BS EN 61788-11:2011

BSI Standards Publication

Superconductivity

Part 11: Residual resistance ratio measurement — Residual resistance ratio of Nb_3Sn composite superconductors

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National foreword

This British Standard is the UK implementation of EN 61788-11:2011. It is identical to IEC 61788-11:2011. It supersedes BS EN 61788-11:2003 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee L/-/90, Super Conductivity.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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(IEC 61788-11:2011)

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European Committee for Electrotechnical Standardization
Comité Européen de Normalisation Electrotechnique
Europäisches Komitee für Elektrotechnische Normung

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Foreword

The text of document 90/268/FDIS, future edition 2 of IEC 61788-11, prepared by IEC TC 90, Superconductivity was submitted to the IEC-CENELEC parallel vote and approved by CENELEC as EN 61788-11:2011.

The following dates are fixed:

• latest date by which the document has to be implemented at national level by publication of an identical national standard or by endorsement	(dop)	2012-05-15
• latest date by which the national standards conflicting with the document have to be withdrawn	(dow)	2014-08-15

This document supersedes EN 61788-11:2003.

The main revisions are the addition of two new annexes "Uncertainty considerations" (Annex B) and "Uncertainty evaluation in test method of RRR for Nb_3Sn" (Annex C).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CENELEC [and/or CEN] shall not be held responsible for identifying any or all such patent rights.

Endorsement notice

The text of the International Standard IEC 61788-11:2011 was approved by CENELEC as a European Standard without any modification.

Annex ZA
(normative)

Normative references to international publications with their corresponding European publications

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

NOTE When an international publication has been modified by common modifications, indicated by (mod), the relevant EN/HD applies.

Publication	Year	Title	EN/HD	Year
IEC 60050-815	-	International Electrotechnical Vocabulary (IEV) - Part 815: Superconductivity	-	-

CONTENTS

INTRODUCTION

Copper or aluminium is used as stabilizer material in multifilamentary Nb_3Sn superconductors and works as an electrical shunt when the superconductivity is interrupted. It also contributes to recovery of the superconductivity by conducting the heat generated in the superconductor to the surrounding coolant. The resistivity of copper used in the composite superconductor in the cryogenic temperature region is an important quantity which influences the stability of the superconductor. The residual resistance ratio is defined as a ratio of the resistance of the superconductor at room temperature to that just above the superconducting transition.

In this International Standard, the test method for the residual resistance ratio of Nb_3Sn composite superconductors is described. The curve method is employed for the measurement of the resistance just above the superconducting transition. Other methods are described in Clause A.3.

SUPERCONDUCTIVITY –

Part 11: Residual resistance ratio measurement – Residual resistance ratio of Nb_3Sn composite superconductors

1 Scope

This part of IEC 61788 covers a test method for the determination of the residual resistance ratio (*RRR*) of Nb_3Sn composite superconductors. This method is intended for use with superconductor specimens that have a monolithic structure with rectangular or round cross-section, *RRR* less than 350 and cross-sectional area less than 3 mm^2, and have received a reaction heat-treatment. Ideally, it is intended that the specimens be as straight as possible; however, this is not always the case, thus care must be taken to measure the specimen in its as received condition. All measurements are done without an applied magnetic field.

The method described in the body of this standard is the “reference” method and optional acquisition methods are outlined in Clause A.3.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60050-815, *International Electrotechnical Vocabulary – Part 815: Superconductivity*

3 Terms and definitions

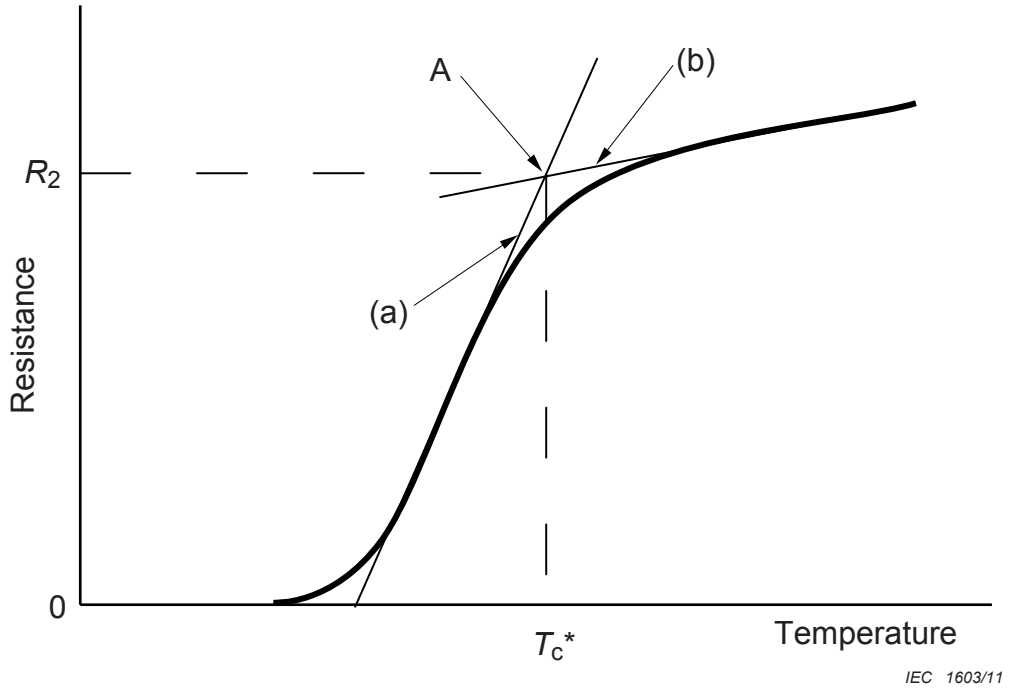
For the purposes of this document, the terms and definitions given in IEC 60050-815 and the following apply.

3.1
residual resistance ratio
RRR
the ratio of resistance at room temperature to the resistance just above the superconducting transition

NOTE In this standard for Nb_3Sn composite superconductors, the room temperature is defined as 293°K (20°C), and the residual resistance ratio is obtained in Equation (1) below, where the resistance (R_1) at 293°K is divided by the resistance (R_2) just above the superconducting transition.

$$RRR = \frac{R_1}{R_2} \quad (1)$$

Figure 1 shows schematically a resistance versus temperature curve acquired on a specimen while measuring cryogenic resistance. Draw a line in Figure 1 where the resistance sharply increases (a), and draw also a line in Figure 1 where the resistance increases gradually (b) with temperature. The value of resistance at the intersection of these two lines at $T=T_c^*$, A, is defined as resistance (R_2) just above the superconducting transition.



Temperature T_c* is that at the intersection point.

Figure 1 – Relationship between temperature and resistance

4 Requirements

The resistance measurement both at room and cryogenic temperatures shall be performed with the four-terminal technique.

The target relative combined standard uncertainty of this method is defined as an expanded uncertainty (k = 2) not to exceed 10°% based on the coefficient of variation (COV) of 5°% in the intercomparison test (see Clause C.2).

5 Apparatus

5.1 Material of measuring base plate

Material of the measuring base plate shall be copper, aluminum, silver or the like whose thermal conductivity is equal to or better than 100°W/(m·K) at liquid helium temperature (4,2 K). The surface of the material shall be covered with an insulating layer (tape or a layer made of polyethylene terephthalate, polyester, polytetrafluoroethylene, etc.) whose thickness is 0,1°mm or less.

5.2 Length of the measuring base plate

The measuring base plate shall be at least 30°mm long in one dimension.

5.3 Cryostat for the resistance, R_2, measurement

The cryostat shall include a specimen support structure and a liquid helium reservoir for the resistance, R_2, measurement. The specimen support structure shall allow the specimen, which is mounted on a measurement base plate, to be lowered and raised into and out of a liquid helium bath. In addition, the specimen support structure shall be made so that a current

can flow through the specimen and the resulting voltage generated along the specimen can be measured.

6 Specimen preparation

The test specimen shall have no joints or splices, and shall be 30°mm or longer. The distance between two voltage taps (L) shall be 25°mm or longer. A thermometer for measuring cryogenic temperature shall be attached near the specimen.

Some mechanical method shall be used to hold the specimen against the insulated layer of the measurement base plate. Special care shall be taken during instrumentation and installation of the specimen on the measurement base plate so that no excessive force, which may cause undesired bending strain or tensile strain, shall be applied to the specimen.

The specimen shall be instrumented with current contacts near each end of the specimen and a pair of voltage contacts over a central portion of the specimen. The specimen shall be mounted on a measurement base plate for these measurements. Both resistance measurements, R_1 and R_2, shall be made on the same specimen and the same mounting.

7 Data acquisition and analysis

7.1 Resistance (R_1) at room temperature

The mounted specimen shall be measured at room temperature (T_m (K)), where T_m satisfies the following condition $273 \leq T_m \leq 308$. A specimen current (I_1 (A)) shall be applied so that the current density is in the range of 0,1°A/mm^2 to 1°A/mm^2 based on the total wire cross-sectional area, and the resulting voltage (U_1 (V)), I_1 and T_m shall be recorded.

Equation°(2) below shall be used to calculate the resistance (R_m) at room temperature. The resistance (R_1) at 293 K shall be calculated using equation (3) for a wire with Cu stabilizer. The value of R_1 shall be set equal to R_m, without any temperature correction, for wires that do not contain a pure Cu component.

$$R_m = \frac{U_1}{I_1} \tag{2}$$

$$R_1 = \frac{R_m}{[1 + 0{,}00393 \cdot (T_m - 293)]} \tag{3}$$

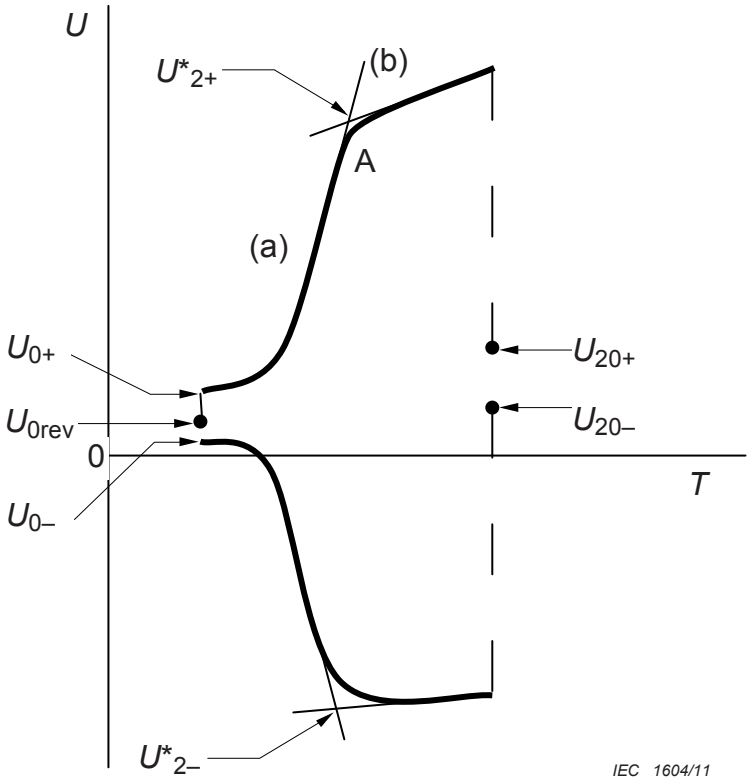
7.2 Resistance (R_2) just above the superconducting transition

7.2.1 The specimen, which is still mounted as it was for the room temperature measurement, shall be placed in the cryostat for electrical measurement specified under 5.3. Alternate cryostats that employ a heating element to sweep the specimen temperature are described in Clause A.2.

7.2.2 The specimen shall be slowly lowered into the liquid helium bath and cooled to liquid helium temperature over a time period of at least 5°min.

7.2.3 During the acquisition phases of the low-temperature R_2 measurements, a specimen current (I_2) shall be applied so that the current density is in the range of 0,1°A/mm^2 to 10 A/mm^2 based on the total wire cross-sectional area and the resulting voltage (U(V)), I_2(A), and specimen temperature (T (K)) shall be recorded. In order to keep the ratio of signal to noise high enough, the measurement shall be carried out under the condition that the

absolute value of resulting voltage above the superconducting transition exceeds 10 μV. An illustration of the data to be acquired and its analysis is shown in Figure 2.



Voltages with subscripts + and – are those obtained in the first and second measurements under positive and negative currents, respectively, and U_{20+} and U_{20-} are those obtained at zero current. For clarity, U_{0rev} is not shown coincident with U_{0-}. Voltages U_{2+}* and U_{2-}* with asterisk are those at the intersection points.

Figure 2 – Voltage (*U*) versus temperature (*T*) curves and definitions of each voltage

7.2.4 When the specimen is in superconducting state and test current (I_2) is applied, two voltages shall be measured nearly simultaneously: U_{0+} (the initial voltage recorded with a positive current polarity) and U_{0rev} (the voltage recorded during a brief change in applied current polarity). A valid R_2 measurement requires that excessive interfering voltages are not present and that the specimen is initially in the superconducting state. Thus, the following condition shall be met for a valid measurement:

$$\frac{|U_{0+} - U_{0rev}|}{\overline{U}_2} < 1\ \% \tag{4}$$

where $\overline{U}_2$ is the average voltage for the specimen in the normal state at cryogenic temperature, which is defined in 7.2.10.

7.2.5 The specimen shall be gradually warmed so that it changes to the normal state completely. When the cryostat for the resistance measurement specified under 5.3 is used, this can be achieved simply by raising the specimen to an appropriate position above the liquid helium level.

7.2.6 The specimen voltage versus temperature curve shall be acquired with the rate of temperature increase maintained between 0,1 K/min and 10 K/min.

7.2.7 The voltage versus temperature curve shall continue to be recorded during the transition into the normal state, up to a temperature somewhat less than 25 K. Then, the specimen current shall be decreased to zero and the corresponding voltage, U_{20+}, shall be recorded at a temperature below 25 K.

7.2.8 The specimen shall then be slowly lowered into the liquid helium bath and cooled to the same temperature, within ± 1 K, where the initial voltage signal U_{0+} was recorded. A specimen current, I_2, with the same magnitude but negative polarity (polarity opposite that used for the initial curve) shall be applied and the voltage U_{0-} shall be recorded at this temperature. The procedural steps 7.2.5 to 7.2.7 shall be repeated to record the voltage versus temperature curve with this negative current. In addition, the recording of U_{20-} shall be made at the same temperature, within ± 1 K, where U_{20+} was recorded.

7.2.9 Each of the two voltages versus temperature curves shall be analyzed by drawing a line (a) through the data where the absolute value of voltage sharply increases with temperature (see Figure 2) and drawing a second line (b) through the data above the transition where the voltage is raised gradually and almost linearly with temperature increase. U_{2+}* and U_{2-}* in Figure 2 shall be determined at the intersection of these two lines for the positive and negative polarity curves respectively.

7.2.10 The corrected voltages, U_{2+} and U_{2-}, shall be calculated using the following equations, $U_{2+} = U_{2+}^* - U_{0+}$ and $U_{2-} = U_{2-}^* - U_{0-}$. The average voltage, $\overline{U}_2$, shall be defined as

$$\overline{U}_2 = \frac{|U_{2+} - U_{2-}|}{2} \tag{5}$$

7.2.11 A valid R_2 measurement requires that the shift of thermoelectric voltage be within acceptable limits during the measurements of the U_{2+} and U_{2-}. Thus, the following condition shall be met for a valid measurement,

$$\frac{|\Delta_+ - \Delta_-|}{\overline{U}_2} < 3\ \% \tag{6}$$

where Δ_+ and Δ_- are defined as $\Delta_+ = U_{20+} - U_{0+}$ and $\Delta_- = U_{20-} - U_{0-}$. If the R_2 measurement does not meet the validity requirements in 7.2.4 and this subclause, then improvement steps either in hardware or experimental operation shall be taken to meet these requirements before results are reported.

7.2.12 Equation (7) shall be used to calculate the measured resistance (R_2) just above the superconducting transition.

$$R_2 = \frac{\overline{U}_2}{I_2} \tag{7}$$

7.3 Residual resistance ratio (*RRR*)

The *RRR* shall be calculated using Equation°(1).

8 Uncertainty and stability of the test method

8.1 Temperature

The room temperature shall be determined with a standard uncertainty not to exceed 0,6°K, while holding the specimen, which is mounted on the measuring base plate, at room temperature.

8.2 Voltage measurement

For the resistance measurement, the voltage signal shall be measured with a relative standard uncertainty not to exceed 0,5 %.

8.3 Current

When the current is directly applied to the specimen with a programmable DC current source, the specimen test current shall be determined with a standard uncertainty not to exceed 0,3°%.

When the specimen test current is determined from a voltage-current characteristic of a standard resistor by the four-terminal technique, the standard resistor, with a relative combined standard uncertainty not to exceed 0,3°%, shall be used .

The fluctuation of d.c. specimen test current, provided by a d.c. power supply, shall be less than 0,5 % during every resistance measurement.

8.4 Dimension

The distance along the specimen between the two voltage taps, (L), shall be determined with a relative combined standard uncertainty not to exceed 5°%.

9 Test report

9.1 *RRR* value

The obtained *RRR* value shall be reported as

$$RRR(1 \pm U_{re}) \quad (n = \cdot \cdot \cdot), \tag{8}$$

where $U_{re} = 2u_r$ ($k = 2$) is the expanded relative uncertainty with u_r denoting the uncertainty, k is a coverage factor and n is the sampling number. It is desired that n be larger than 4 so that the normal distribution can be assumed for the estimation of the standard deviation. If n is not sufficiently large, a square distribution shall be assumed. In case of $n = 1$ the analytic method described in Annex C shall be used with $b/R_2 = 1{,}46 \times 10^{-2}$ estimated from the intercomparison test.

9.2 Specimen

The test report for the result of the measurements shall also include the following items, if known:

a) manufacturer;

b) classification and/or symbol;

c) shape and area of the cross-section;

d) dimensions of the cross-sectional area;

e) number of filaments;

f) diameter of the filaments;

g) Cu to non-copper ratio.

9.3 Test conditions

9.3.1 The following test conditions shall be reported for the measurements of R_1 and R_2:

a) total length of the specimen;

b) distance between the voltage taps (L) ;

c) length of the current contacts;

d) transport currents (I_1 and I_2);

e) current densities (I_1 and I_2 divided by the total wire cross-sectional area);

f) voltages (U_1, U_{0+}, U_{0rev}, U_{2+}*, U_{20+}, U_{0-}, U_{2-}*, U_{20-}, and $\overline{U}_2$);

g) resistances (R_m, R_1, and R_2) ;

h) material, shape, and dimensions of the base plate;

i) installation method of the specimen in the base plate;

j) insulating material of the base plate.

9.3.2 The following test conditions shall be reported for the measurements of R_1:

a) temperature setting and holding method of the specimen;

b) T_m: Temperature for measurement of R_m.

9.3.3 The following test conditions shall be reported for the measurements of R_2:

a) rate of increasing temperature;

b) method of cooling down and heating up.

Annex A
(informative)

Additional information relating to the measurement of the residual resistance ratio (*RRR*)

A.1 Recommendation on specimen mounting orientation

Horizontal mounting of the wire on the base plate is recommended, since this mounting orientation can reduce possible thermal gradient along the wire compared to the vertical mounting orientation. Here the horizontal mounting orientation means that the wire axis is parallel to the surface of liquid helium.

A.2 Alternate methods for increasing temperature of specimen above superconducting transition temperature

The following methods are also recommended for increasing temperature above the superconducting transition of the specimen. The rate of increasing temperature of the whole specimen within a range between 0,1 K/min and 10 K/min should be applied for these methods. In order to dampen the rate of increasing temperature and to avoid a large temperature gradient, special care should be taken in selecting heater power, heat capacity (the specimen with the measuring base plate) and the distance between the heater and the specimen.

a) Heater method

   The specimen can be heated above the superconducting transition by a heater installed in the measuring base plate after taking the specimen out of the liquid helium bath in the cryostat.

b) Adiabatic methods

   1) Adiabatic method

      In this method, the cryostat holds a chamber in which the specimen, a sample holder, a heater and so on are contained. Before the chamber is immersed in the liquid helium bath, air inside the chamber is pumped out and helium gas is filled. Then, the chamber is immersed in the liquid helium bath and the specimen will be cooled to a temperature of 5 K or lower. After the helium gas is pumped out, the specimen can be heated above the superconducting transition by the heater under adiabatic condition.

   2) Quasi-adiabatic method

      In this method, the cryostat holds the specimen a certain distance above the liquid helium bath for the entire cryogenic measurement. A thermal anchor from the measuring base plate to the liquid helium bath allows the specimen to be cooled to a temperature of 5 K or lower. The specimen can be heated above the superconducting transition by a heater located in the measuring base plate under quasi-adiabatic condition.

c) Refrigerator method

   In this method, an electromechanical apparatus (a refrigerator) is used to cool the specimen, which is mounted to a measuring base plate, to a temperature of 5 K or lower. The specimen can be heated above the superconducting transition by a heater or by controlling the refrigerator power.

A.3 Alternative R_2 measurement method

The following methods can optionally be used for acquisition of R_2.

a) Fixed temperature method

In this method R_2 is directly determined at a fixed temperature of 20°K, which is above and near the transition temperature, instead of the method described in 7.2. In this case, it is desirable that the whole specimen is at a uniform temperature and the fixed temperature of 20°K should be determined with a combined standard uncertainty not to exceed 0,6°K. The fixed temperature and the combined standard uncertainty should be noted in the test report. Also the U_{0+} and U_{0-}, which are defined in the body of the text, should be recorded as the zero voltage level in the fixed method. In order to eliminate the influence of thermoelectric voltage, two voltage signals of the specimen, say U_{2+} and U_{2-}, should be acquired nearly simultaneously by reversal of the test current. For the fixed method, the effect of thermoelectric voltage on determination of cryogenic resistance R_2 can well be eliminated.

b) Computer-based method

A computer can be used to control the current direction and warming of the specimen and to measure the voltage-temperature curve. Changes in current direction by periodic current reversals or periodic current on and off cycles are used to correct for off-set voltages in order that the measurements can be made during one cycle of changing the specimen temperature. This method is useful when the transition to the normal state is not too fast. The effect of thermoelectric voltage should also be checked.

c) Other simplified methods with periodic checks

Simplified methods without temperature measurement might also be accepted, if an operator with sufficient experience performs the measurement using a given apparatus and if the following condition is satisfied. If a simplified laboratory practice can be shown, through periodic checks, to achieve the same result as the method in this standard, within its stated uncertainty, then the simplified practice can be used in place of this reference method. These periodic checks could be accomplished by doing one of the following:

1) an interlaboratory comparison where one laboratory uses the reference method and another laboratory uses their simplified method;
2) a single laboratory comparison where one laboratory “checks” its simplified method against the reference method;
3) periodic measurement of a small set of reference samples with well-known *RRR* values using the simplified method.

Annex B
(informative)

Uncertainty considerations

B.1 Overview

In 1995, a number of international standards organizations, including IEC, decided to unify the use of statistical terms in their standards. It was decided to use the word “uncertainty” for all quantitative (associated with a number) statistical expressions and eliminate the quantitative use of “precision” and “accuracy.” The words “accuracy” and “precision” could still be used qualitatively. The terminology and methods of uncertainty evaluation are standardized in the Guide to the Expression of Uncertainty in Measurement (GUM) [1][1].

It was left to each TC to decide if they were going to change existing and future standards to be consistent with the new unified approach. Such change is not easy and creates additional confusion, especially for those who are not familiar with statistics and the term uncertainty. At the June 2006 TC 90 meeting in Kyoto, it was decided to implement these changes in future standards.

Converting “accuracy” and “precision” numbers to the equivalent “uncertainty” numbers requires knowledge about the origins of the numbers. The coverage factor of the original number may have been 1, 2, 3 or some other number. A manufacturer’s specification that can sometimes be described by a rectangular distribution will lead to a conversion number of $1/\sqrt{3}$. The appropriate coverage factor was used when converting the original number to the equivalent standard uncertainty. The conversion process is not something that the user of the standard needs to address for compliance to TC 90 standards, it is only explained here to inform the user about how the numbers were changed in this process. The process of converting to uncertainty terminology does not alter the user’s need to evaluate their measurement uncertainty to determine if the criteria of the standard are met.

The procedures outlined in TC 90 measurement standards were designed to limit the uncertainty of any quantity that could influence the measurement, based on the Convener’s engineering judgment and propagation of error analysis. Where possible, the standards have simple limits for the influence of some quantities so that the user is not required to evaluate the uncertainty of such quantities. The overall uncertainty of a standard was then confirmed by an interlaboratory comparison.

B.2 Definitions

Statistical definitions can be found in three sources: the GUM, the International Vocabulary of Basic and General Terms in Metrology (VIM)[2], and the NIST Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results (NIST)[3]. Not all statistical terms used in this standard are explicitly defined in the GUM. For example, the terms “relative standard uncertainty” and “relative combined standard uncertainty” are used in the GUM (5.1.6, Annex J), but they are not formally defined in the GUM (see [3]).

B.3 Consideration of the uncertainty concept

Statistical evaluations in the past frequently used the Coefficient of Variation (COV) which is the ratio of the standard deviation and the mean (N.B. the COV is often called the relative standard deviation). Such evaluations have been used to assess the precision of the measurements and give the closeness of repeated tests. The standard uncertainty (SU)

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1 The figures in square brackets refer to the reference documents in Clause B.5 of this Annex.

depends more on the number of repeated tests and less on the mean than the COV and therefore in some cases gives a more realistic picture of the data scatter and test judgment. The example below shows a set of electronic drift and creep voltage measurements from two nominally identical extensometers using same signal conditioner and data acquisition system. The n = 10 data pairs are taken randomly from the spreadsheet of 32 000 cells. Here, extensometer number one (E_1) is at zero offset position whilst extensometer number two (E_2) is deflected to 1°mm. The output signals are in volts.

Table B.1 – Output signals from two nominally identical extensometers

Output signal [V]	
E_1	E_2
0,001 220 70	2,334 594 73
0,000 610 35	2,334 289 55
0,001 525 88	2,334 289 55
0,001 220 70	2,334 594 73
0,001 525 88	2,334 594 73
0,001 220 70	2,333 984 38
0,001 525 88	2,334 289 55
0,000 915 53	2,334 289 55
0,000 915 53	2,334 594 73
0,001 220 70	2,334 594 73

Table B.2 – Mean values of two output signals

Mean ($\overline{X}$) [V]	
E_1	E_2
0,001 190 19	2,334 411 62

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n} \qquad [\mathrm{V}] \tag{B.1}$$

Table B.3 – Experimental standard deviations of two output signals

Experimental standard deviation (s) [V]	
E_1	E_2
0,000 303 48	0,000 213 381

$$s = \sqrt{\frac{1}{n-1} \cdot \sum_{i=1}^{n} \left(X_i - \overline{X}\right)^2} \qquad [\mathrm{V}] \tag{B.2}$$

Table B.4 – Standard uncertainties of two output signals

Standard uncertainty (u) [V]	
E_1	E_2
0,000 095 97	0,000 067 48

$$u = \frac{s}{\sqrt{n}} \quad [\mathrm{V}] \tag{B.3}$$

Table B.5 – Coefficient of variations of two output signals

Coefficient of variation (COV) [%]	
E_1	E_2
25,498 2	0,009 1

$$COV = \frac{s}{\overline{X}} \tag{B.4}$$

The standard uncertainty is very similar for the two extensometer deflections. In contrast, the coefficient of variation *COV* is nearly a factor of 2 800 different between the two data sets. This shows the advantage of using the standard uncertainty which is independent of the mean value.

B.4 Uncertainty evaluation example for TC 90 standards

The observed value of a measurement does not usually coincide with the true value of the measurand. The observed value may be considered as an estimate of the true value. The uncertainty is part of the "measurement error" which is an intrinsic part of any measurement. The magnitude of the uncertainty is both a measure of the metrological quality of the measurements and improves the knowledge about the measurement procedure. The result of any physical measurement consists of two parts: an estimate of the true value of the measurand and the uncertainty of this “best” estimate. The GUM, within this context, is a guide for a transparent, standardized documentation of the measurement procedure. One can attempt to measure the true value by measuring “the best estimate” and using uncertainty evaluations which can be considered as two types: Type A uncertainties (repeated measurements in the laboratory in general expressed in the form of Gaussian distributions) and Type B uncertainties (previous experiments, literature data, manufacturer’s information, etc. often provided in the form of rectangular distributions).

The calculation of uncertainty using the GUM procedure is illustrated in the following example:

a) The user must derive in a first step a mathematical measurement model in form of identified measurand as a function of all input quantities. A simple example of such a model is given for the uncertainty of a force measurement using a load cell:

   Force as measurand = W (weight of standard as expected) + d_W (manufacturer’s data) + d_R (repeated checks of standard weight/day) + d_{Re} (reproducibility of checks at different days).

   Here the input quantities are: the measured weight of standard weights using different balances (Type°A), manufacturer’s data (Type°B), repeated test results using the digital electronic system (Type B), and reproducibility of the final values measured on different days (Type B).

b) The user should identify the type of distribution for each input quantity (e.g. Gaussian distributions for Type°A measurements and rectangular distributions for Type°B measurements).

c) Evaluate the standard uncertainty of the Type A measurements,

$u_A = \frac{s}{\sqrt{n}}$ where, s is the experimental standard deviation and n is the total number of measured data points.

d) Evaluate the standard uncertainties of the Type°B measurements:

$u_B = \sqrt{\frac{1}{3} \cdot d_W{}^2 + ...}$ where, d_W is the range of rectangular distributed values

e) Calculate the combined standard uncertainty for the measurand by combining all the standard uncertainties using the expression:

$$u_c = \sqrt{u_A^2 + u_B^2}$$

In this case, it has been assumed that there is no correlation between input quantities. If the model equation has terms with products or quotients, the combined standard uncertainty is evaluated using partial derivatives and the relationship becomes more complex due to the sensitivity coefficients [4, 5].

f) Optional – the combined standard uncertainty of the estimate of the referred measurand can be multiplied by a coverage factor (e. g. 1 for 68 % or 2 for 95 % or 3 for 99 %) to increase the probability that the measurand can be expected to lie within the interval.

g) Report the result as the estimate of the measurand ± the expanded uncertainty, together with the unit of measurement, and, at a minimum, state the coverage factor used to compute the expanded uncertainty and the estimated coverage probability.

To facilitate the computation and standardize the procedure, use of appropriate certified commercial software is a straightforward method that reduces the amount of routine work [6, 7]. In particular, the indicated partial derivatives can be easily obtained when such a software tool is used. Further references for the guidelines of measurement uncertainties are given in [3, 8, and 9].

B.5 Reference documents of Annex B

[1] ISO/IEC Guide 98-3:2008, *Uncertainty of measurement – Part 3: Guide to the expression of uncertainty in measurement* (GUM 1995)

[2] ISO/IEC Guide 99:2007, *International vocabulary of metrology – Basic and general concepts and associated terms (VIM)*

[3] TAYLOR, B.N. and KUYATT, C.E. *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results.* NIST Technical Note 1297, 1994

[4] KRAGTEN, J. Calculating standard deviations and confidence intervals with a universally applicable spreadsheet technique. *Analyst*, 1994,119, 2161-2166

[5] EURACHEM / CITAC Guide CG 4, Second edition:2000, *Quantifying Uncertainty in Analytical Measurement*

[6] Available at http://www.gum.dk/e-wb-home/gw_home.html (cited 2011-04-04)

[7] Available at <http://www.isgmax.com/> (cited 2011-04-04)

[8] CHURCHILL, E., HARRY, H.K., and COLLE,R., *Expression of the Uncertainties of Final Measurement Results*. NBS Special Publication 644 (1983)

[9] JAB NOTE Edition 1:2003, *Estimation of Measurement Uncertainty (Electrical Testing / High Power Testing)*

Annex C
(informative)

Uncertainty evaluation in test method of *RRR* for Nb_3Sn

C.1 Evaluation of uncertainty

Uncertainty in the residual resistance ratio is composed of the uncertainty in the room temperature resistance (u_{R1}) and that in the cryogenic resistance (u_{R2}). In the following the coverage factor k is assumed to be 1 for simplicity.

The residual resistance ratio of the superconducting wire is given by $X=R_1/R_2$. If the deviations of R_1 and R_2 from their statistical averages are ΔR_1 and ΔR_2, the deviation of the residual resistance ratio, ΔX, is

$$\frac{\Delta X}{X} = \frac{\Delta R_1}{R_1} - \frac{\Delta R_2}{R_2}. \tag{C.1}$$

Hence, the relative uncertainty of X is

$$\frac{u}{X} = \left[\left(\frac{u_{R1}}{R_1} \right)^2 + \left(\frac{u_{R2}}{R_2} \right)^2 \right]^{1/2}. \tag{C.2}$$

Since the room temperature resistance is given by

$$R_1 = \frac{U_1}{[1 + 0{,}00393(T_m - 293)]I_1} \qquad [\Omega], \tag{C.3}$$

the deviation of R_1 is

$$\begin{aligned} \Delta R_1 &= \frac{\partial R_1}{\partial U_1}\Delta U_1 + \frac{\partial R_1}{\partial T_m}\Delta T_m + \frac{\partial R_1}{\partial I_1}\Delta I_1 \\ &= \frac{1}{1 + 0{,}00393(T_m - 293)}\left(\frac{\Delta U_1}{I_1} - 0{,}00393 R_1 \Delta T_m - \frac{U_1}{I_1^2}\Delta I_1 \right) \\ &\cong \frac{\Delta U_1}{I_1} - 0{,}00393 R_1 \Delta T_m - \frac{U_1}{I_1^2}\Delta I_1 \qquad [\Omega], \end{aligned} \tag{C.4}$$

where ΔU_1, ΔT_m and ΔI_1 are the deviations of the voltage, temperature and applied current, respectively. The approximation in equation (C.4) is based on the fact that the effect of difference of temperature from 293°K (20°C) on sensitivity coefficients is small. Its effect on the final target uncertainty is 0,2°% at most (for measurement at 273 K (0°C)). The corresponding deviation of the room temperature can be divided as

$$\Delta T_m = \Delta T_{m1} + \Delta T_{m2} \qquad [\mathrm{K}], \tag{C.5}$$

where ΔT_{m1} is a difference between the measured room temperature and the specimen temperature, and ΔT_{m2} is the deviation caused by the bolometer. Thus, the uncertainty in the room temperature resistance is given by

$$u_{R1} = \left[\left(\frac{u_{U1}}{I_1} \right)^2 + u_{RTm1}^2 + (0{,}00393 R_1)^2 u_{Tm2}^2 + \left(\frac{U_1}{I_1^2} \right)^2 u_{I1}^2 \right]^{1/2} \quad [\Omega], \tag{C.6}$$

where u_{U1} [V] is the type B uncertainty in the room temperature voltage ($u_{U1}/U_1 = 0{,}005/\sqrt{3}$), u_{I1} [A] is the type B uncertainty in the room temperature current ($u_{I1}/I_1 = 0{,}005/\sqrt{3}$), u_{Tm2} [K] is the type B uncertainty in the room temperature measurement using a bolometer ($u_{Tm2} = 1/\sqrt{3}$ [K]). The u_{RTm1} [Ω] is the type B uncertainty in R_1 due to the difference of the room temperature from the specimen temperature and is formally expressed as $u_{RTm1} = -0{,}00393 I_1 u_{Tm1}$. However, u_{Tm1} is not obtained from mathematical model but u_{Tm1} is directly estimated as $\pm 1{,}7\%$ of R_1 from the results of round robin test on *RRR* of Nb-Ti [1][2]. Assuming a similar situation, it can also be assumed as $u_{RTm1}/R_1 = 0{,}017/\sqrt{3}$.

In the cryogenic resistance measurement the specimen voltage is measured twice with changing the current direction. It should be noted that the voltage at the transition is determined by drawing two straight lines and an appreciable uncertainty may appear in these analyses. This uncertainty is denoted by *b*. Then, the uncertainty in the cryogenic temperature resistance is similarly given by

$$u_{R2} = \left[2\left(\frac{u_{U2}}{I_2} \right)^2 + 2b^2 + \left(\frac{U_2}{I_2^2} \right)^2 u_{I2}^2 \right]^{1/2} \quad [\Omega], \tag{C.7}$$

where u_{U2} [V] is the type B uncertainty due to the voltmeter, u_{I2} [A] is the type B uncertainty in the current. In the above $u_{U2}/U_2 = 0{,}005/\sqrt{3}$ and $u_{I2}/I_2 = 0{,}005/\sqrt{3}$.

From the above analysis the relative uncertainty in the residual resistance ratio is given by

$$\frac{u}{(R_1/R_2)} = \left[\left(\frac{u_{R1}}{R_1} \right)^2 + \left(\frac{u_{R2}}{R_2} \right)^2 \right]^{1/2} = \left[1{,}43 \times 10^{-4} + 2\left(\frac{b}{R_2} \right)^2 \right]^{1/2}. \tag{C.8}$$

Table C.1 – Uncertainty of each measurement

uncertainty	type	value	remarks
u_{U1}/U_1	B	$0{,}005/\sqrt{3}$	$\lvert\Delta U_1\rvert/U_1 < 0{,}005$
u_{I1}/I_1	B	$0{,}005/\sqrt{3}$	$\lvert\Delta I_1\rvert/I_1 < 0{,}005$
u_{Tm}	B	$1/\sqrt{3}$ K	$\lvert\Delta T_m\rvert < 1$K
u_{U2}/U_2	B	$0{,}005/\sqrt{3}$	$\lvert\Delta U_2\rvert/U_2 < 0{,}005$
u_{I2}/I_2	B	$0{,}005/\sqrt{3}$	$\lvert\Delta I_2\rvert/I_2 < 0{,}005$

2 The references in square brackets refer to the reference documents at the end of this annex.

C.2 Reason for large COV value in the intercomparison test

The COV of the intercomparison test for Nb_3Sn samples was 6,07 % [2]. This value is much larger than that for Nb-Ti, 2,44 %, although there is no contribution from additional uncertainty in correction of the strain effect. For clarification of this reason an intercomparison test was performed between two laboratories for three Nb_3Sn samples, the two of which were cut from the same batch of heat treatment. The *RRR* value obtained using the reference method agreed within 1 % between the two laboratories for the three samples as shown in Table C.2, while the *RRR* values were different between the two samples obtained from the same batch. This indicates that the large COV value in the former intercomparison test originated from inhomogeneity of samples, while the test method itself was fairly accurate. The source of this inhomogeneity may be due to the high sensitivity to heat treatment conditions or due to defects of the diffusion barrier. Since a common *RRR* specification is that, in order to pass, the value must be greater than a minimum value, the existence of inhomogeneities may require that several specimens of a given wire be measured and reported.

Table C.2 – Obtained values of R_1, R_2 and *RRR* for three Nb_3Sn samples.

Sample	Lab. 1			Lab. 2		
	R_1(293°K)[Ω]	$R_2(T_c^*)$[Ω]	*RRR*	R_1(293°K)[Ω]	$R_2(T_c^*)$[Ω]	*RRR*
B	$1{,}593\times10^{-3}$	$1{,}49\times10^{-5}$	107	$1{,}61\times10^{-3}$	$1{,}49\times10^{-5}$	108
C	$1{,}719\times10^{-3}$	$1{,}66\times10^{-5}$	104	$1{,}74\times10^{-3}$	$1{,}66\times10^{-5}$	105
D	$1{,}619\times10^{-3}$	$1{,}61\times10^{-5}$	100	$1{,}65\times10^{-3}$	$1{,}62\times10^{-5}$	101

For this reason the uncertainty in the test method of *RRR* for Nb_3Sn is expected to be as low as that for Nb-Ti. Therefore, the value of b/R_2=1,46 × 10^{-2} obtained in intercomparison test for *RRR* measurement in Nb-Ti can also be used to estimate the uncertainty of *RRR* in Nb_3Sn with Eq. (C.4) (see Annex C in [3]). In addition, the result shown in Table C.2 indicates that the main difference between the measurements in the two laboratories comes from the observed values of R_1. This is considered to be caused by the uncertainty in the room temperature (see Annex C in [3]).

C.3 Reference documents of Annex B

[1] MATSUSHITA T., OTABE E.S., MURASE S., OSAMURA K. and HUA CY., *Advances in Superconductivity* XI, Tokyo, Springer, 1999, p. 1507.

[2] MURASE S., SAITOH T., MORIAI H., MATSUSHITA T and OSAMURA K., *Advances in Superconductivity* XI, Tokyo, Springer, 1999, p.1511.

[3] IEC 61788-4 [3)], *Superconductivity – Part 4: Residual resistance ratio measurement – Residual resistance ratio of Nb-Ti composite superconductors*

3) Third edition, to be published.

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