published as it was done in the past.
- Models more specific to the NDA measurement processes are being reviewed by the ESARDA/NDA Working Group to monitor and assess the sources of major uncertainties in actual inspectors' measurements. This will hopefully involve uncertainty assessments in line with the above guides as well as periodical estimates of actual Performance Values and the development of interlaboratory measurement evaluation programmes for NDA.
- Results of experimental qualifications of recommended sampling procedures^[103-112] should be made available to the inspectorates to substantiate and expand ITVs for the uncertainty components in sampling procedures.
- The IAEA will also follow with the greatest interest developments in bulk measurements and elemental assays of spent fuel solutions and their impact on the accuracy of the accountability of large throughputs and inventories of nuclear materials at large plants now coming under safeguards.

The IAEA will continue its cooperation on the above topics with Euratom, with State authorities and with the expert groups, which were involved in the review of the ITVs 2000. The next revision of the ITVs will also be another opportunity to seek further contributions from more countries and organizations.

Annex 1 Statistical Terminology

The statistical terminology used in this document is given here in an effort to promote better understanding of the statistical concepts discussed herein. The terminology is divided into four groups as follows:

Group 1: Basic terms

(true value, conventional true value, measurand, measurement, measurement result,

measurement error, uncertainty, uncertainty component, expectation, expected value, mean, variance, standard deviation, sample standard deviation, experimental standard deviation, error parameter, sample, estimation, statistic, estimator, estimate).

Group 2: Selected sources and classes of error

(bulk measurement error, sampling error, random error of result, random error of measurement, systematic error of result, systematic error of measurement, calibration error, bias, relative error).

Group 3: Descriptors

(precision, accuracy, repeatability, repeatability conditions, reproducibility, reproducibility conditions).

Group 4: Safeguards specific use of terms

(random sampling, inspection by attribute, alarm level, significant difference, defect, discrepancy, detection probability, false alarm, risk).

The definitions of these terms, as used in this document, are given to the extent feasible, in a way which is consistent with the latest internationally recognized standards or manuals. The relevant source^[14,54-56,114] of the definition is specified in the following tables.

Annex 2

Use of Performance Values for Inspection Purposes and Their Limitations

The Performance Values (see chapter 3) are used in planning inspections and in drawing inferences based on the declared values of the operator and on the measured values of the inspector^[56].

From an inspection planning viewpoint, they allow calculation of sample sizes for NDA and for DA verification methods that are optimal with respect to achieving the desired level of defect detection probability with the minimum number of samples.

When evaluating the verification data, they serve first to define item-level alarms, or reject limits, such that if a given item paired difference, d_{ij} , exceeds the limit L in absolute value, it is identified as a discrepancy, where L is defined by the equation:

$$L = z_{\alpha} \left[u^2(r) + u^2(s) \right]^{1/2} \quad (\text{A2.1})$$

where z_{α} is the normal probability distribution factor associated with the probability α of declaring a false alarm. Current practice is to take $z_{\alpha} = 3$, which results in a false alarm probability of less than 0.3 %.

The item paired differences are calculated on either an absolute or relative basis, as was mentioned in chapter 3. Of course, for a homogeneous stratum, it makes no difference whether absolute or relative differences are calculated.

In addition to defining attribute test reject limits as just described, performance values are also used in calculating the variances used in material balance evaluations for material unaccounted for (MUF), operator-inspector difference (D), and the inspector's estimate of MUF, (MUF-D).

In a large facility the probability of detection will be driven by the amount of material. Regardless of how accurately and precisely material is measured, σ will be large because the amount of material is large. In such cases, the probability of detecting diversion by means of a material balance evaluation will be small and additional safeguards measures such as near real-time accountancy (NRTA) are called for.

The users of the Performance Values must remain aware of a number of limitations in their meaning or content.

Plant operational or economic constraints may inflate the variance components of the operator-inspector differences significantly compared to the capability of current measurement technology. The safeguards inspector must indeed verify that the uncertainties in the plant measurement system are not deliberately inflated in order to reduce the detection capability of the verification measurements. The latter concern increases with the throughput or material inventory of the plant. There will therefore always be a need for Target Values providing an accepted measure of the capability of current measurement technology under reasonably economic and operational conditions encountered in the industry.

Conversely, paired comparisons do not detect the measurement errors or uncertainties, which are common

to the operator and inspector. For example, if both use the same reference material for calibration, the uncertainty of the certified value of the reference material will appear as a common systematic component in both results. The common component can also be of a random nature; random sampling errors are common, for instance, when the operator and the inspector measure the same sample or separate aliquots of the same sample.

These common components do not affect the uncertainties of the differences between operator's and inspector's measurements on a single stratum. They can, however, mask a potential bias with respect to the true amount of material. Consequently the use of Performance Values can lead to underestimation of the total uncertainties in the operator's declarations or in the material balance differences over the plant. Independent measurement evidence, free from such common mode uncertainties, is hence needed.

The user of the Performance Values must also know that the estimate of the between inspection effects, $s(\Delta)$, becomes less precise as the random uncertainty component, $s(\epsilon)$, increases. When the inspector's uncertainties are large compared to the operator's values, it becomes difficult to obtain a precise estimate of the operator's uncertainties, and vice-versa. This is frequently the case when the operator's data come from DA measurements while the inspector measures by NDA. The paired comparisons can lead to an overestimation of the random uncertainties of the operator's DA measurements, and, at the same time, to a poor estimate of the between-inspection effects in the inspector's NDA results. As a further complication, estimates of these parameters will be affected when the operator's values are based in part on nominal or average values. A separate evaluation of the performance of individual measurement methods is necessary to guard against such potential problems.

GROUP 1: BASIC TERMS		
Term	Definition	Ref.
true value	Value consistent with the definition of a given particular quantity. NOTES: 1. This is a value that would be obtained by a perfect measurement. 2. True values are by nature indeterminate.	[55] 1.19
conventional true value	Value attributed to a particular quantity and accepted, sometimes by convention, as having an uncertainty appropriate for a given purpose. NOTE: "Conventional true value" is sometimes called <i>assigned value</i> , <i>best estimate</i> of the value, <i>conventional value</i> , or <i>reference value</i> .	[55] 1.20
measurand	Particular quantity subject to measurement.	[55] 2.6
measurement	Set of operations having the object of determining a value of a quantity.	[55] 2.1
measurement result	Value attributed to a measurand, obtained by measurement NOTES: 1. When the term "result of a measurement" is used, it should be made clear whether it refers to: - the value indicated by the measurement instrument - the uncorrected result - the corrected result and whether several values are averaged. 2. A complete statement of the result of a measurement includes information about the uncertainty of measurement.	[55] 3.1
measurement error	Result of a measurement minus a true value of the measurand. NOTES: 1. Since a true value cannot be determined, in practice a conventional true value is used. 2. The quantity is sometimes called <i>absolute error</i> of measurement when it is necessary to distinguish it from <i>relative error</i> .	[55] 3.10
uncertainty (of measurement)	Parameter associated with the result of a measurement, characterizing the dispersion of the values that could reasonably be attributed to the measurand. NOTE: The parameter may be, for example, a standard deviation (in which case the uncertainty is also called the <i>standard uncertainty</i>), or the width of a confidence interval.	[55] 3.9
uncertainty component	Uncertainty arising from a distinct source contributing to the overall uncertainty. NOTES: 1. If there is correlation between any components then this has to be taken into account by determining the covariance. 2. It is often possible to evaluate the combined effect of several components. 3. Where components whose contribution is evaluated together are correlated, there may be no additional need to take account of the correlation	[14] 2.3.1
expectation, expected value, mean	If X is a continuous random variable having the probability density function $f(x)$ then the expectation (or expected value or mean), if it exists, is $\mu_x = E(X) = \int x \cdot f(x) dx$ (the integral being extended over the intervals of variation of X).	[54] 1.18
variance	$\sigma^2 = V(X) = E [X - E(X)]^2$ The variance is the expectation of the square of the <i>centred random variable</i> (i.e. a random variable the expectation of which equals zero).	[54] 1.22

GROUP 1: BASIC TERMS		
Term	Definition	Ref.
standard deviation	$\sigma = \sqrt{V(X)}$	[54] 1.23
sample standard deviation, experimental standard deviation	<p>For a series of n measurements of the same measurand, the quantity s characterizing the dispersion of the results and given by the formula:</p> $s = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1}}$ <p>x_i being the result of the i^{th} measurement and \bar{x} being the arithmetic mean of the n results considered.</p> <p>NOTES:</p> <ol style="list-style-type: none"> 1. Considering the series of n measurements as a sample of a population, \bar{x} is an unbiased estimate of the mean μ, and s^2 is an unbiased estimate of the variance σ^2 of that distribution. 2. The expression s/\sqrt{n} provides an estimate of the standard deviation of the distribution of \bar{x} and is called the <i>experimental standard deviation of the mean</i> (it is sometimes, but incorrectly, called <i>standard error</i> or <i>standard error of the mean</i>). 	[55] 3.8
error parameter	<p>Synonymous for error variance.</p> <p>In general, the result of a particular measurement, x_i, may be modelled by</p> $x_i = \mu + \varepsilon_{1i} + \varepsilon_{2i} + \varepsilon_{3i} + \dots,$ <p>where μ is the true value of the measured quantity and the $\varepsilon_{.i}$ are individual errors made during the particular measurement i according to the various potential sources of errors 1, 2, 3, ...</p> <p>If, for example, the error ε_{1i} is a representation of a centred random variable ε_1 (mean zero): $E(\varepsilon_1) = 0$, then the variance $E(\varepsilon_1^2) = V(\varepsilon_1) = \sigma_{\varepsilon_1}^2$ is called an error parameter.</p> <p>If the individual sources of error (or fluctuation) are independent from each other, then the overall variance of the measurement results can be calculated by</p> $V(X) = \sigma_X^2 = \sigma_{\varepsilon_1}^2 + \sigma_{\varepsilon_2}^2 + \sigma_{\varepsilon_3}^2 + \dots = \sum (\sigma_{\varepsilon_{.}}^2)$	
sample	<ol style="list-style-type: none"> 1. One or more of the individual items into which a population is divided, taken with the intention to provide information on that population. 2. A portion of material taken from a larger batch of material with the intention to be representative for that larger batch with respect to the characteristics under consideration. 	various
estimation	The operation of assigning, from the observations in a sample, numerical values to the parameters of a distribution chosen as the statistical model of the population from which this sample is taken.	[54] 2.49
statistic	<p>A function of the sample random variables.</p> <p>NOTE:</p> <p>A statistic, as a function of random variables, is also a random variable and as such it assumes different values from sample to sample. The value of the statistic obtained by using the observed values in this function may be used in a statistical test or as an estimate of a population parameter, such as a mean or a standard deviation.</p>	[54] 2.45
estimator	A statistic used to estimate a population parameter.	[54] 2.50
estimate	The value of the estimator obtained as a result of an estimation.	[54] 2.51

GROUP 2: SELECTED SOURCES and CLASSES of ERRORS		
Term	Definition	Ref.
bulk measurement error	The measured mass (volume) of an item minus its true mass (volume).	[55] 3.10
sampling error	The true value for the portion of material constituting the sample minus the true value for the larger batch of material for which the sample is intended to be representative.	
random error (of result)	A component of the error, which, among a number of test results for the same characteristic, varies in an unpredictable way. NOTE: The random error of an analytical result cannot be compensated for, but it can be usually reduced by increasing the number of observations.	[54] 3.9
random error (of measurement)	Result of a measurement minus the mean that would result from an infinite number of measurements of the same measurand carried out under repeatability conditions. NOTE: 1. Random measurement error is equal to measurement error minus systematic measurement error. 2. Because only a finite number of measurements can be made, it is possible to determine only an estimate of random error.	[55] 3.13
systematic error (of result)	A component of the error, which, among a number of test results for the same characteristic, remains constant or varies in a predictable way. NOTES: 1. Systematic errors and their causes may be known or unknown. 2. Under constant measurement conditions, the systematic error is independent of the number of measurements made and therefore cannot be reduced by increasing the number of analyses.	[54] 3.10
systematic error (of measurement)	Mean that would result from an infinite number of measurements of the same measurand carried out under repeatability conditions minus a true value of the measurand. NOTES: 1. It is important to observe the restriction "under repeatability conditions". The value of the systematic measurement error may remain constant as long as the measurement conditions remain unaltered. However it may vary, in an unpredictable manner, with the changing of the measurement conditions or the settings of the measurement system. The systematic measurement error is therefore systematic (or constant) only with respect to a given set of measurement results, while it is at the same time a random component of the error on a long term perspective. Hence the systematic measurement error possesses a probability distribution (with expectation zero) and can be represented by a random statistical variable over a sufficiently long period of time. 2. The systematic error components under consideration in the present document are all of this dual nature.	[55] 3.14
calibration error	An error associated with a given calibration. Hence, a systematic measurement error component with respect to all measurements performed with the same calibration.	[114] 7.9
bias	The difference between the expectation of the test result and an accepted reference value (conventional true value). NOTE: 1. Bias can also be described as the total of all long term systematic error components; i.e., those components of the error that do not vary even under reproducibility conditions. 2. Like the <i>true value</i> , bias is by nature indeterminate. 3. If the bias is estimated and corrected for, the uncertainty of the correction must still be taken into account.	[54] 3.13
relative error	The absolute error of the measurement divided by the true value of the measurand. Frequently expressed as a percentage value (i.e. multiplied by hundred).	

GROUP 3: DESCRIPTORS		
Term	Definition	Ref.
precision	The closeness of agreement between independent test results obtained under stipulated conditions. NOTE: 1. Quantitative measures of precision depend critically on the "stipulated conditions". Repeatability conditions and reproducibility conditions are particular sets of extreme stipulated conditions. 2. The standard deviation of the test results is one usual quantitative measure of precision. A larger standard deviation represents less precision. 3. Precision depends only on the distribution of random errors and therefore does not relate to deviations from the true value.	[54] 3.14
accuracy (of measurement)	The closeness of agreement between the result of a measurement and a true value of the measurand. NOTE: 1. "Accuracy" is a qualitative concept. 2. When applied to a set of test results, accuracy involves a combination of random error components and a systematic error component.	[54] 3.11 [55] 3.5
repeatability	Precision under repeatability conditions.	[54] 3.15
repeatability conditions	Conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time. NOTE: 1. Repeatability conditions constitute one specific and extreme set of conditions. 2. Repeatability relates practically to smallest fluctuation that is reasonably achievable among independent test results.	[54] 3.16
reproducibility	Precision under reproducibility conditions	[54] 3.20
reproducibility conditions	Conditions where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment. NOTE: 1. Reproducibility conditions constitute another specific and extreme set of conditions. 2. Reproducibility relates practically to the largest fluctuation that is reasonably encountered among independent test results. 3. Operator - inspector differences are typically generated under reproducibility conditions.	[54] 3.21

GROUP 4: SAFEGUARDS SPECIFIC USE OF TERMS		
Term	Definition	Ref.
random sampling	<p>Random sampling is a method for taking a sample from a group of distinct items. A random sample is selected by a procedure that gives each item a fixed and determinate (usually equal) probability of selection.</p> <p>NOTE: When applied to non-distinct things, such as samples of bulk material, the method of selection does not usually indicate probabilities of selection of the samples. In principle, when drawing samples from a larger bulk of material, its content could conceivably be divided into a large number of possible samples of a certain size; some procedure would be used to give each potential sample an equal chance of being selected. The actual sampling method is designed to achieve the same result in a practical way, so that the statistical theory based on the concept of random sampling can be applied.</p>	[56] 2.8
inspection by attributes	<p>In attributes inspection, the item inspected is classified as being either acceptable or not (i.e. a defect) on the basis of the measurement.</p> <p>NOTE: Attributes inspection has nothing to do with the quality of measurement.</p>	[56] 6.2
alarm level	<p>A synonym for a critical value in the terminology of testing an hypothesis.</p> <p>NOTE: In the subject context, the alarm level is the value of an operator-inspector difference which, if exceeded in absolute value, is cause for labelling the item in question a defect during inspection by attributes.</p>	
significant difference, defect, or discrepancy	<p>Three synonyms for an operator-inspector difference that exceeds the alarm level in absolute value.</p>	[114] 8.21
detection probability	<p>The probability that an item with a true operator-inspector difference of a given amount will be declared to be a discrepancy. More generally, when referring to a safeguards index such as MUF or D, it is the probability that the index will be found to differ significantly from its hypothesised value for a given true value of the index.</p>	[114] 5.17
false alarm	<p>In attribute testing, declaring an item to be a defect when the true operator-inspector difference is zero is a false alarm. A similar definition applies to tests on MUF and $\hat{D} = 0$.</p>	[114] 5.18
risk	<p>A synonym for the probability of reaching the incorrect conclusion in hypothesis testing. Two types of risks are usually considered:</p> <ol style="list-style-type: none"> 1. the risk of false alarm, defined above, 2. the risk of non-detection, associated with the failure to detect a "true" defect. 	

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