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INTERNATIONAL STANDARD

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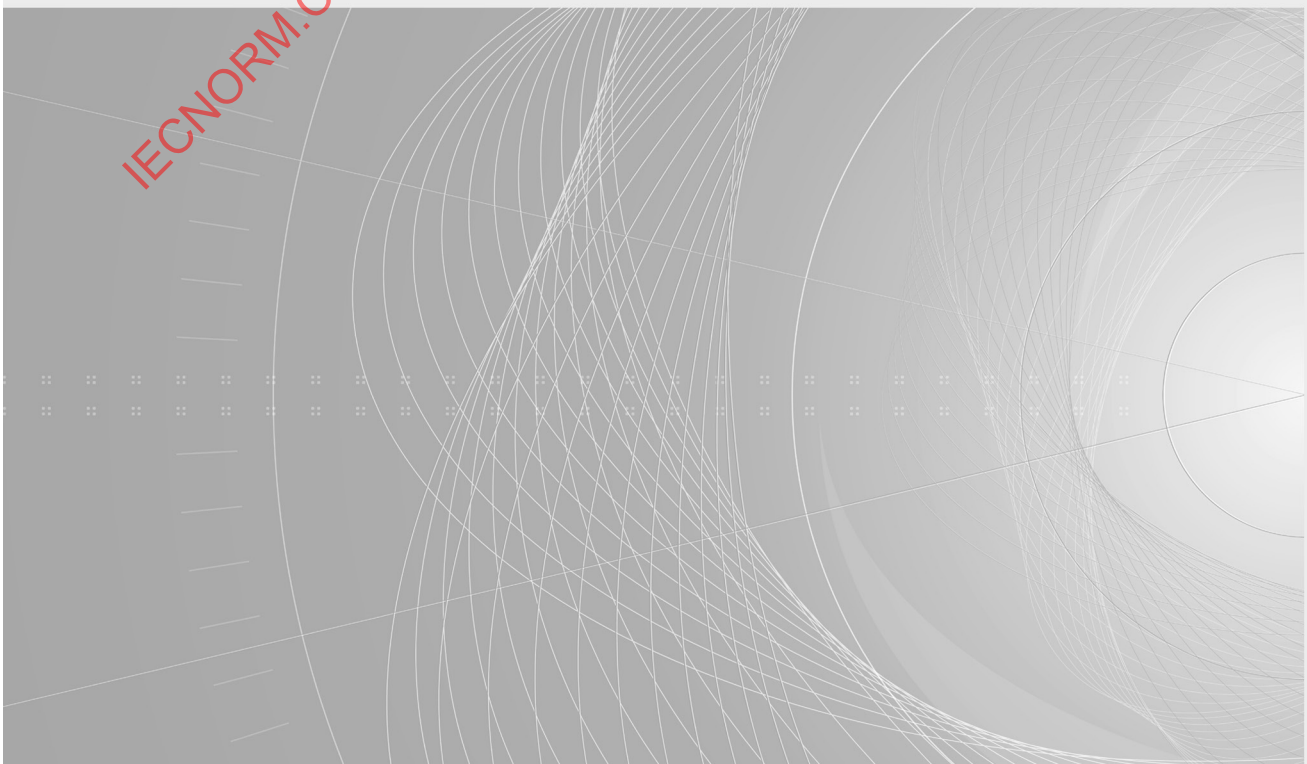
Superconductivity –
Part 23: Residual resistance ratio measurement – Residual resistance ratio of cavity-grade Nb superconductors



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SUPERCONDUCTIVITY –

Part 23: Residual resistance ratio measurement – Residual resistance ratio of cavity-grade Nb superconductors

FOREWORD

1) The International Electrotechnical Commission (IEC) is a worldwide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of IEC is to promote international co-operation on all questions concerning standardization in the electrical and electronic fields. To this end and in addition to other activities, IEC publishes International Standards, Technical Specifications, Technical Reports, Publicly Available Specifications (PAS) and Guides (hereafter referred to as "IEC Publication(s)"). Their preparation is entrusted to technical committees; any IEC National Committee interested in the subject dealt with may participate in this preparatory work. International, governmental and non-governmental organizations liaising with the IEC also participate in this preparation. IEC collaborates closely with the International Organization for Standardization (ISO) in accordance with conditions determined by agreement between the two organizations.
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This redline version of the official IEC Standard allows the user to identify the changes made to the previous edition IEC 61788-23:2021. A vertical bar appears in the margin wherever a change has been made. Additions are in green text, deletions are in strikethrough red text.

IEC 61788-23 has been prepared by IEC technical committee 90: Superconductivity. It is an International Standard.

This third edition cancels and replaces the second edition published in 2021. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to the previous edition:

a) The principle is changed to represent the present test method.

The text of this International Standard is based on the following documents:

Draft	Report on voting
90/515/FDIS	90/519/RVD

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this International Standard is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/publications.

A list of all parts in the IEC 61788 series, published under the general title *Superconductivity,* can be found on the IEC website.

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under webstore.iec.ch in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn, or
- revised.

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INTRODUCTION

High-purity niobium is the chief material used to make superconducting radio-frequency cavities. Similar grades of niobium ~~may~~ can be used in the manufacture of superconducting wire. Procurement of raw materials and quality assurance of delivered products often use the residual resistance ratio (RRR) to specify or assess the purity of a metal. RRR is defined for non-superconducting metals as the ratio of electrical resistance measured at room temperature (293 K) to the resistance measured for the same specimen at low temperature (~4,2 K). The low-temperature value is often called the residual resistance. Higher purity is associated with higher values of RRR.

Niobium presents special problems due to its transformation to a superconducting state at ~9 K, so DC electrical resistance is effectively zero below this temperature. The definition above would then yield an infinite value for RRR. This document describes a test method to determine the residual resistance value by using a plot of the resistance to temperature as the test specimen is gradually warmed through the superconducting transition in the absence of an applied magnetic field. This results in a determination of the residual resistance at just above superconducting transition, ~10 K, from which RRR is subsequently determined.

International Standards also exist to determine the RRR of superconducting wires. In contrast to superconducting wires, which are usually a composite of a superconducting material and a non-superconducting material and the RRR value is representative of only the non-superconducting component, here the entire specimen is composed of superconducting niobium. Frequently, niobium is procured as a sheet, bar, ~~tube,~~ or rod, and not as a wire. For such forms, test specimens will likely be a few millimetres in the dimensions transverse to electric current flow. This difference is significant when making electrical resistance measurements, since niobium samples will likely be much longer than that for the same length-to-diameter ratio as a wire, and higher electrical current ~~may~~ can be required to produce sufficient voltage signals. Guidance for sample dimensions and electrical connections is provided in Annex A. Test apparatus should also take into consideration aspects such as the orientation of a test specimen relative to the liquid helium surface, accessibility through ports on common liquid helium dewars, design of current contacts, and minimization of thermal gradients over long specimen lengths. These aspects distinguish this document from similar wire standards.

Other test methods have been used to determine RRR. Some methods use a measurement at a temperature other than 293 K for the high resistance value. Some methods use extrapolations at 4,2 K in the absence of an applied magnetic field for the low resistance value. Other methods use an applied magnetic field to suppress superconductivity at 4,2 K. A comparison between this document and some other test methods is presented in Annex A. Note that systematic differences of up to 10 % are produced by these other methods, which is larger than the target uncertainty of this document. It is therefore important to apply this document or the appropriate corrections listed in Annex A according to the test method used.

Whenever possible, this test method should be transferred to vendors and collaborators who also perform RRR measurements. To promote consistency, the results of inter-laboratory comparisons are described in Clause C.2.

SUPERCONDUCTIVITY –

Part 23: Residual resistance ratio measurement – Residual resistance ratio of cavity-grade Nb superconductors

1 Scope

This part of IEC 61788 addresses a test method for the determination of the residual resistance ratio (RRR), r_{RRR}, of cavity-grade niobium. This method is intended for high-purity niobium grades with 150 < r_{RRR} < 600. The test method is valid for specimens with rectangular or round cross-section, cross-sectional area greater than 1 mm^2 but less than 20 mm^2, and a length not less than 10 nor more than 25 times the width or diameter.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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* (available at: https://www.electropedia.org/)

3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- IEC Electropedia: available at https://www.electropedia.org/
- ISO Online browsing platform: available at https://www.iso.org/obp

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

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

where

R_1 is the resistance at room temperature, 293 K;

R_2 is the resistance just above the superconducting transition, at ~10 K.

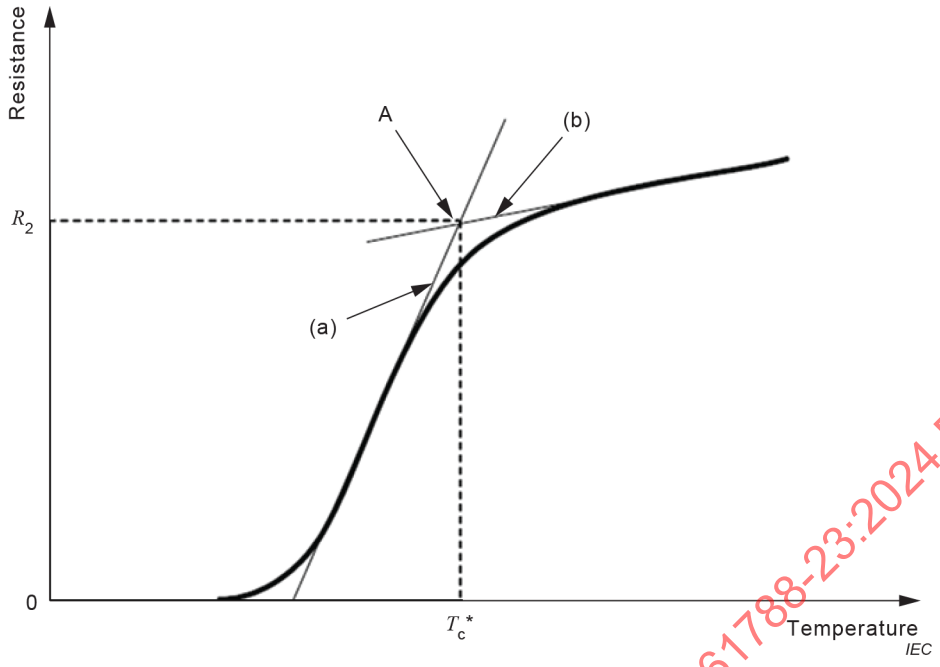

Figure 1 – Relationship between temperature and resistance near the superconducting transition

Note 1 to entry: In this document, the room temperature is defined as 20 °C = 293 K, and r_{RRR} is obtained as follows: Figure 1 shows schematically resistance versus temperature data and the graphical procedure used to determine the value of R_2. In Figure 1, the region of maximum slope is extrapolated upward in resistance, as shown by line (a), and the region of minimum slope at temperatures above the transition temperature is extrapolated downward in temperature, as shown by line (b). The intersection of these extrapolations at point A determines the value of R_2 as well as a temperature value T_c^*.

Note 2 to entry: The value T_c^* is similar to the transition value defined in [1], and ~~should not be confused with~~ is different from the value defined at the midpoint of the transition, called T_c^* in [2].

Note 3 to entry: Some standards or documented techniques, e.g. [3], [4], [5], [6], define r_{RRR} with the value of R_1 determined at a temperature other than 293 K, or the value of R_2 determined at a temperature below the superconducting transition. The user's ~~of this document should be alert for~~ attention is drawn to such differences in definition.

4 Principle

The 4-point DC electrical resistance technique shall be performed both at room temperature and at cryogenic temperature. The test of resistance ~~may~~ shall be done ~~either~~ as a function of temperature ~~or~~. Another test method of resistance as a function of time with increasing temperature is described in A.4.2.

The relative combined standard uncertainty of this method is 3 % with coverage factor 2.

Measurements shall have the following attributes.

a) Measuring current is sufficiently high to provide voltage signals of the order of 1 μV. For electrical safety, maximum current density should never exceed 1 A mm^{-2}.

b) Contact resistance for current leads is sufficiently low to avoid excessive heating of the sample. Typical cryogenic measurement conditions require power dissipation at contacts to be less than 1 mW.

c) Sample sizes are sufficiently large to minimize ~~effects from~~ the probability of cutting and handling damage. Typical samples are 1 mm to 3 mm in cross-sectional dimension and greater than 5 mm^2 in cross-sectional area.

d) Sample length is at least 10 times and not more than 25 times the width or diameter.

Annex A discusses considerations for sample dimensions and measuring current.

5 Measurement apparatus

5.1 Mandrel or base plate

A straight mandrel or base plate shall be used to support the specimen. Possible materials of construction include pure copper, pure aluminium, pure silver, electrical grades of Cu-Zr, Cu-Cr-Zr, Cu-Be, and other copper alloys, electrical grades of Al-Mg, Al-Ag, and other aluminium alloys, and electrical grades of silver alloys. These provide high thermal conductivity and serve to remove thermal gradients during measurement. The specimen shall be insulated from the mandrel. Possible insulating materials include polyethylene terephthalate, polyester, and polytetrafluoroethylene, which ~~may~~ can be applied as foils, tapes, or coatings. Glass-fibre reinforced epoxy or other composite materials with good thermal conductivity at cryogenic temperature ~~may~~ can also be used.

The base plate should have a clean and smooth surface finish. There should be no burrs, ridges, seams, or other asperities that ~~may~~ can affect the specimen. High-purity niobium specimens are soft and are susceptible to indentation by surface flaws, and such indentations ~~may~~ can alter the sample and invalidate the resistance measurement.

The mandrel or base plate shall support the entire length and width of the specimen. Mandrel or base plate geometry should not impose a bending strain of more than 0,2 % on the sample.

A thermometer accurate to 0,1 K is helpful but not required. The mandrel or base plate ~~may~~ can incorporate a mounting for a cryogenic thermometer directly against the body of the mandrel or base plate and near the centre of the test specimen.

Practical base plates are at least 30 mm in length to accommodate assembly of pieces and handling of samples by human hands. Multiple samples ~~may~~ can be mounted against a single base plate.

5.2 Cryostat and support of mandrel or base plate

The apparatus shall make provisions for mechanical support of the mandrel or base plate. In addition, such support shall provide electrical leads to carry currents for samples and thermometers, and measure their voltages. For R_1 and R_2 measurements, the support shall permit current to flow through only the sample, so that the entire resulting voltage measured is only that generated by the sample.

The support structure shall permit measurement of both R_1 and R_2 without dismounting or remounting the test specimen. Measurement of R_2 shall require the use of a cryostat, which shall, moreover, integrate with the support.

The cryostat shall include a liquid helium reservoir at the bottom of a substantial vertical column. A support structure shall accommodate the raising and lowering of the sample into or out of the helium bath. In addition, anchoring of the sample position, either when immersed in liquid helium or suspended above the surface of the liquid at an arbitrary height, shall be provided. Such suspension permits the equilibration of temperature during measurement and slow increase of temperature with height above the helium bath. Alternatively, immersion of the sample into the bath followed by reduction of the bath level via boil-off or pressurized transfer can also be used to vary temperature.

A heater ~~may~~ can be employed to warm the mandrel or base plate. The heater should be distributed along the mandrel and excessive power settings should be avoided. For instance, a point source of 1 W heat input operating at the centre of a 1 cm^2 mandrel upon which a 5 cm sample is mounted could produce ~~thermal gradients~~ temperature difference of 2,5 K along the sample if the thermal conductivity is 100 W m^{-1} K^{-1}.

Proper cryogenic techniques shall be followed for the construction of the cryostat and apparatus. This includes the use of low thermal conductivity materials such as thin-walled stainless steel tubes, composite materials, ceramics, and insulation, to prevent excessive boil-off due to heat conduction from the surroundings. A can or shield ~~may~~ can surround the base plate or mandrel with mounted sample to improve thermal stability. Provisions for pressure relief and vacuum isolation of the liquid helium should be incorporated with the apparatus.

6 Specimen preparation

High-purity niobium is quite malleable, and even the slightest force can produce deformation of the material. Since dislocations are one source of electron scattering, specimen deformation can inadvertently contribute to the residual resistivity and affect the test result. Therefore, special protocols shall be observed when preparing the specimen. Cutting techniques shall avoid heat and strain to the extent possible. Discharge machining, fluid-jet cutting, or low-speed conventional machining are acceptable and widely-used techniques for applications using high-purity niobium. Specimens cut from larger pieces shall be protected and immobilized against a support piece during transport. Operations to de-burr samples shall not bend, excessively heat or otherwise damage the sample. Light sanding with fine paper is one acceptable approach.

Specimens should be rectangular or circular bars with uniform cross-section. Long sides of the specimen shall be parallel. Any twisting or curvature shall be avoided to ensure that bending or torsion is not applied to the test specimen during mounting to the mandrel or base plate. Specimens that form an arc or a U shape are acceptable provided that the entire curvature can be supported on a plane, without applying torsion to the bent specimen.

The specimen shall be clean and have no trace of residues from cutting fluids or any other surface contaminants. Degreasing with solvents, followed by ultrasonic cleaning using a mild water-based detergent, followed by rinsing with distilled or ultra-pure water, then drying in air, is preferred for cleaning residues. Chemical etching to clean the surface poses a risk of introducing contaminants, especially hydrogen and oxygen, and should be avoided. Gentle mechanical polishing of the regions where voltage taps and current leads attach is usually sufficient to remove surface oxides. Coating these regions with indium foil or another metal, for example by evaporation or sputtering, is an acceptable method to protect polished contacts provided that coating the entire specimen is avoided.

The test specimen shall be a single piece and shall not include any joints or splices.

A mechanical method shall be used to affix the test specimen to the mandrel or base plate. Installation and instrumentation of the specimen shall not apply excessive force, bending strain, tensile strain, or torsion to the specimen.

The test specimen shall be instrumented with current contacts near each end of the specimen and a pair of voltage contacts over the central portion between the current contacts (i.e. a 4-point measurement technique). The voltage contacts shall be separated from the current contacts by a distance no smaller than the largest dimension (width, thickness, or diameter) perpendicular to the specimen length.

7 Data acquisition and analysis

7.1 Data acquisition hardware

Modern power supplies can be computer controlled and come with a variety of features that permit remote control of the current output. Use of such power supplies is not required but could greatly enable automation of the data acquisition. Pulsed modes permit application of current only when voltage signals are being acquired, thereby removing heat generated in the sample during the off cycle. If pulsed current application is used, the pulse duration shall include ample periods for voltage signals to settle and be filtered.

Some power supplies incorporate an internal shunt to regulate the output current. If such a power supply is used, the internal shunt shall be calibrated periodically with an external shunt and voltage measurement.

The test set-up ~~may~~ can establish an arbitrary baseline voltage U_0, which might be detectable when the sample is in the superconducting state and the power supply is off. The value U_0 can drift over time due to changes in the thermal environment and other effects. More complex hardware includes compensation for drift and automatic nulling such that the time average of U_0 is 0. Digital voltage meters are not required but greatly enhance the data acquisition. Besides compensation for drift and voltage nulling, filtering and internal compensation for thermally-induced voltages can improve the accuracy of the voltage measurement. Filtering should average voltage signals for a time at least as long as the thermal time constant of the apparatus at low temperature, typically of the order of 0,1 s to 10 s. It is important to understand how voltages are corrected for drift and thermal effects. Sensitive voltage meters, especially nanovolt meters, require a pre-amplifier that ~~needs to~~ shall be at thermal equilibrium, which can require several hours of operation in advance of the measurement.

Data acquisition via computer greatly facilitates the recording and reporting of data.

7.2 Resistance (R_1) at room temperature

The ambient temperature T_1 of the measurement laboratory shall be measured. A specimen current I_1 shall be applied in accordance with the requirements in Clause 4. The resulting voltage U_1 shall be recorded together with I_1 and T_1. The resistance shall be determined by

$$R_1 = \frac{U_1}{I_1}\left[1 - 0{,}003\,7\ \left(T_1 - 293\right)\right] \tag{2}$$

with T_1 in units of kelvin. The coefficient 0,003 7 reflects the experimentally observed rate of change of resistance with temperature given in [7] [1] over the interval 273 K to 300 K.

1 Numbers in square brackets refer to the Bibliography.

7.3 Residual resistance (R_2) just above the superconducting transition

The measurement of R_2 shall be made with the sample still mounted on the mandrel or base plate for the measurement of R_1.

The specimen shall be placed in a cryostat as specified in 5.2. The specimen shall be slowly lowered into a liquid helium bath and cooled to liquid helium temperature. While a vigorous boil-off of liquid helium will accompany the initial cool down, removal of heat from the mandrel, especially if it is shielded, can require a time period of more than 5 min. Current ~~may~~ can be applied, and voltage ~~may~~ can be monitored during this period, but no measurement shall be made until the vigorous boil-off of liquid helium has subsided.

After the boil-off rate is suitable for measurements, a voltage measurement U_0 shall be recorded while the sample is immersed in liquid helium. The sample is likely to be in the superconducting state under these conditions. Current I_2 shall then be applied in accordance with requirements of Clause 4 and with considerations of Clause 5. Voltage readings U_0^+ and U_0^- shall be acquired for forward and reverse current polarity, respectively. Any differences between U_0, U_0^+, and U_0^- shall be recorded.

The specimen shall then be gradually warmed so that a transition from the superconducting state into the normal state occurs gradually. An apparatus that conforms to Clause ~~6~~ 5 will permit gradual warming of the specimen by raising the level of the mandrel above the level of the liquid helium bath, for example. Two voltages U_2^+ and U_2^- shall be measured almost simultaneously with the application of the same measuring current I_2 with forward and reverse polarity, respectively. The current shall not be applied when measurements are not being recorded. The voltage U_2 shall be determined by

$$U_2 = \frac{\left|U_2^+ - U_2^-\right|}{2} \tag{3}$$

where it should be noted that the sign of U_2^- is opposite that of U_2^+; i.e. Formula (3) indicates an average of the two numbers approximately equal in magnitude. A resistance R shall be determined from the voltage by

$$R = \frac{U_2}{I_2} \tag{4}$$

As the sample is warmed, values R shall be recorded as a function of the temperature T determined by the thermometer attached to the mandrel or base plate. Graphical aids and data analysis software are acceptable tools for plotting the resistance versus temperature curve and performing extrapolations.

A resistance versus temperature curve shall thus be obtained as in Figure 1. The resistance versus temperature curve shall be continuously recorded until a temperature of at least 15 K is reached. The resistance versus temperature curve shall be analysed by drawing a line through the region of steepest slope near the midpoint of the resistance rise, line (a) on Figure 1, and extrapolating this line sufficiently above the value of R recorded at 15 K. A second line shall be drawn through the region of the resistance versus temperature curve above the transition, line (b) in Figure 1, and this line shall be extrapolated to sufficiently lower temperature such that it intersects with line (a). The intersection is labelled as point A in Figure 1. The value of resistance R_2 corresponding to intersection point A shall be recorded, along with the value of temperature T_c^* corresponding to intersection point A.

7.4 Validation of the residual resistance measurement

The determination of R_2 shall be valid if all the following criteria are met.

Interfering voltages shall be such that

$$\frac{\left|U_0^+ - U_0^-\right|}{I_2 R_2} < 3\ \% \tag{5}$$

Thermal drift or scatter shall be such that, for consecutive values U_2^+ and U_2^- recorded with temperature near T_c^*,

$$\frac{\left|U_2^+ + U_2^-\right|}{I_2 R_2} < 3\ \% \tag{6}$$

The ambient temperature shall be such that

$$283\ \text{K} < T_1 < 303\ \text{K} \tag{7}$$

7.5 Residual resistance ratio

The RRR shall be calculated using Formula (1) and recorded.

8 Uncertainty of the test method

Based on the outcome of inter-laboratory comparison, discussed fully in Clause C.2, a typical uncertainty across laboratories of 0,3 % to 1,3 % has been obtained.

9 Test report

9.1 General

A test report shall be provided to summarize the findings of the RRR test procedure.

9.2 Test information

The following shall be included to record the test information:

a) date and time of the measurement;
b) operator name;
c) edition of IEC 61788-23 followed.

9.3 Specimen information

The following information pertinent to the specimen shall be included in the test report:

a) vendor's heat treatment, fabrication, or other tracking information such as a purchase order number;
b) sheet or piece identification number, if any;
c) specimen shape and orientation relative to the helium bath.

9.4 Test conditions

The following test conditions shall be included in the test report:

a) room temperature T_1;
b) transport currents I_1 and I_2;
c) voltages U_1 and U_2, noting that U_2 varies with temperature and therefore requires reporting as a table or graph;
d) resistances R_1 and R_2;
e) voltages U_0, U_0^+, U_0^- or validation, Formula (5);

The following additional information ~~may~~ can be included in the test report:

f) voltage tap distance L;
g) specimen dimensions and cross-sectional area A;
h) resistivity $\rho_1 = R_1 A L^{-1}$ and $\rho_2 = R_2 A L^{-1}$.

9.5 RRR value

The RRR value shall be quoted as $r_{RRR} \pm u_{RRR}$, for example 300 ± 19,2 ($k = 2$), where u_{RRR} is the combined standard uncertainty in accordance with Annex C. Alternatively, r_{RRR} ~~may~~ can be quoted as a minimum value, for example 285 minimum, to denote the lower limit of the confidence interval represented by the uncertainty. It is not necessary to report the uncertainty for a single measurement. Results should be expressed as three significant figures if not otherwise specified.

Additional information relating to the measurement of RRR is given in Annex A. Annex B describes definitions and an example of uncertainty in measurement. Uncertainty evaluation in the reference test method of RRR for ~~composite~~ Nb superconductors is given in Annex C.

Annex A
(informative)

Additional information relating to the measurement of RRR

A.1 Considerations for specimens and apparatus

The requirements in Clause 4 imply several general guidelines for preparing specimens and the configuration of the measurement apparatus.

a) Niobium sheet stock is typically 2 mm to 5 mm thick. This implies a typical cross-sectional area A of a sample of ~10 mm^2 = 0,1 cm^2 if a bar is machined with width approximately the same as the sheet thickness.

b) Voltage tap separation depends on the apparatus dimensions, but cannot be longer than about 80 % of the length of the niobium bar cut from sheets. To conserve liquid helium, this is about 10 cm maximum, so a voltage tap separation L of 2 cm to 5 cm is reasonable.

c) Given that the resistivity ρ of pure niobium at 293 K is approximately 15 μΩ·cm, a typical resistance of the niobium bar is $R = \rho L/A$ = 15 μΩ·cm × 5 cm / 0,1 cm^2 = 750 μΩ.

d) If r_{RRR} = 300, then a resistance of 750 / 300 = 2,5 μΩ can be expected at ~10 K. Thus, given the scope described in Clause 1, a resistance of ~~1,5~~ 1,3 μΩ to 5,0 μΩ is observed.

e) To produce a measurement signal of ~1 μV at 10 K, as required by Clause 4, a current of 1 μV / 2,5 μΩ = 0,4 A will be required. Thus, a target of 1 A measuring current should be used to provide ample allowance for variations in RRR among different specimens. This corresponds to a current density of approximately 0,1 A/mm^2.

f) As an alternative guideline, assuming a voltage of 1 μV at 10 K is produced by 1 A measuring current for r_{RRR} = 300, then L/A = 1 μΩ × 300 / 15 μΩ cm = 20 cm^{-1}. If the sample width w is the same as its 0,2 cm to 0,5 cm thickness, then the aspect ratio of the sample $L/w = (L/A) \times (A/w)$ = 20 cm^{-1} × (w^2/w) = 20 cm^{-1} × w is approximately 4 to 10. This justifies the requirement d) of Clause 4.

   With a measuring current of 1 A, a contact resistance of 1 mW / (1 A)^2 = 1 mΩ is likely to be achieved. This resistance is typical of that produced by contacts with ~1 mm^2 area. Examples are

   1) a clean set screw contacting clean Nb metal,
   2) a clean conductive spring clip contacting clean Nb metal,
   3) a conductive terminal clamp anchored by a screw or spring, or
   4) a tightly wound fine copper wire (diameter about 0,2 mm) that surrounds the contact region, with solder connection between current leads and wire.

   Polishing the contact area, or applying a soft metal such as indium, ~~may~~ can be used to reduce contact resistance. Contacts with small area, such as pin contacts or blade contacts, will probably not yield a suitable contact resistance.

g) To provide proper thermal sinking and thermal contact to thermometers, good thermal conductors should be used for the mandrel or base plate that supports the sample. Such materials include copper, silver, or aluminium. Mild alloys of these metals increase the mechanical strength without greatly reducing the thermal conductivity.

h) Niobium is susceptible to electron scattering by dislocations. The sample should not be bent in any way after being prepared in the shape to be tested. Special care should be taken during instrumentation and installation of the specimen on the base plate so that no excessive force, which can cause undesired bending strain or tensile strain, would be applied to the specimen. Ideally, it is intended that the specimen be as straight as possible; however, this is not always the case, thus care should be taken to measure the specimen in its as received condition.

A.2 Considerations for specimen mounting orientation

The orientation of the specimen relative to the cryostat is not specified. The considerations in Clause A.1 result in a specimen that can be much longer than the typical width of the orifice at the top of a measurement dewar. Long samples are therefore conveniently inserted through a small dewar orifice in a vertical orientation. However, thermal gradients along the specimen can also result in such cases. Horizontal orientation can reduce significantly any thermal gradients, but might also place undue constraints on the sample dimensions. U-shaped specimens both accommodate the ability to lie flat in a horizontal orientation as well as provide a sufficient length between voltage taps, as long as no bending strain has been introduced during the specimen preparation procedure.

A.3 Alternative methods for increasing temperature of specimen above superconducting transition temperature

A.3.1 General

The methods in A.3.2, A.3.3 a), A.3.3 b), and A.3.3 c) are also recommended for increasing temperature above the superconducting transition of the specimen. The rate of temperature increase of the whole specimen within a range between 0,1 K/min and 10 K/min should be applied for these methods. Heater power, heat capacity (of the specimen with the measuring mandrel or the measuring base plate) and the distance between the heater and the specimen should be selected appropriately to dampen the rate of temperature increase and avoid a large temperature gradient.

A.3.2 Heater method

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

A.3.3 Controlled methods

a) 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 is cooled to a temperature below the critical temperature. After the helium gas is pumped out, the specimen can be heated above the superconducting transition by the heater under adiabatic condition.

b) 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 measurement mandrel or the measurement base plate to the liquid helium bath allows the specimen to be cooled to a temperature below the critical temperature. The specimen can be heated above the superconducting transition by a heater located in the measurement mandrel or the measurement 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 measurement mandrel or a measurement base plate, to a temperature below the critical temperature. The specimen can be heated above the superconducting transition by a heater or by controlling the refrigerator power.

A.4 Other test methods

A.4.1 General

Documents [4], [5], [6], [8] have compared several methods for determining the residual resistivity ratio of niobium.

A.4.2 Measurement of resistance versus time

If R as a function of time with increasing temperature is recorded, then a resistance versus time curve is obtained, as in Figure A.1. The resistance versus time curve is continuously recorded both below and above the transition. The specimen should not be re-cooled without re-starting the acquisition of R versus t. The resistance versus time data are analysed by drawing a line through the region of steepest slope near the midpoint of the resistance rise, line (a) on Figure A.1, and extrapolating this line above the value of R recorded after the transition occurs, where t_c^* represents the time at which the transition is completed. A second line is drawn through the region of the resistance versus time data after the transition occurs, line (b) in Figure A.1, and this line is extrapolated to an earlier time such that it intersects with line (a). The intersection is labelled as point A in Figure A.1. The value of resistance R_2 corresponding to intersection point A is recorded.

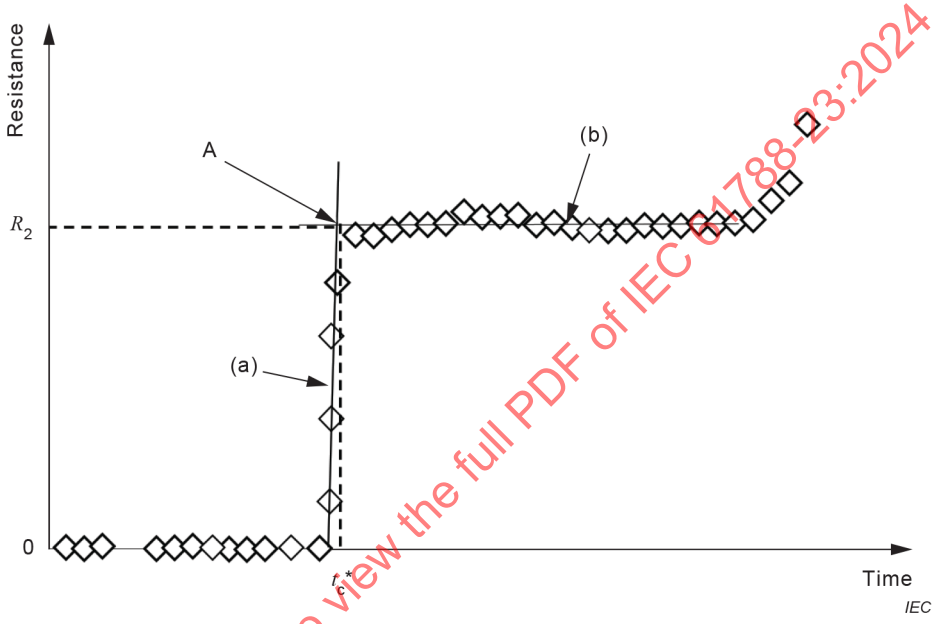

Figure A.1 – Determination of the value of R_2 from a resistance versus time plot

A.4.3 Comparison of ice point and room temperature

~~Standard specifications of niobium metal may specify that~~ The value of R_1 ~~be~~ is obtained at 273 K in [3]. According to Formula (2), the discrepancy between values of R_1 between 273 K and 293 K is 7,4 %. Actual measurements, drawn from the inter-laboratory comparison described in Clause C.2, reveal a slightly higher difference of 8,2 % ± 0,2 %.

A.4.4 Extrapolation of the resistance to 4,2 K

In [4], [5], [6] and [8], it was pointed out that the phonon contribution to niobium resistivity has a temperature dependence below 10 K. In this regime, the temperature dependence of resistance $R(T)$ can be fit with low uncertainty by a function of the form

$$R(T) = R_0 + aT^2 + bT^3 J_3(\theta_D/T) \quad \text{(A.1)}$$

where

R_0 is a residual resistance,

a and b are constants,

θ_D is the Debye temperature, and

$J_3(x)$ is the Grüneisen integral of the third kind.

In [4], linear plots of $R(T)$ versus T^3 were observed, suggesting that the coefficient a is small. A correction formula, to account for the difference between measurements of residual resistance at ~10 K and by extrapolation to 4,2 K, was derived in [5]. This formula is re-written here to give a relationship between RRR as determined by this document and a value determined by a ratio of R_1 to the resistance extrapolated to 4,2 K:

$$\frac{R_1}{R(4{,}2\ \mathrm{K})} = \frac{r_{\mathrm{RRR}}}{1 - 2{,}16 \times 10^{-4} r_{\mathrm{RRR}}} \tag{A.2}$$

For r_{RRR} = 300, Formula (A.2) will produce a resistance ratio of 321, resulting in a difference of 7 %.

A.4.5 Use of magnetic field to suppress superconductivity at 4,2 K

Superconductivity can be suppressed by the application of a magnetic field. For niobium, application of a field higher than 1 T will produce the normal state. In this case, a magneto-resistance appears, so a fit of the data shall be used to extrapolate to a value of R(4,2 K) at zero applied field. In [4] and [6], it was pointed out that fits generally obey Kohler's rule with linear dependence of R on an applied field H for field perpendicular to current. Curvature of $R(H)$ produced significant deviation from Kohler's rule for samples measured with current parallel to field. An acceptable uncertainty of 4 % was noted for field perpendicular to current, whereas uncertainty was 14 % for field parallel to current.

Agreement between the value of R(4,2 K) produced by extrapolation of magnetic field and that determined by extrapolation of temperature in A.4.4 was noted in [5]. Therefore, Formula (A.2) to estimate resistance at 4,2 K by extrapolation applies.

A.4.6 AC techniques

In [5], the use of a contactless method to apply an AC magnetic field and detect the signal according to induced current was described. However, this technique requires calibration by transfer of a reference sample measured either according to this document or to one of the techniques mentioned above. No direct relationship between the value of RRR obtained according to this document and the signal produced by the AC technique was described.

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 ISO/IEC Guide 98-3:2008 [9].

It was left to each technical committee to decide whether 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 IEC 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~~ can 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~~ is required to address for ~~compliance~~ conformance to IEC 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 IEC TC 90 measurement standards were designed to limit the uncertainty of any quantity that could influence the measurement, based on the IEC TC 90 experts' 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 inter-laboratory comparison.

B.2 Definitions

Statistical definitions can be found in three sources: ISO/IEC Guide 98-3:2008 [9], ISO/IEC Guide 99:2007 [10], and the NIST Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results (NIST) [11]. Not all statistical terms used in this document are explicitly defined in ISO/IEC Guide 98-3:2008. For example, the terms "relative standard uncertainty" and "relative combined standard uncertainty" are used in ISO/IEC Guide 98-3:2008, e.g. 5.1.6, Annex J, but are not formally defined in that document (see [11]).

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 (note that 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) 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 in Table B.1 shows a set of electronic drift and creep voltage measurements from two nominally identical extensometers using the 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.2, Table B.3, Table B.4 and Table B.5 show the mean values, experimental standard deviations, standard uncertainties and COV values of two output signals, respectively.

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

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

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

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

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

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

Table B.5 – Coefficients of variation of two output signals

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

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

The standard uncertainty is very similar for the two extensometer deflections. In contrast, the coefficient of variation (X_{COV}) is different by nearly a factor of 2 800 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 IEC TC 90 standards

The observed value of a measurement does not usually coincide with the true value of the measurand. The observed value ~~may~~ can 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. ISO/IEC Guide 98-3:2008, 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 ISO/IEC Guide 98-3:2008 procedure is illustrated in the following example.

a) Derive in the first step a mathematical measurement model in the form of an 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, F_{LC}:

$$F_{LC} = F_m + d_W + d_R + d_{Re}$$

where F_m, d_W, d_R, and d_{Re} represent the force expected due to an applied standard mass, the manufacturer's data, repeated checks of standard weight per day and the reproducibility of checks on different days, respectively.

Here the input quantities are the measured force 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) Identify the type of distribution for each input quantity (e.g. Gaussian distributions for Type A measurements and rectangular distributions for Type B measurements).

Evaluate the standard uncertainty of the Type A measurements, $u_A = s/\sqrt{n}$, where s is the experimental standard deviation and n is the total number of measured data points.

c) Evaluate the standard uncertainties of the Type B measurements:

$$u_B = \sqrt{\frac{1}{3}d_w^2 + \cdots}$$

where d_w is the range of rectangular distributed values.

d) 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 [12], [13].

e) 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.

f) 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 commercial software is a straightforward method that reduces the amount of routine work [14], [15]. In particular, the indicated partial derivatives can be easily obtained when such a software tool is used.

Annex C
(informative)

Uncertainty evaluation for resistance ratio measurement of Nb superconductors

C.1 Evaluation of uncertainty

C.1.1 Room temperature measurement uncertainty

The uncertainty of the test method arises from the terms in Formula (1), namely the uncertainty in room temperature resistance u_{R1} and the uncertainty in cryogenic resistance u_{R2}. For simplicity, the coverage factor k is kept equal to 1.

Since r_{RRR} is defined by Formula (1) as R_1/R_2, deviations Δr_{RRR} can be expressed by

$$\frac{\Delta r_{\mathrm{RRR}}}{r_{\mathrm{RRR}}} = \frac{\Delta R_1}{R_1} - \frac{\Delta R_2}{R_2} \tag{C.1}$$

The uncertainty u_{RRR} in the RRR measurement can then be expressed as

$$\frac{u_{\mathrm{RRR}}}{r_{\mathrm{RRR}}} = \left[\left(\frac{u_{R1}}{R_1} \right)^2 + \left(\frac{u_{R2}}{R_2} \right)^2 \right]^{0,5} \tag{C.2}$$

Formula (2) can be rearranged as

$$R_1 = \frac{U_1}{I_1}(2{,}084 - 0{,}003\,7\ T_1) \tag{C.3}$$

from which the deviations ΔR_1 can be expressed as

$$\Delta R_1 = (2{,}084 - 0{,}003\,7\ T_1)\left(\Delta U_1 \frac{1}{I_1} - \Delta I_1 \frac{U_1}{I_1^2} \right) - \Delta T_1 \frac{0{,}003\,7\ U_1}{I_1} \tag{C.4}$$

By expressing the deviations in Formula (C.4) as the uncertainties u_{U1}, u_{I1} and u_{T1}, respectively, the uncertainty of R_1 is

$$u_{R1} = \left[\left(\frac{C}{I_1} \right)^2 u_{U1}^2 + \left(\frac{CU_1}{I_1^2} \right)^2 u_{I1}^2 + \left(\frac{0{,}003\,7 U_1}{I_1} \right)^2 u_{T1}^2 \right]^{0,5} \quad \text{(C.5)}$$

where the constant C = (2,084 − 0,003 $7T_1$) lies between 0,97 and 1,03 when Formula (7) is valid.

C.1.2 Cryogenic measurement uncertainty

The cryogenic measurement relies on acquiring a number of data points R defined by Formula (4). For each measured data point R_i, deviations ΔU_2 and ΔI_2 exist. They produce an uncertainty u_{Ri} according to

$$u_{Ri} = \left[2 \left(\frac{u_{U2}}{I_2} \right)^2 + \left(\frac{U_2}{I_2^2} \right)^2 u_{I2}^2 \right]^{0,5} \quad \text{(C.6)}$$

where u_{U2} is the uncertainty in U_2 and u_{I2} is the uncertainty in I_2. Note that the factor 2 accounts for the averaging of values U_2^+ and U_2^- [1]. It is assumed that the uncertainties in Formula (C.6) are not dependent on temperature or time. Using conservative ratings for uncertainties that are often met by modern electronics, $u_{U2} = 0{,}002\,8\ U_2$ and $u_{I2} = 0{,}002\,8\ I_2$ (see Table C.1), it can be estimated that, under ideal conditions, $u_{Ri}/R_i = 0{,}5\%$. However, this assessment assumes that thermal gradients, thermal currents, and other factors do not contribute systematic variations in addition to the random uncertainty imposed by the measurement equipment. In some test configurations, observed uncertainty for each data point can be 5 %.

The measurement technique discussed in 7.3 requires the user of this document to separate the points R_i into two ensembles: one related to line (a) and one related to line (b). For either ensemble, the data points R_i will vary around the mean values $\hat{R}_i$ by an uncertainty u_{Ri}. When the temperature method of Figure 1 is used, each ensemble of R_i values approximates the linear function $\hat{R}_i(T) = \alpha T + \beta$, and linear regression analysis can be used to assess the uncertainties u_α and u_β. A parallel analysis applies to the method of Figure C.1.

The ~~uncertainty~~ standard deviations of regression for line (a) and line (b), denoted by s_a and s_b, respectively, can be expressed as

$$s_{a,b} = \sqrt{\frac{\sum_{a,b} \left(R_i - \hat{R}_i \right)^2}{N_{a,b} - 2}} \quad \text{(C.7)}$$

where the subscripts a and b denote the ensemble of points used for line (a) or line (b), respectively. The denominator indicates that two degrees of freedom have been removed from the number, N_{a} or N_{b}, of data points in ensemble (a) or ensemble (b), respectively, due to the conditions that the ensemble of data points represents the true population, and that the set of data points is representative of the true data. Formula (C.7) thus requires the recording of at least three data points for each line to keep the denominator positive.

For the temperature method, the uncertainty of slope for lines (a) and (b), denoted as u_{α}^{a} and u_{α}^{b}, respectively, depends on the ensemble of measured temperature T_i relative to the average temperatures $\overline{T}_{\mathrm{a}}$ and $\overline{T}_{\mathrm{b}}$ of ensembles (a) and (b), respectively, via

$$u_{\alpha}^{\mathrm{a,b}} = \frac{s_{\mathrm{a,b}}}{\sqrt{\sum_{\mathrm{a,b}}\left(T_i - \overline{T}_{\mathrm{a,b}}\right)^2}} \tag{C.8}$$

Here, the subscripts again denote different ensembles of data points associated with either line (a) or line (b). Formula (C.8) indicates that measurements over a broader range of temperature will reduce the uncertainty in the slope, provided that a linear regression holds for the full temperature range. A similar expression can be derived for recording measurements as a function of time.

The uncertainty of intercept at T_{c}^{*} for either line can be expressed as

$$u_{\beta}^{\mathrm{a,b}} = s_{\mathrm{a,b}}\sqrt{\frac{\sum_{\mathrm{a,b}}\left(T_i - T_{\mathrm{c}}^{*}\right)^2}{N_{\mathrm{a,b}}\sum_{\mathrm{a,b}}\left(T_i - \overline{T}_{\mathrm{a,b}}\right)^2}} \tag{C.9}$$

Since the uncertainty in Formula (C.9) lies along the vertical line used to determine T_{c}^{*}, it is important to add a correction ~~needs to be added~~ to account for the extrapolation of line (b) onto line (a) to arrive at the uncertainty u_{R2} in the value of R_2. This can be approximated by

$$u_{R2} = \left[\left(\alpha_{\mathrm{b}} u_{\beta}^{\mathrm{a}} / \alpha_{\mathrm{a}}\right)^2 + \left(u_{\beta}^{\mathrm{b}}\right)^2\right]^{0,5} \tag{C.10}$$

A graphical representation of the uncertainty is shown in Figure C.1. Note that any contributions of thermoelectric voltage are not considered in this uncertainty analysis.

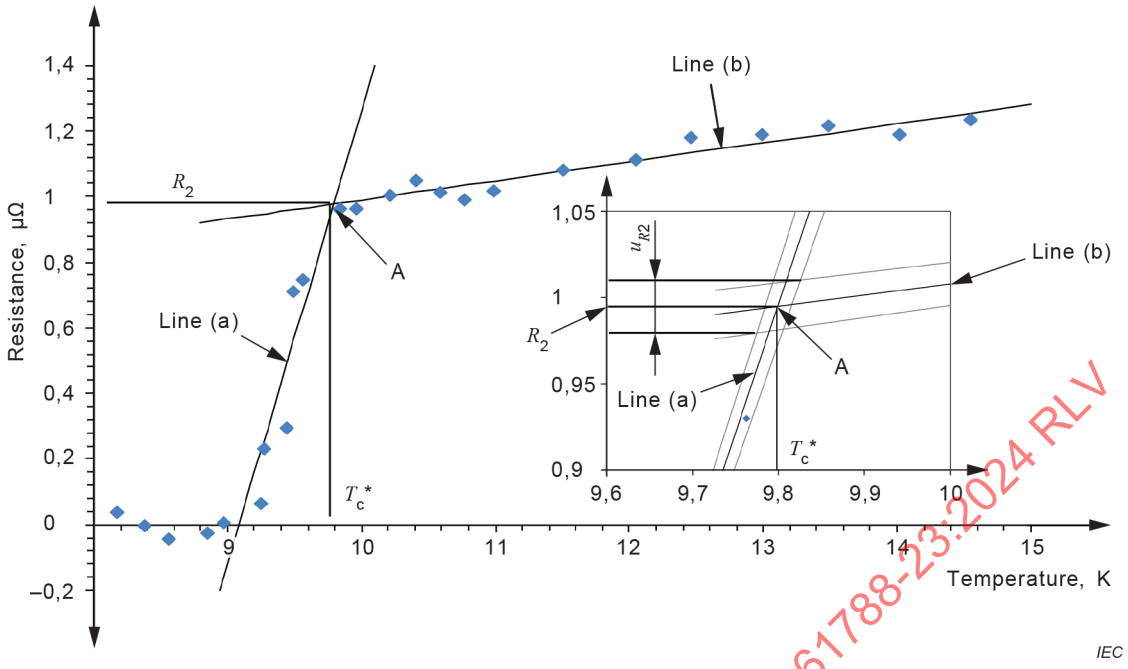

Figure C.1 – Graphical description of the uncertainty of regression related to the measurement of R_2

C.1.3 Estimation of uncertainty for typical experimental conditions

The cryogenic resistance uncertainty is estimated here, assuming that cryogenic measurements acquired five data points for line (a) and 10 data points for line (b). The data ensemble (a) is assumed to be acquired in 0,2 K intervals with $\bar{T}_a$ = 9,5 K, and ensemble (b) is assumed to be acquired in 0,5 K intervals with $\bar{T}_b$ = 11,0 K, with values $R_i \geq 1{,}0$ μΩ above the transition and T_c^* = 9,8 K. The slope of line (a) is 2 μΩ K^{-1}, and the slope of line (b) is 0,05 μΩ K^{-1}. For this estimation, an uncertainty u_{Ri} = 0,05 ~~μV~~ μΩ is assumed for each data point. From Formula (C.7), $s_a \approx 0{,}065$ μΩ, and $s_b \approx 0{,}055$ μΩ. From Formula (C.8), a careful fit for line (a) would have an uncertainty of slope of 0,21 μΩ K^{-1}, while the fit for line (b) would have an uncertainty of slope of 0,010 μΩ K^{-1}. The intercepts of lines (a) and (b) with a hypothetical vertical line at T_c^* would have an uncertainty from Formula (C.9) of 0,069 μΩ and 0,032 μΩ, respectively. The combined uncertainty u_{R2} from Formula (C.10) is then 0,032 μΩ. Note that the uncertainty u_{R2} scales approximately as $u_{Ri}(2/N_b)^{0,5}$ since point A lies at the end of line (b).

Table C.1 – Uncertainty of measured parameters

Uncertainty	Type	Value	Remarks
u_{U1} / U_1	B	0,28 %	Instrument uncertainty of 0,5 %
u_{I1} / I_1	B	0,28 %	Instrument uncertainty of 0,5 %
u_{T1}	B	0,58 K	Thermometer uncertainty of 1,0 K
u_{U2} / U_2	B	0,28 %	Instrument uncertainty of 0,5 %
u_{I2} / I_2	B	0,28 %	Instrument uncertainty of 0,5 %

These additional parameters apply for the room temperature uncertainty estimation: T_1 = 300 K, U_1 = 3,00 μV, I_1 = 10,0 mA. These parameters give r_{RRR} = 292 for the value of R_2 above. In view of the data in Table C.1, u_{U1} = 0,002 8 × U_1 = 8,40 nV, u_{I1} = 0,002 8 × I_1 = 28,0 μA, and u_{T1} = 0,58 K. From Formula (C.5), u_{R1} = 1,32 μΩ.

From Formula (C.2), it can be estimated that u_{RRR} = 9,43. That is, r_{RRR} = 292 ± 9,43 for this example. This uncertainty is dominated by the uncertainty u_{R2}.

As summarized in Clause C.2, inter-laboratory comparison results gave a typical uncertainty much lower than the value (9,43/292) = 3,2 % assessed above. High-quality equipment, careful design of apparatus, and well-controlled test conditions ~~may~~ can have resulted in lower values of u_{R2} compared with the assumed uncertainty of 0,05 ~~μV~~ μΩ. Higher measuring current I_2 and acquisition of more data points than assumed for N_b above ~~may~~ can also contribute to reduced uncertainty according to Formula (C.6) and Formula (C.9).

C.2 Inter-laboratory comparison summary

Four international testing laboratories participated in an inter-laboratory comparison of 10 niobium specimens. All specimens were prepared by the vendor in a manner consistent with this document. Test techniques included both that defined by this document as well as those described by A.4.1, A.4.2 and A.4.3, all of which are methods that use liquid helium. Table C.2 summarizes the results of the inter-laboratory comparison. A typical uncertainty across laboratories of 0,3 % to 1,3 % has thus been obtained.

Table C.2 – RRR values obtained by inter-laboratory comparison using liquid helium

Sample	RRR by International Standard					
	Laboratory 1	Laboratory 2	Laboratory 3	Laboratory 4	Average	~~$\sigma N^{-0,5}$~~ $sN^{-0,5}$ (% of average)
1	367	370	364		367	0,4
2	347	348	348		348	< 0,1
3	391	390	386		389	0,3
4	379	378	375		377	0,3
5	417	418	418	417	418	< 0,1
6	408	395	417		407	1,3
7	382	377	381		380	0,3
8	383	370	378		380	0,3
9	344	328	341		338	1,2
10	332	332	333	336	333	0,3

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IEC 61788-23

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INTERNATIONAL STANDARD

NORME INTERNATIONALE

colour inside

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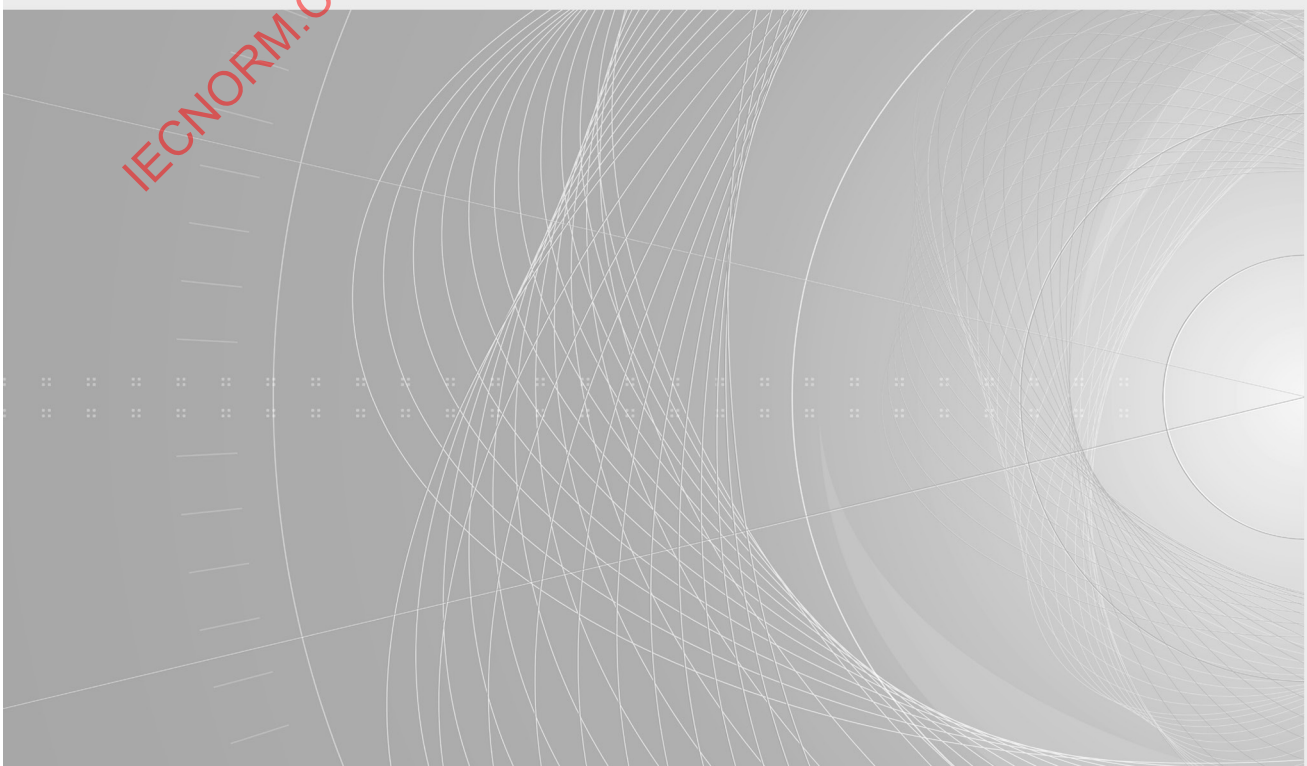

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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SUPERCONDUCTIVITY –

Part 23: Residual resistance ratio measurement – Residual resistance ratio of cavity-grade Nb superconductors

FOREWORD

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IEC 61788-23 has been prepared by IEC technical committee 90: Superconductivity. It is an International Standard.

This third edition cancels and replaces the second edition published in 2021. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to the previous edition:

a) The principle is changed to represent the present test method.

The text of this International Standard is based on the following documents:

Draft	Report on voting
90/515/FDIS	90/519/RVD

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this International Standard is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/publications.

A list of all parts in the IEC 61788 series, published under the general title *Superconductivity,* can be found on the IEC website.

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under webstore.iec.ch in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn, or
- revised.

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INTRODUCTION

High-purity niobium is the chief material used to make superconducting radio-frequency cavities. Similar grades of niobium can be used in the manufacture of superconducting wire. Procurement of raw materials and quality assurance of delivered products often use the residual resistance ratio (RRR) to specify or assess the purity of a metal. RRR is defined for non-superconducting metals as the ratio of electrical resistance measured at room temperature (293 K) to the resistance measured for the same specimen at low temperature (~4,2 K). The low-temperature value is often called the residual resistance. Higher purity is associated with higher values of RRR.

Niobium presents special problems due to its transformation to a superconducting state at ~9 K, so DC electrical resistance is effectively zero below this temperature. The definition above would then yield an infinite value for RRR. This document describes a test method to determine the residual resistance value by using a plot of the resistance to temperature as the test specimen is gradually warmed through the superconducting transition in the absence of an applied magnetic field. This results in a determination of the residual resistance at just above superconducting transition, ~10 K, from which RRR is subsequently determined.

International Standards also exist to determine the RRR of superconducting wires. In contrast to superconducting wires, which are usually a composite of a superconducting material and a non-superconducting material and the RRR value is representative of only the non-superconducting component, here the entire specimen is composed of superconducting niobium. Frequently, niobium is procured as a sheet, bar or rod, and not as a wire. For such forms, test specimens will likely be a few millimetres in the dimensions transverse to electric current flow. This difference is significant when making electrical resistance measurements, since niobium samples will likely be much longer than that for the same length-to-diameter ratio as a wire, and higher electrical current can be required to produce sufficient voltage signals. Guidance for sample dimensions and electrical connections is provided in Annex A. Test apparatus should also take into consideration aspects such as the orientation of a test specimen relative to the liquid helium surface, accessibility through ports on common liquid helium dewars, design of current contacts, and minimization of thermal gradients over long specimen lengths. These aspects distinguish this document from similar wire standards.

Other test methods have been used to determine RRR. Some methods use a measurement at a temperature other than 293 K for the high resistance value. Some methods use extrapolations at 4,2 K in the absence of an applied magnetic field for the low resistance value. Other methods use an applied magnetic field to suppress superconductivity at 4,2 K. A comparison between this document and some other test methods is presented in Annex A. Note that systematic differences of up to 10 % are produced by these other methods, which is larger than the target uncertainty of this document. It is therefore important to apply this document or the appropriate corrections listed in Annex A according to the test method used.

Whenever possible, this test method should be transferred to vendors and collaborators who also perform RRR measurements. To promote consistency, the results of inter-laboratory comparisons are described in Clause C.2.

SUPERCONDUCTIVITY –

Part 23: Residual resistance ratio measurement – Residual resistance ratio of cavity-grade Nb superconductors

1 Scope

This part of IEC 61788 addresses a test method for the determination of the residual resistance ratio (RRR), r_{RRR}, of cavity-grade niobium. This method is intended for high-purity niobium grades with 150 < r_{RRR} < 600. The test method is valid for specimens with rectangular or round cross-section, cross-sectional area greater than 1 mm^2 but less than 20 mm^2, and a length not less than 10 nor more than 25 times the width or diameter.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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* (available at: https://www.electropedia.org/)

3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- IEC Electropedia: available at https://www.electropedia.org/
- ISO Online browsing platform: available at https://www.iso.org/obp

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

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

where

R_1 is the resistance at room temperature, 293 K;

R_2 is the resistance just above the superconducting transition, at ~10 K.

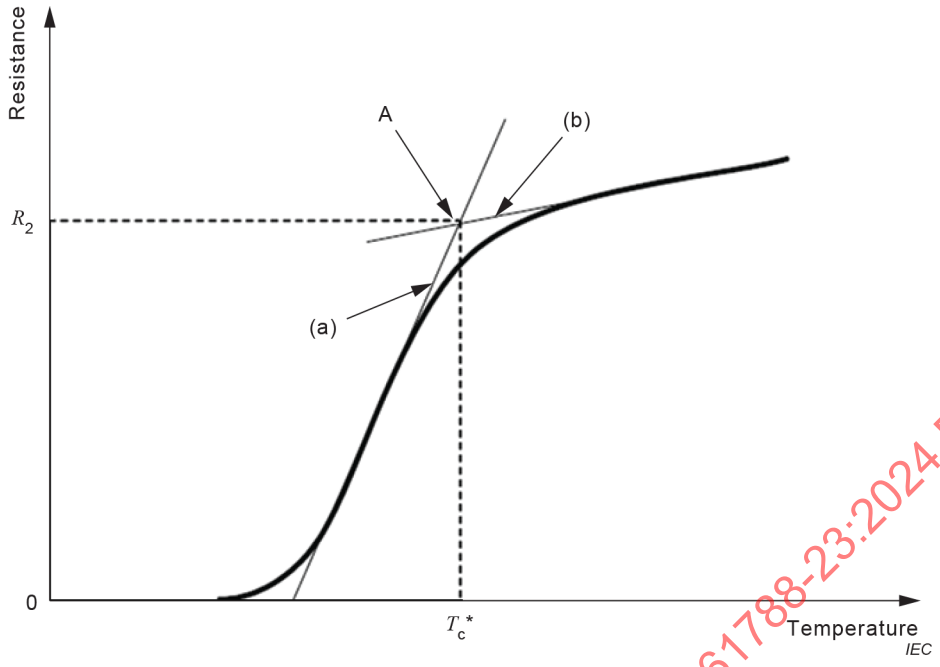

Figure 1 – Relationship between temperature and resistance near the superconducting transition

Note 1 to entry: In this document, the room temperature is defined as 20 °C = 293 K, and r_{RRR} is obtained as follows: Figure 1 shows schematically resistance versus temperature data and the graphical procedure used to determine the value of R_2. In Figure 1, the region of maximum slope is extrapolated upward in resistance, as shown by line (a), and the region of minimum slope at temperatures above the transition temperature is extrapolated downward in temperature, as shown by line (b). The intersection of these extrapolations at point A determines the value of R_2 as well as a temperature value T_c^*.

Note 2 to entry: The value T_c^* is similar to the transition value defined in [1], and is different from the value defined at the midpoint of the transition, called T_c^* in [2].

Note 3 to entry: Some standards or documented techniques, e.g. [3], [4], [5], [6], define r_{RRR} with the value of R_1 determined at a temperature other than 293 K, or the value of R_2 determined at a temperature below the superconducting transition. The user's attention is drawn to such differences in definition.

4 Principle

The 4-point DC electrical resistance technique shall be performed both at room temperature and at cryogenic temperature. The test of resistance shall be done as a function of temperature. Another test method of resistance as a function of time with increasing temperature is described in A.4.2.

The relative combined standard uncertainty of this method is 3 % with coverage factor 2.

Measurements shall have the following attributes.

b) Measuring current is sufficiently high to provide voltage signals of the order of 1 μV. For electrical safety, maximum current density should never exceed 1 A mm^{-2}.

c) Contact resistance for current leads is sufficiently low to avoid excessive heating of the sample. Typical cryogenic measurement conditions require power dissipation at contacts to be less than 1 mW.

d) Sample sizes are sufficiently large to minimize the probability of cutting and handling damage. Typical samples are 1 mm to 3 mm in cross-sectional dimension and greater than 5 mm^2 in cross-sectional area.

e) Sample length is at least 10 times and not more than 25 times the width or diameter.

Annex A discusses considerations for sample dimensions and measuring current.

5 Measurement apparatus

5.1 Mandrel or base plate

A straight mandrel or base plate shall be used to support the specimen. Possible materials of construction include pure copper, pure aluminium, pure silver, electrical grades of Cu-Zr, Cu-Cr-Zr, Cu-Be, and other copper alloys, electrical grades of Al-Mg, Al-Ag, and other aluminium alloys, and electrical grades of silver alloys. These provide high thermal conductivity and serve to remove thermal gradients during measurement. The specimen shall be insulated from the mandrel. Possible insulating materials include polyethylene terephthalate, polyester, and polytetrafluoroethylene, which can be applied as foils, tapes, or coatings. Glass-fibre reinforced epoxy or other composite materials with good thermal conductivity at cryogenic temperature can also be used.

The base plate should have a clean and smooth surface finish. There should be no burrs, ridges, seams, or other asperities that can affect the specimen. High-purity niobium specimens are soft and are susceptible to indentation by surface flaws, and such indentations can alter the sample and invalidate the resistance measurement.

The mandrel or base plate shall support the entire length and width of the specimen. Mandrel or base plate geometry should not impose a bending strain of more than 0,2 % on the sample.

A thermometer accurate to 0,1 K is helpful but not required. The mandrel or base plate can incorporate a mounting for a cryogenic thermometer directly against the body of the mandrel or base plate and near the centre of the test specimen.

Practical base plates are at least 30 mm in length to accommodate assembly of pieces and handling of samples by human hands. Multiple samples can be mounted against a single base plate.

5.2 Cryostat and support of mandrel or base plate

The apparatus shall make provisions for mechanical support of the mandrel or base plate. In addition, such support shall provide electrical leads to carry currents for samples and thermometers, and measure their voltages. For R_1 and R_2 measurements, the support shall permit current to flow through only the sample, so that the entire resulting voltage measured is only that generated by the sample.

The support structure shall permit measurement of both R_1 and R_2 without dismounting or remounting the test specimen. Measurement of R_2 shall require the use of a cryostat, which shall, moreover, integrate with the support.

The cryostat shall include a liquid helium reservoir at the bottom of a substantial vertical column. A support structure shall accommodate the raising and lowering of the sample into or out of the helium bath. In addition, anchoring of the sample position, either when immersed in liquid helium or suspended above the surface of the liquid at an arbitrary height, shall be provided. Such suspension permits the equilibration of temperature during measurement and slow increase of temperature with height above the helium bath. Alternatively, immersion of the sample into the bath followed by reduction of the bath level via boil-off or pressurized transfer can also be used to vary temperature.

A heater can be employed to warm the mandrel or base plate. The heater should be distributed along the mandrel and excessive power settings should be avoided. For instance, a point source of 1 W heat input operating at the centre of a 1 cm^2 mandrel upon which a 5 cm sample is mounted could produce temperature difference of 2,5 K along the sample if the thermal conductivity is 100 W m^{-1} K^{-1}.

Proper cryogenic techniques shall be followed for the construction of the cryostat and apparatus. This includes the use of low thermal conductivity materials such as thin-walled stainless steel tubes, composite materials, ceramics, and insulation, to prevent excessive boil-off due to heat conduction from the surroundings. A can or shield can surround the base plate or mandrel with mounted sample to improve thermal stability. Provisions for pressure relief and vacuum isolation of the liquid helium should be incorporated with the apparatus.

6 Specimen preparation

High-purity niobium is quite malleable, and even the slightest force can produce deformation of the material. Since dislocations are one source of electron scattering, specimen deformation can inadvertently contribute to the residual resistivity and affect the test result. Therefore, special protocols shall be observed when preparing the specimen. Cutting techniques shall avoid heat and strain to the extent possible. Discharge machining, fluid-jet cutting, or low-speed conventional machining are acceptable and widely-used techniques for applications using high-purity niobium. Specimens cut from larger pieces shall be protected and immobilized against a support piece during transport. Operations to de-burr samples shall not bend, excessively heat or otherwise damage the sample. Light sanding with fine paper is one acceptable approach.

Specimens should be rectangular or circular bars with uniform cross-section. Long sides of the specimen shall be parallel. Any twisting or curvature shall be avoided to ensure that bending or torsion is not applied to the test specimen during mounting to the mandrel or base plate. Specimens that form an arc or a U shape are acceptable provided that the entire curvature can be supported on a plane, without applying torsion to the bent specimen.

The specimen shall be clean and have no trace of residues from cutting fluids or any other surface contaminants. Degreasing with solvents, followed by ultrasonic cleaning using a mild water-based detergent, followed by rinsing with distilled or ultra-pure water, then drying in air, is preferred for cleaning residues. Chemical etching to clean the surface poses a risk of introducing contaminants, especially hydrogen and oxygen, and should be avoided. Gentle mechanical polishing on the regions where voltage taps and current leads attach is usually sufficient to remove surface oxides. Coating these regions with indium foil or another metal, for example by evaporation or sputtering, is an acceptable method to protect polished contacts provided that coating the entire specimen is avoided.

The test specimen shall be a single piece and shall not include any joints or splices.

A mechanical method shall be used to affix the test specimen to the mandrel or base plate. Installation and instrumentation of the specimen shall not apply excessive force, bending strain, tensile strain, or torsion to the specimen.

The test specimen shall be instrumented with current contacts near each end of the specimen and a pair of voltage contacts over the central portion between the current contacts (i.e. a 4-point measurement technique). The voltage contacts shall be separated from the current contacts by a distance no smaller than the largest dimension (width, thickness, or diameter) perpendicular to the specimen length.

7 Data acquisition and analysis

7.1 Data acquisition hardware

Modern power supplies can be computer controlled and come with a variety of features that permit remote control of the current output. Use of such power supplies is not required but could greatly enable automation of the data acquisition. Pulsed modes permit application of current only when voltage signals are being acquired, thereby removing heat generated in the sample during the off cycle. If pulsed current application is used, the pulse duration shall include ample periods for voltage signals to settle and be filtered.

Some power supplies incorporate an internal shunt to regulate the output current. If such a power supply is used, the internal shunt shall be calibrated periodically with an external shunt and voltage measurement.

The test set-up can establish an arbitrary baseline voltage U_0, which might be detectable when the sample is in the superconducting state and the power supply is off. The value U_0 can drift over time due to changes in the thermal environment and other effects. More complex hardware includes compensation for drift and automatic nulling such that the time average of U_0 is 0. Digital voltage meters are not required but greatly enhance the data acquisition. Besides compensation for drift and voltage nulling, filtering and internal compensation for thermally-induced voltages can improve the accuracy of the voltage measurement. Filtering should average voltage signals for a time at least as long as the thermal time constant of the apparatus at low temperature, typically of the order of 0,1 s to 10 s. It is important to understand how voltages are corrected for drift and thermal effects. Sensitive voltage meters, especially nanovolt meters, require a pre-amplifier that shall be at thermal equilibrium, which can require several hours of operation in advance of the measurement.

Data acquisition via computer greatly facilitates the recording and reporting of data.

7.2 Resistance (R_1) at room temperature

The ambient temperature T_1 of the measurement laboratory shall be measured. A specimen current I_1 shall be applied in accordance with the requirements in Clause 4. The resulting voltage U_1 shall be recorded together with I_1 and T_1. The resistance shall be determined by

$$R_1 = \frac{U_1}{I_1}\left[1 - 0{,}003\,7\ \left(T_1 - 293\right)\right] \tag{2}$$

with T_1 in units of kelvin. The coefficient 0,003 7 reflects the experimentally observed rate of change of resistance with temperature given in [7] [1] over the interval 273 K to 300 K.

1 Numbers in square brackets refer to the Bibliography.

7.3 Residual resistance (R_2) just above the superconducting transition

The measurement of R_2 shall be made with the sample still mounted on the mandrel or base plate for the measurement of R_1.

The specimen shall be placed in a cryostat as specified in 5.2. The specimen shall be slowly lowered into a liquid helium bath and cooled to liquid helium temperature. While a vigorous boil-off of liquid helium will accompany the initial cool down, removal of heat from the mandrel, especially if it is shielded, can require a time period of more than 5 min. Current can be applied, and voltage can be monitored during this period, but no measurement shall be made until the vigorous boil-off of liquid helium has subsided.

After the boil-off rate is suitable for measurements, a voltage measurement U_0 shall be recorded while the sample is immersed in liquid helium. The sample is likely to be in the superconducting state under these conditions. Current I_2 shall then be applied in accordance with requirements of Clause 4 and with considerations of Clause 5. Voltage readings U_0^+ and U_0^- shall be acquired for forward and reverse current polarity, respectively. Any differences between U_0, U_0^+, and U_0^- shall be recorded.

The specimen shall then be gradually warmed so that a transition from the superconducting state into the normal state occurs gradually. An apparatus that conforms to Clause 5 will permit gradual warming of the specimen by raising the level of the mandrel above the level of the liquid helium bath, for example. Two voltages U_2^+ and U_2^- shall be measured almost simultaneously with the application of the same measuring current I_2 with forward and reverse polarity, respectively. The current shall not be applied when measurements are not being recorded. The voltage U_2 shall be determined by

$$U_2 = \frac{\left|U_2^+ - U_2^-\right|}{2} \tag{3}$$

where it should be noted that the sign of U_2^- is opposite that of U_2^+; i.e. Formula (3) indicates an average of the two numbers approximately equal in magnitude. A resistance R shall be determined from the voltage by

$$R = \frac{U_2}{I_2} \tag{4}$$

As the sample is warmed, values R shall be recorded as a function of the temperature T determined by the thermometer attached to the mandrel or base plate. Graphical aids and data analysis software are acceptable tools for plotting the resistance versus temperature curve and performing extrapolations.

A resistance versus temperature curve shall thus be obtained as in Figure 1. The resistance versus temperature curve shall be continuously recorded until a temperature of at least 15 K is reached. The resistance versus temperature curve shall be analysed by drawing a line through the region of steepest slope near the midpoint of the resistance rise, line (a) on Figure 1, and extrapolating this line sufficiently above the value of R recorded at 15 K. A second line shall be drawn through the region of the resistance versus temperature curve above the transition, line (b) in Figure 1, and this line shall be extrapolated to sufficiently lower temperature such that it intersects with line (a). The intersection is labelled as point A in Figure 1. The value of resistance R_2 corresponding to intersection point A shall be recorded, along with the value of temperature T_c^* corresponding to intersection point A.

7.4 Validation of the residual resistance measurement

The determination of R_2 shall be valid if all the following criteria are met.

Interfering voltages shall be such that

$$\frac{\left|U_0^+ - U_0^-\right|}{I_2 R_2} < 3\ \% \tag{5}$$

Thermal drift or scatter shall be such that, for consecutive values U_2^+ and U_2^- recorded with temperature near T_c^*,

$$\frac{\left|U_2^+ + U_2^-\right|}{I_2 R_2} < 3\ \% \tag{6}$$

The ambient temperature shall be such that

$$283\ \text{K} < T_1 < 303\ \text{K} \tag{7}$$

7.5 Residual resistance ratio

The RRR shall be calculated using Formula (1) and recorded.

8 Uncertainty of the test method

Based on the outcome of inter-laboratory comparison, discussed fully in Clause C.2, a typical uncertainty across laboratories of 0,3 % to 1,3 % has been obtained.

9 Test report

9.1 General

A test report shall be provided to summarize the findings of the RRR test procedure.

9.2 Test information

The following shall be included to record the test information:

a) date and time of the measurement;
b) operator name;
c) edition of IEC 61788-23 followed.

9.3 Specimen information

The following information pertinent to the specimen shall be included in the test report:

a) vendor's heat treatment, fabrication, or other tracking information such as a purchase order number;
b) sheet or piece identification number, if any;
c) specimen shape and orientation relative to the helium bath.

9.4 Test conditions

The following test conditions shall be included in the test report:

a) room temperature T_1;
b) transport currents I_1 and I_2;
c) voltages U_1 and U_2, noting that U_2 varies with temperature and therefore requires reporting as a table or graph;
d) resistances R_1 and R_2;
e) voltages U_0, U_0^+, U_0^- or validation, Formula (5);

The following additional information can be included in the test report:

f) voltage tap distance L;
g) specimen dimensions and cross-sectional area A;
h) resistivity $\rho_1 = R_1 A L^{-1}$ and $\rho_2 = R_2 A L^{-1}$.

9.5 RRR value

The RRR value shall be quoted as $r_{\mathrm{RRR}} \pm u_{\mathrm{RRR}}$, for example 300 ± 19,2 ($k = 2$), where u_{RRR} is the combined standard uncertainty in accordance with Annex C. Alternatively, r_{RRR} can be quoted as a minimum value, for example 285 minimum, to denote the lower limit of the confidence interval represented by the uncertainty. It is not necessary to report the uncertainty for a single measurement. Results should be expressed as three significant figures if not otherwise specified.

Additional information relating to the measurement of RRR is given in Annex A. Annex B describes definitions and an example of uncertainty in measurement. Uncertainty evaluation in the reference test method of RRR for Nb superconductors is given in Annex C.

Annex A
(informative)

Additional information relating to the measurement of RRR

A.1 Considerations for specimens and apparatus

The requirements in Clause 4 imply several general guidelines for preparing specimens and the configuration of the measurement apparatus.

a) Niobium sheet stock is typically 2 mm to 5 mm thick. This implies a typical cross-sectional area A of a sample of ~10 mm^2 = 0,1 cm^2 if a bar is machined with width approximately the same as the sheet thickness.

b) Voltage tap separation depends on the apparatus dimensions, but cannot be longer than about 80 % of the length of the niobium bar cut from sheets. To conserve liquid helium, this is about 10 cm maximum, so a voltage tap separation L of 2 cm to 5 cm is reasonable.

c) Given that the resistivity ρ of pure niobium at 293 K is approximately 15 μΩ·cm, a typical resistance of the niobium bar is $R = \rho L/A$ = 15 μΩ·cm × 5 cm / 0,1 cm^2 = 750 μΩ.

d) If r_{RRR} = 300, then a resistance of 750 / 300 = 2,5 μΩ can be expected at ~10 K. Thus, given the scope described in Clause 1, a resistance of 1,3 μΩ to 5,0 μΩ is observed.

e) To produce a measurement signal of ~1 μV at 10 K, as required by Clause 4, a current of 1 μV / 2,5 μΩ = 0,4 A will be required. Thus, a target of 1 A measuring current should be used to provide ample allowance for variations in RRR among different specimens. This corresponds to a current density of approximately 0,1 A/mm^2.

f) As an alternative guideline, assuming a voltage of 1 μV at 10 K is produced by 1 A measuring current for r_{RRR} = 300, then L/A = 1 μΩ × 300 / 15 μΩ cm = 20 cm^{-1}. If the sample width w is the same as its 0,2 cm to 0,5 cm thickness, then the aspect ratio of the sample $L/w = (L/A) \times (A/w)$ = 20 cm^{-1} × (w^2/w) = 20 cm^{-1} × w is approximately 4 to 10. This justifies the requirement d) of Clause 4.

   With a measuring current of 1 A, a contact resistance of 1 mW / (1 A)^2 = 1 mΩ is likely to be achieved. This resistance is typical of that produced by contacts with ~1 mm^2 area. Examples are

   1) a clean set screw contacting clean Nb metal,
   2) a clean conductive spring clip contacting clean Nb metal,
   3) a conductive terminal clamp anchored by a screw or spring, or
   4) a tightly wound fine copper wire (diameter about 0,2 mm) that surrounds the contact region, with solder connection between current leads and wire.

   Polishing the contact area, or applying a soft metal such as indium, can be used to reduce contact resistance. Contacts with small area, such as pin contacts or blade contacts, will probably not yield a suitable contact resistance.

g) To provide proper thermal sinking and thermal contact to thermometers, good thermal conductors should be used for the mandrel or base plate that supports the sample. Such materials include copper, silver, or aluminium. Mild alloys of these metals increase the mechanical strength without greatly reducing the thermal conductivity.

h) Niobium is susceptible to electron scattering by dislocations. The sample should not be bent in any way after being prepared in the shape to be tested. Special care should be taken during instrumentation and installation of the specimen on the base plate so that no excessive force, which can cause undesired bending strain or tensile strain, would be applied to the specimen. Ideally, it is intended that the specimen be as straight as possible; however, this is not always the case, thus care should be taken to measure the specimen in its as received condition.

A.2 Considerations for specimen mounting orientation

The orientation of the specimen relative to the cryostat is not specified. The considerations in Clause A.1 result in a specimen that can be much longer than the typical width of the orifice at the top of a measurement dewar. Long samples are therefore conveniently inserted through a small dewar orifice in a vertical orientation. However, thermal gradients along the specimen can also result in such cases. Horizontal orientation can reduce significantly any thermal gradients, but might also place undue constraints on the sample dimensions. U-shaped specimens both accommodate the ability to lie flat in a horizontal orientation as well as provide a sufficient length between voltage taps, as long as no bending strain has been introduced during the specimen preparation procedure.

A.3 Alternative methods for increasing temperature of specimen above superconducting transition temperature

A.3.1 General

The methods in A.3.2, A.3.3 a), A.3.3 b), and A.3.3 c) are also recommended for increasing temperature above the superconducting transition of the specimen. The rate of temperature increase of the whole specimen within a range between 0,1 K/min and 10 K/min should be applied for these methods. Heater power, heat capacity (of the specimen with the measuring mandrel or the measuring base plate) and the distance between the heater and the specimen should be selected appropriately to dampen the rate of temperature increase and avoid a large temperature gradient.

A.3.2 Heater method

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

A.3.3 Controlled methods

a) 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 is cooled to a temperature below the critical temperature. After the helium gas is pumped out, the specimen can be heated above the superconducting transition by the heater under adiabatic condition.

b) 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 measurement mandrel or the measurement base plate to the liquid helium bath allows the specimen to be cooled to a temperature below the critical temperature. The specimen can be heated above the superconducting transition by a heater located in the measurement mandrel or the measurement 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 measurement mandrel or a measurement base plate, to a temperature below the critical temperature. The specimen can be heated above the superconducting transition by a heater or by controlling the refrigerator power.

A.4 Other test methods

A.4.1 General

Documents [4], [5], [6], [8] have compared several methods for determining the residual resistivity ratio of niobium.

A.4.2 Measurement of resistance versus time

If R as a function of time with increasing temperature is recorded, then a resistance versus time curve is obtained, as in Figure A.1. The resistance versus time curve is continuously recorded both below and above the transition. The specimen should not be re-cooled without re-starting the acquisition of R versus t. The resistance versus time data are analysed by drawing a line through the region of steepest slope near the midpoint of the resistance rise, line (a) on Figure A.1, and extrapolating this line above the value of R recorded after the transition occurs, where t_c^* represents the time at which the transition is completed. A second line is drawn through the region of the resistance versus time data after the transition occurs, line (b) in Figure A.1, and this line is extrapolated to an earlier time such that it intersects with line (a). The intersection is labelled as point A in Figure A.1. The value of resistance R_2 corresponding to intersection point A is recorded.

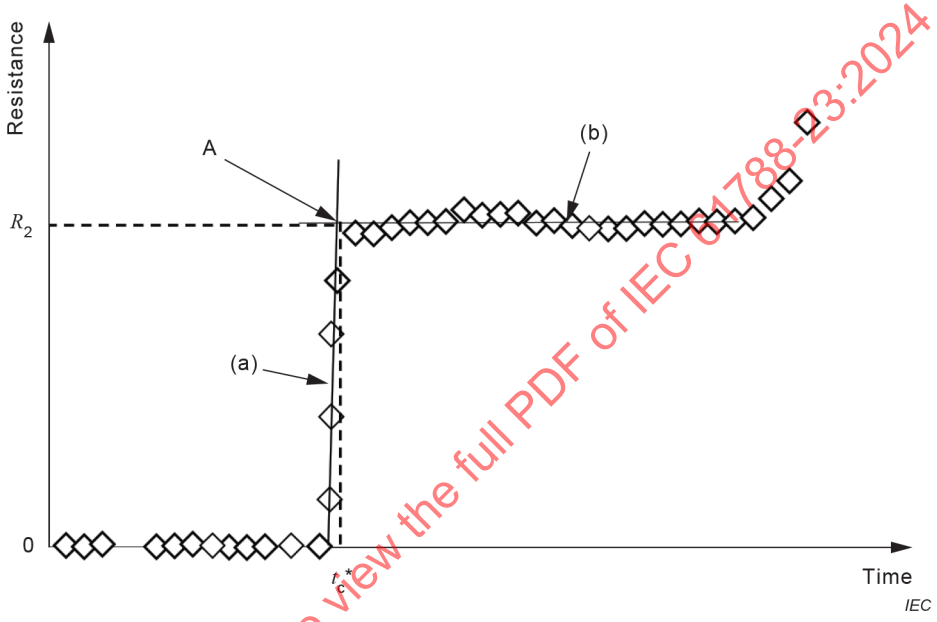

Figure A.1 – Determination of the value of R_2 from a resistance versus time plot

A.4.3 Comparison of ice point and room temperature

The value of R_1 is obtained at 273 K in [3]. According to Formula (2), the discrepancy between values of R_1 between 273 K and 293 K is 7,4 %. Actual measurements, drawn from the inter-laboratory comparison described in Clause C.2, reveal a slightly higher difference of 8,2 % ± 0,2 %.

A.4.4 Extrapolation of the resistance to 4,2 K

In [4], [5], [6] and [8], it was pointed out that the phonon contribution to niobium resistivity has a temperature dependence below 10 K. In this regime, the temperature dependence of resistance $R(T)$ can be fit with low uncertainty by a function of the form

$$R(T) = R_0 + aT^2 + bT^3 J_3(\theta_D/T) \quad \text{(A.1)}$$

where

R_0 is a residual resistance,

a and b are constants,

θ_D is the Debye temperature, and

$J_3(x)$ is the Grüneisen integral of the third kind.

In [4], linear plots of $R(T)$ versus T^3 were observed, suggesting that the coefficient a is small. A correction formula, to account for the difference between measurements of residual resistance at ~10 K and by extrapolation to 4,2 K, was derived in [5]. This formula is re-written here to give a relationship between RRR as determined by this document and a value determined by a ratio of R_1 to the resistance extrapolated to 4,2 K:

$$\frac{R_1}{R(4{,}2\ \mathrm{K})} = \frac{r_{\mathrm{RRR}}}{1 - 2{,}16 \times 10^{-4} r_{\mathrm{RRR}}} \tag{A.2}$$

For r_{RRR} = 300, Formula (A.2) will produce a resistance ratio of 321, resulting in a difference of 7 %.

A.4.5 Use of magnetic field to suppress superconductivity at 4,2 K

Superconductivity can be suppressed by the application of a magnetic field. For niobium, application of a field higher than 1 T will produce the normal state. In this case, a magneto-resistance appears, so a fit of the data shall be used to extrapolate to a value of R(4,2 K) at zero applied field. In [4] and [6], it was pointed out that fits generally obey Kohler's rule with linear dependence of R on an applied field H for field perpendicular to current. Curvature of $R(H)$ produced significant deviation from Kohler's rule for samples measured with current parallel to field. An acceptable uncertainty of 4 % was noted for field perpendicular to current, whereas uncertainty was 14 % for field parallel to current.

Agreement between the value of R(4,2 K) produced by extrapolation of magnetic field and that determined by extrapolation of temperature in A.4.4 was noted in [5]. Therefore, Formula (A.2) to estimate resistance at 4,2 K by extrapolation applies.

A.4.6 AC techniques

In [5], the use of a contactless method to apply an AC magnetic field and detect the signal according to induced current was described. However, this technique requires calibration by transfer of a reference sample measured either according to this document or to one of the techniques mentioned above. No direct relationship between the value of RRR obtained according to this document and the signal produced by the AC technique was described.

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 ISO/IEC Guide 98-3:2008 [9].

It was left to each technical committee to decide whether 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 IEC 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 can 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 is required to address for conformance to IEC 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 IEC TC 90 measurement standards were designed to limit the uncertainty of any quantity that could influence the measurement, based on the IEC TC 90 experts' 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 inter-laboratory comparison.

B.2 Definitions

Statistical definitions can be found in three sources: ISO/IEC Guide 98-3:2008 [9], ISO/IEC Guide 99:2007 [10], and the NIST Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results (NIST) [11]. Not all statistical terms used in this document are explicitly defined in ISO/IEC Guide 98-3:2008. For example, the terms "relative standard uncertainty" and "relative combined standard uncertainty" are used in ISO/IEC Guide 98-3:2008, e.g. 5.1.6, Annex J, but are not formally defined in that document (see [11]).

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 (note that 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) 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 in Table B.1 shows a set of electronic drift and creep voltage measurements from two nominally identical extensometers using the 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.2, Table B.3, Table B.4 and Table B.5 show the mean values, experimental standard deviations, standard uncertainties and COV values of two output signals, respectively.

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

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

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

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

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

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

Table B.5 – Coefficients of variation of two output signals

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

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

The standard uncertainty is very similar for the two extensometer deflections. In contrast, the coefficient of variation (X_{COV}) is different by nearly a factor of 2 800 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 IEC TC 90 standards

The observed value of a measurement does not usually coincide with the true value of the measurand. The observed value can 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. ISO/IEC Guide 98-3:2008, 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 ISO/IEC Guide 98-3:2008 procedure is illustrated in the following example.

a) Derive in the first step a mathematical measurement model in the form of an 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, F_{LC}:

$$F_{\mathrm{LC}} = F_{\mathrm{m}} + d_{\mathrm{W}} + d_{\mathrm{R}} + d_{\mathrm{Re}}$$

where F_{m}, d_{W}, d_{R}, and d_{Re} represent the force expected due to an applied standard mass, the manufacturer's data, repeated checks of standard weight per day and the reproducibility of checks on different days, respectively.

Here the input quantities are the measured force 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) Identify the type of distribution for each input quantity (e.g. Gaussian distributions for Type A measurements and rectangular distributions for Type B measurements).

Evaluate the standard uncertainty of the Type A measurements, $u_{\mathrm{A}} = s/\sqrt{n}$, where s is the experimental standard deviation and n is the total number of measured data points.

c) Evaluate the standard uncertainties of the Type B measurements:

$$u_{\mathrm{B}} = \sqrt{\frac{1}{3}d_{\mathrm{w}}^2 + \cdots}$$

where d_{w} is the range of rectangular distributed values.

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

$$u_{\mathrm{C}} = \sqrt{u_{\mathrm{A}}^2 + u_{\mathrm{B}}^2}$$

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 [12], [13].

e) 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.

f) 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 commercial software is a straightforward method that reduces the amount of routine work [14], [15]. In particular, the indicated partial derivatives can be easily obtained when such a software tool is used.

Annex C
(informative)

Uncertainty evaluation for resistance ratio measurement of Nb superconductors

C.1 Evaluation of uncertainty

C.1.1 Room temperature measurement uncertainty

The uncertainty of the test method arises from the terms in Formula (1), namely the uncertainty in room temperature resistance u_{R1} and the uncertainty in cryogenic resistance u_{R2}. For simplicity, the coverage factor k is kept equal to 1.

Since r_{RRR} is defined by Formula (1) as R_1/R_2, deviations Δr_{RRR} can be expressed by

$$\frac{\Delta r_{\mathrm{RRR}}}{r_{\mathrm{RRR}}} = \frac{\Delta R_1}{R_1} - \frac{\Delta R_2}{R_2} \tag{C.1}$$

The uncertainty u_{RRR} in the RRR measurement can then be expressed as

$$\frac{u_{\mathrm{RRR}}}{r_{\mathrm{RRR}}} = \left[\left(\frac{u_{R1}}{R_1} \right)^2 + \left(\frac{u_{R2}}{R_2} \right)^2 \right]^{0,5} \tag{C.2}$$

Formula (2) can be rearranged as

$$R_1 = \frac{U_1}{I_1}(2{,}084 - 0{,}003\,7\ T_1) \tag{C.3}$$

from which the deviations ΔR_1 can be expressed as

$$\Delta R_1 = (2{,}084 - 0{,}003\,7\ T_1)\left(\Delta U_1 \frac{1}{I_1} - \Delta I_1 \frac{U_1}{I_1^2} \right) - \Delta T_1 \frac{0{,}003\,7\ U_1}{I_1} \tag{C.4}$$

By expressing the deviations in Formula (C.4) as the uncertainties u_{U1}, u_{I1} and u_{T1}, respectively, the uncertainty of R_1 is

$$u_{R1} = \left[\left(\frac{C}{I_1} \right)^2 u_{U1}^2 + \left(\frac{CU_1}{I_1^2} \right)^2 u_{I1}^2 + \left(\frac{0{,}003\,7 U_1}{I_1} \right)^2 u_{T1}^2 \right]^{0,5} \quad \text{(C.5)}$$

where the constant C = (2,084 − 0,003 $7T_1$) lies between 0,97 and 1,03 when Formula (7) is valid.

C.1.2 Cryogenic measurement uncertainty

The cryogenic measurement relies on acquiring a number of data points R defined by Formula (4). For each measured data point R_i, deviations ΔU_2 and ΔI_2 exist. They produce an uncertainty u_{Ri} according to

$$u_{Ri} = \left[2\left(\frac{u_{U2}}{I_2} \right)^2 + \left(\frac{U_2}{I_2^2} \right)^2 u_{I2}^2 \right]^{0,5} \quad \text{(C.6)}$$

where u_{U2} is the uncertainty in U_2 and u_{I2} is the uncertainty in I_2. Note that the factor 2 accounts for the averaging of values U_2^+ and U_2^- [1]. It is assumed that the uncertainties in Formula (C.6) are not dependent on temperature or time. Using conservative ratings for uncertainties that are often met by modern electronics, $u_{U2} = 0{,}002\,8\ U_2$ and $u_{I2} = 0{,}002\,8\ I_2$ (see Table C.1), it can be estimated that under ideal conditions, $u_{Ri}/R_i = 0{,}5\%$. However, this assessment assumes that thermal gradients, thermal currents, and other factors do not contribute systematic variations in addition to the random uncertainty imposed by the measurement equipment. In some test configurations, observed uncertainty for each data point can be 5 %.

The measurement technique discussed in 7.3 requires the user of this document to separate the points R_i into two ensembles: one related to line (a) and one related to line (b). For either ensemble, the data points R_i will vary around the mean values $\hat{R}_i$ by an uncertainty u_{Ri}. When the temperature method of Figure 1 is used, each ensemble of R_i values approximates the linear function $\hat{R}_i(T) = \alpha T + \beta$, and linear regression analysis can be used to assess the uncertainties u_α and u_β. A parallel analysis applies to the method of Figure C.1.

The standard deviations of regression for line (a) and line (b), denoted by s_a and s_b, respectively, can be expressed as

$$s_{\mathrm{a,b}} = \sqrt{\frac{\sum_{\mathrm{a,b}} \left(R_i - \hat{R}_i \right)^2}{N_{\mathrm{a,b}} - 2}} \quad \text{(C.7)}$$

where the subscripts a and b denote the ensemble of points used for line (a) or line (b), respectively. The denominator indicates that two degrees of freedom have been removed from the number, N_{a} or N_{b}, of data points in ensemble (a) or ensemble (b), respectively, due to the conditions that the ensemble of data points represents the true population, and that the set of data points is representative of the true data. Formula (C.7) thus requires the recording of at least three data points for each line to keep the denominator positive.

For the temperature method, the uncertainty of slope for lines (a) and (b), denoted as u_{α}^{a} and u_{α}^{b}, respectively, depends on the ensemble of measured temperature T_i relative to the average temperatures $\overline{T}_{\mathrm{a}}$ and $\overline{T}_{\mathrm{b}}$ of ensembles (a) and (b), respectively, via

$$u_{\alpha}^{\mathrm{a,b}} = \frac{s_{\mathrm{a,b}}}{\sqrt{\sum_{\mathrm{a,b}}\left(T_i - \overline{T}_{\mathrm{a,b}}\right)^2}} \tag{C.8}$$

Here, the subscripts again denote different ensembles of data points associated with either line (a) or line (b). Formula (C.8) indicates that measurements over a broader range of temperature will reduce the uncertainty in the slope, provided that a linear regression holds for the full temperature range. A similar expression can be derived for recording measurements as a function of time.

The uncertainty of intercept at T_{c}^{*} for either line can be expressed as

$$u_{\beta}^{\mathrm{a,b}} = s_{\mathrm{a,b}}\sqrt{\frac{\sum_{\mathrm{a,b}}\left(T_i - T_{\mathrm{c}}^{*}\right)^2}{N_{\mathrm{a,b}}\sum_{\mathrm{a,b}}\left(T_i - \overline{T}_{\mathrm{a,b}}\right)^2}} \tag{C.9}$$

Since the uncertainty in Formula (C.9) lies along the vertical line used to determine T_{c}^{*}, it is important to add a correction to account for the extrapolation of line (b) onto line (a) to arrive at the uncertainty u_{R2} in the value of R_2. This can be approximated by

$$u_{R2} = \left[\left(\alpha_{\mathrm{b}} u_{\beta}^{\mathrm{a}} / \alpha_{\mathrm{a}}\right)^2 + \left(u_{\beta}^{\mathrm{b}}\right)^2\right]^{0,5} \tag{C.10}$$

A graphical representation of the uncertainty is shown in Figure C.1. Note that any contributions of thermoelectric voltage are not considered in this uncertainty analysis.

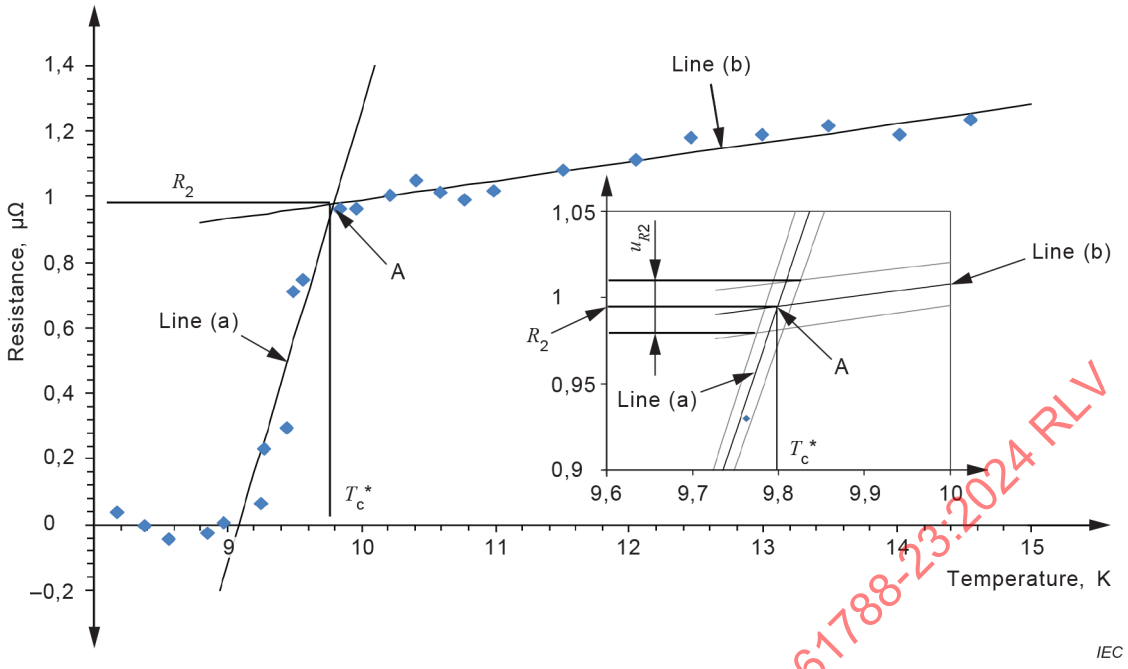

Figure C.1 – Graphical description of the uncertainty of regression related to the measurement of R_2

C.1.3 Estimation of uncertainty for typical experimental conditions

The cryogenic resistance uncertainty is estimated here, assuming that cryogenic measurements acquired five data points for line (a) and 10 data points for line (b). The data ensemble (a) is assumed to be acquired in 0,2 K intervals with $\overline{T}_a$ = 9,5 K, and ensemble (b) is assumed to be acquired in 0,5 K intervals with $\overline{T}_b$ = 11,0 K, with values R_i ≥ 1,0 μΩ above the transition and T_c^* = 9,8 K. The slope of line (a) is 2 μΩ K^{-1}, and the slope of line (b) is 0,05 μΩ K^{-1}. For this estimation, an uncertainty u_{Ri} = 0,05 μΩ is assumed for each data point. From Formula (C.7), s_a ≈ 0,065 μΩ, and s_b ≈ 0,055 μΩ. From Formula (C.8), a careful fit for line (a) would have an uncertainty of slope of 0,21 μΩ K^{-1}, while the fit for line (b) would have an uncertainty of slope of 0,010 μΩ K^{-1}. The intercepts of lines (a) and (b) with a hypothetical vertical line at T_c^* would have an uncertainty from Formula (C.9) of 0,069 μΩ and 0,032 μΩ, respectively. The combined uncertainty u_{R2} from Formula (C.10) is then 0,032 μΩ. Note that the uncertainty u_{R2} scales approximately as $u_{Ri}(2/N_b)^{0,5}$ since point A lies at the end of line (b).

Table C.1 – Uncertainty of measured parameters

Uncertainty	Type	Value	Remarks
u_{U1} / U_1	B	0,28 %	Instrument uncertainty of 0,5 %
u_{I1} / I_1	B	0,28 %	Instrument uncertainty of 0,5 %
u_{T1}	B	0,58 K	Thermometer uncertainty of 1,0 K
u_{U2} / U_2	B	0,28 %	Instrument uncertainty of 0,5 %
u_{I2} / I_2	B	0,28 %	Instrument uncertainty of 0,5 %

These additional parameters apply for the room temperature uncertainty estimation: T_1 = 300 K, U_1 = 3,00 µV, I_1 = 10,0 mA. These parameters give r_{RRR} = 292 for the value of R_2 above. In view of the data in Table C.1, u_{U1} = 0,002 8 × U_1 = 8,40 nV, u_{I1} = 0,002 8 × I_1 = 28,0 µA, and u_{T1} = 0,58 K. From Formula (C.5), u_{R1} = 1,32 µΩ.

From Formula (C.2), it can be estimated that u_{RRR} = 9,43. That is, r_{RRR} = 292 ± 9,43 for this example. This uncertainty is dominated by the uncertainty u_{R2}.

As summarized in Clause C.2, inter-laboratory comparison results gave a typical uncertainty much lower than the value (9,43/292) = 3,2 % assessed above. High-quality equipment, careful design of apparatus, and well-controlled test conditions can have resulted in lower values of u_{R2} compared with the assumed uncertainty of 0,05 µΩ. Higher measuring current I_2 and acquisition of more data points than assumed for N_b above can also contribute to reduced uncertainty according to Formula (C.6) and Formula (C.9).

C.2 Inter-laboratory comparison summary

Four international testing laboratories participated in an inter-laboratory comparison of 10 niobium specimens. All specimens were prepared by the vendor in a manner consistent with this document. Test techniques included both that defined by this document as well as those described by A.4.1, A.4.2 and A.4.3, all of which are methods that use liquid helium. Table C.2 summarizes the results of the inter-laboratory comparison. A typical uncertainty across laboratories of 0,3 % to 1,3 % has thus been obtained.

Table C.2 – RRR values obtained by inter-laboratory comparison using liquid helium

Sample	RRR by International Standard					
	Laboratory 1	Laboratory 2	Laboratory 3	Laboratory 4	Average	$sN^{-0,5}$ (% of average)
1	367	370	364		367	0,4
2	347	348	348		348	< 0,1
3	391	390	386		389	0,3
4	379	378	375		377	0,3
5	417	418	418	417	418	< 0,1
6	408	395	417		407	1,3
7	382	377	381		380	0,3
8	383	370	378		380	0,3
9	344	328	341		338	1,2
10	332	332	333	336	333	0,3

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SOMMAIRE

COMMISSION ÉLECTROTECHNIQUE INTERNATIONALE

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SUPRACONDUCTIVITÉ –

Partie 23: Mesurage du rapport de résistance résiduelle – Rapport de résistance résiduelle des supraconducteurs de Nb à cavités

AVANT-PROPOS

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Cette troisième édition annule et remplace la deuxième édition parue en 2021. Cette édition constitue une révision technique.

Cette édition inclut les modifications techniques majeures suivantes par rapport à l'édition précédente:

a) le principe a été modifié pour refléter la méthode d'essai actuelle.

Le texte de cette Norme internationale est issu des documents suivants:

Projet	Rapport de vote
90/515/FDIS	90/519/RVD

Le rapport de vote indiqué dans le tableau ci-dessus donne toute information sur le vote ayant abouti à son approbation.

La langue employée pour l'élaboration de cette Norme internationale est l'anglais.

Ce document a été rédigé selon les Directives ISO/IEC, Partie 2, il a été développé selon les Directives ISO/IEC, Partie 1 et les Directives ISO/IEC, Supplément IEC, disponibles sous www.iec.ch/members_experts/refdocs. Les principaux types de documents développés par l'IEC sont décrits plus en détail sous www.iec.ch/publications.

Une liste de toutes les parties de la série IEC 61788, publiées sous le titre général *Supraconductivité*, se trouve sur le site web de l'IEC.

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INTRODUCTION

Le niobium de grande pureté est la matière principale utilisée dans la fabrication des cavités radioélectriques supraconductrices. Des nuances similaires de niobium peuvent être utilisées dans la fabrication de fils supraconducteurs. Le rapport de résistance résiduelle (RRR) est souvent utilisé dans le cadre de l'approvisionnement en matières premières et l'assurance qualité des produits livrés pour spécifier ou évaluer la pureté d'un métal. Le RRR est défini pour des métaux non supraconducteurs comme le rapport de la résistance électrique mesurée à la température ambiante (293 K) sur la résistance mesurée pour la même éprouvette à basse température (~4,2 K). La valeur à basse température est souvent appelée résistance résiduelle. Une plus grande pureté est associée à des valeurs plus élevées de RRR.

Le niobium pose des problèmes particuliers en raison de son passage à un état supraconducteur à ~9 K, la résistance électrique en courant continu étant effectivement nulle au-dessous de cette température. La définition ci-dessus donne alors une valeur infinie du RRR. Le présent document spécifie une méthode d'essai qui permet de déterminer la valeur de la résistance résiduelle à l'aide d'une courbe de la résistance en fonction de la température, puisque l'éprouvette s'échauffe progressivement au cours de la transition supraconductrice en l'absence de l'application d'un champ magnétique. Cette méthode donne une valeur de résistance résiduelle juste au-dessus de la transition supraconductrice, ~10 K, qui sert de base à la détermination du RRR.

Il existe également des Normes internationales qui permettent de déterminer le RRR des fils supraconducteurs. Contrairement aux fils supraconducteurs, qui sont généralement constitués d'un matériau supraconducteur et d'un matériau non supraconducteur (la valeur du RRR étant seulement représentative du composant non supraconducteur), l'intégralité de l'éprouvette utilisée dans le présent document est constituée de niobium supraconducteur. Le niobium est souvent obtenu sous la forme d'une feuille, d'une barre ou d'une tige, et non sous la forme d'un fil. De ce fait, les éprouvettes sont susceptibles de présenter des dimensions transversales de l'ordre de quelques millimètres à la circulation du courant électrique. Cette différence est importante lors du mesurage de la résistance électrique, puisque les échantillons de niobium sont susceptibles d'être beaucoup plus longs que ceux d'un fil pour un même rapport longueur/diamètre, et il peut être nécessaire de disposer d'un courant électrique plus élevé pour produire suffisamment de signaux de tension. L'Annexe A fournit des recommandations pour les dimensions des échantillons et les connexions électriques. De même, il convient que l'appareillage d'essai prenne en considération les aspects suivants: l'orientation de l'éprouvette par rapport à la surface de l'hélium liquide, l'accessibilité par des orifices sur les vases de Dewar types d'hélium liquide, la conception des contacts de courant et la réduction le plus possible des gradients thermiques sur d'importantes longueurs d'éprouvettes. Ces aspects différencient le présent document des normes similaires relatives aux fils.

D'autres méthodes d'essai ont été utilisées pour calculer le RRR. Certaines méthodes procèdent à un mesurage à une température autre que 293 K pour la valeur de la résistance élevée. Certaines méthodes utilisent des extrapolations à 4,2 K en l'absence d'application d'un champ magnétique pour la valeur de résistance faible. D'autres méthodes appliquent un champ magnétique pour supprimer la supraconductivité à 4,2 K. L'Annexe A établit une comparaison entre le présent document et d'autres méthodes d'essai. Noter que ces autres méthodes produisent des différences systématiques jusqu'à 10 %, ce qui représente une valeur plus importante que l'incertitude cible du présent document. Il est donc important d'appliquer le présent document ou les corrections appropriées indiquées à l'Annexe A selon la méthode d'essai utilisée.

Il convient de fournir dans la mesure du possible cette méthode d'essai aux fournisseurs et aux collaborateurs qui effectuent également des mesurages du RRR. Pour assurer la cohérence, les résultats des comparaisons interlaboratoires sont décrits à l'Article C.2.

SUPRACONDUCTIVITÉ –

Partie 23: Mesurage du rapport de résistance résiduelle – Rapport de résistance résiduelle des supraconducteurs de Nb à cavités

1 Domaine d'application

La présente partie de l'IEC 61788 spécifie une méthode d'essai pour la détermination du rapport de résistance résiduelle (RRR), r_{RRR}, du niobium à cavités. Cette méthode est destinée aux nuances de niobium de grande pureté de $150 < r_{RRR} < 600$. La méthode d'essai est valide pour des éprouvettes à sections rectangulaires ou circulaires, de surface de section supérieure à 1 mm^2, mais inférieure à 20 mm^2, et dont la longueur n'est pas inférieure à 10 fois ni supérieure à 25 fois la largeur ou le diamètre.

2 Références normatives

Les documents suivants sont cités dans le texte de sorte qu'ils constituent, pour tout ou partie de leur contenu, des exigences du présent document. Pour les références datées, seule l'édition citée s'applique. Pour les références non datées, la dernière édition du document de référence s'applique (y compris les éventuels amendements).

IEC 60050-815, *Vocabulaire Électrotechnique International – Partie 815: Supraconductivité* (disponible à l'adresse: https://www.electropedia.org/)

3 Termes et définitions

Pour les besoins du présent document, les termes et définitions de l'IEC 60050-815 ainsi que les suivants s'appliquent.

L'ISO et l'IEC tiennent à jour des bases de données terminologiques destinées à être utilisées en normalisation, consultables aux adresses suivantes:

- IEC Electropedia: disponible à l'adresse https://www.electropedia.org/
- ISO Online browsing platform: disponible à l'adresse https://www.iso.org/obp

3.1
rapport de résistance résiduelle
RRR
r_{RRR}
rapport de la résistance à la température ambiante à la résistance juste au-dessus de la transition supraconductrice

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

où

R_1 est la résistance à la température ambiante, 293 K;

R_2 est la résistance juste au-dessus de la transition supraconductrice, à ~10 K.

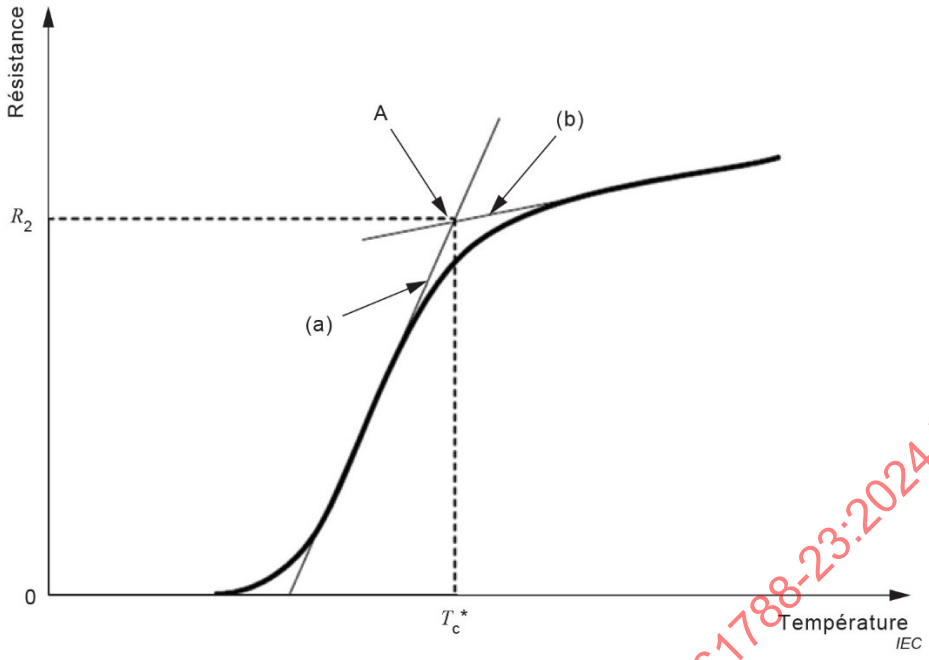

Figure 1 – Rapport entre la température et la résistance à proximité de la transition supraconductrice

Note 1 à l'article: Dans le présent document, la température ambiante est définie comme étant de 20 °C = 293 K, et r_{RRR} est obtenu comme suit: La Figure 1 représente de manière schématique la résistance en fonction des données de température et la procédure graphique utilisée pour déterminer la valeur de R_2. Sur la Figure 1, la région de pente maximale est extrapolée en résistance vers le haut, comme cela est indiqué par la ligne (a), et la région de pente minimale à des températures supérieures à la température de transition est extrapolée en température vers le bas, comme cela est indiqué par la ligne (b). Le point d'intersection A de ces extrapolations détermine la valeur de R_2 ainsi qu'une valeur de température T_c^*.

Note 2 à l'article: La valeur T_c^* est similaire à la valeur de transition définie dans [1], et elle est différente de la valeur définie au point médian de la transition, appelée T_c^* dans [2].

Note 3 à l'article: Certaines normes ou techniques documentées, par exemple [3], [4], [5], [6], définissent r_{RRR} avec la valeur de R_1 déterminée à une température autre que 293 K, ou la valeur de R_2 déterminée à une température inférieure à la transition supraconductrice. L'attention de l'utilisateur est attirée sur ces différences contenues dans la définition.

4 Principe

La technique de résistance électrique en courant continu à 4 points doit être réalisée à la fois à la température ambiante et à la température cryogénique. L'essai de résistance doit être effectué en fonction de la température. Une autre méthode d'essai de résistance en fonction du temps avec une montée en température est décrite en A.4.2.

L'incertitude type composée relative de cette méthode est de 3 % avec un facteur d'élargissement de 2.

Les mesurages doivent avoir les attributs suivants.

a) Le courant de mesure est suffisamment élevé pour fournir des signaux de tension de l'ordre de 1 µV. Pour la sécurité électrique, il convient que la densité de courant maximale ne dépasse jamais 1 A mm^{-2}.

b) La résistance de contact des conducteurs de courant est suffisamment faible pour éviter un échauffement excessif de l'échantillon. Les conditions de mesure cryogénique types exigent une dissipation de puissance inférieure à 1 mW au niveau des contacts.

c) Les effectifs d'échantillons sont suffisamment importants pour réduire le plus possible la probabilité de dommages dus à la coupe et à la manipulation. Les échantillons types présentent une section transversale comprise entre 1 mm et 3 mm et une surface de section supérieure à 5 mm^2.

d) La longueur de l'échantillon est d'au moins 10 fois et d'au maximum 25 fois la largeur ou le diamètre.

L'Annexe A traite des considérations relatives aux dimensions de l'échantillon et au courant de mesure.

5 Appareillage de mesure

5.1 Mandrin ou embase

Un mandrin ou une embase rectiligne doit être utilisé(e) pour supporter l'éprouvette. Les matériaux possibles de construction comprennent le cuivre pur, l'aluminium pur, l'argent pur, les nuances électriques de Cu-Zr, Cu-Cr-Zr, Cu-Be et autres alliages de cuivre, les nuances électriques d'Al-Mg, d'Al-Ag et autres alliages d'aluminium ainsi que les nuances électriques d'alliages d'argent. Ceux-ci offrent une conductivité thermique élevée et servent à éliminer les gradients thermiques pendant le mesurage. L'éprouvette doit être isolée du mandrin. Les matériaux isolants éventuels qui peuvent être appliqués sous forme de feuilles, de rubans ou de revêtements comprennent le polyéthylène téréphtalate, le polyester et le polytétrafluoroéthylène. De la résine époxyde renforcée de fibre de verre ou d'autres matériaux composites qui ont une bonne conductivité thermique à la température cryogénique peuvent également être utilisés.

Il convient que l'embase présente un fini de surface propre et lisse. Il convient que l'embase soit exempte de bavures, arêtes, coutures ou autres aspérités qui peuvent altérer l'éprouvette. Les éprouvettes de niobium de grande pureté sont souples et sont susceptibles de subir des indentations causées par des défauts de surface. Ces indentations peuvent altérer l'échantillon et invalider le mesurage de la résistance.

Le mandrin ou l'embase doit supporter toute la longueur et toute la largeur de l'éprouvette. Il convient que la géométrie du mandrin ou de l'embase n'exerce aucune contrainte de flexion supérieure à 0,2 % sur l'échantillon.

Un thermomètre d'une exactitude de 0,1 K est utile, mais n'est pas exigé. Le mandrin ou l'embase peut intégrer un montage pour placer un thermomètre cryogénique directement sur le corps du mandrin ou de l'embase et à proximité du centre de l'éprouvette.

Dans la pratique, la longueur des embases est d'au moins 30 mm pour permettre l'assemblage des pièces et la manipulation des échantillons à la main. Plusieurs échantillons peuvent être montés sur une seule embase.

5.2 Cryostat et support du mandrin ou de l'embase

L'appareillage doit prévoir un support mécanique du mandrin ou de l'embase. De plus, ce support doit fournir des fils électriques pour le transport du courant pour les échantillons et les thermomètres, et doit mesurer leurs tensions. Pour les mesurages de R_1 et de R_2, le support ne doit laisser passer le courant qu'à travers l'échantillon, de sorte que toute la tension résultante mesurée soit uniquement celle générée par l'échantillon.

La structure de support doit permettre de mesurer à la fois R_1 et R_2 sans démontage ni remontage de l'éprouvette. Le mesurage de R_2 doit exiger l'utilisation d'un cryostat qui, de plus, doit s'intégrer au support.

Le cryostat doit comprendre un réservoir à hélium liquide au fond d'une grande colonne verticale. Une structure de support doit permettre d'immerger l'échantillon dans le bain d'hélium et de l'en sortir. De plus, une pièce d'ancrage de la position de l'échantillon, lorsqu'il est immergé dans l'hélium liquide ou suspendu au-dessus de la surface du liquide à une hauteur arbitraire, doit être prévue. Cette suspension permet de stabiliser la température pendant le mesurage et de l'augmenter lentement en fonction de la hauteur au-dessus du bain d'hélium. En variante, l'immersion de l'échantillon dans le bain, suivie de la réduction du niveau du bain par évaporation ou transfert sous pression peut également permettre de faire varier la température.

Un réchauffeur peut servir à chauffer le mandrin ou l'embase. Il convient que le réchauffeur répartisse la chaleur le long du mandrin et il convient d'éviter des réglages d'intensité excessifs. Par exemple, une source ponctuelle d'entrée de chaleur de 1 W qui fonctionne au centre d'un mandrin de 1 cm^2 sur lequel est monté un échantillon de 5 cm peut produire une différence de température de 2,5 K le long de l'échantillon si la conductivité thermique est de 100 W m^{-1} K^{-1}.

Des techniques cryogéniques appropriées doivent être appliquées pour la construction du cryostat et de l'appareillage. Cela comprend l'utilisation de matériaux à faible conductivité thermique tels que des tubes en acier inoxydable à paroi mince, des matériaux composites, de la céramique et des isolants pour éviter l'évaporation excessive due à la conduction thermique qui émane de l'environnement. L'embase ou le mandrin qui comporte l'échantillon peut être entouré d'un boîtier ou d'un blindage pour augmenter la stabilité thermique. Il convient de prévoir sur l'appareillage des dispositifs pour la limitation de pression et l'isolation sous vide de l'hélium liquide.

6 Préparation de l'éprouvette

Le niobium de grande pureté est assez malléable, de sorte que même la plus petite force peut déformer le matériau. Étant donné que les dislocations sont une source de diffusion d'électrons, la déformation de l'éprouvette peut contribuer par inadvertance à la résistivité résiduelle et compromettre le résultat de l'essai. Des protocoles spéciaux doivent donc être suivis lors de la préparation de l'éprouvette. Les techniques de découpage doivent dans toute la mesure du possible éviter la chaleur et la contrainte. L'usinage par électroérosion, le découpage par jet de fluide ou l'usinage conventionnel à faible vitesse sont des techniques acceptables et largement utilisées pour les applications qui utilisent du niobium de grande pureté. Les éprouvettes découpées à partir de pièces plus grandes doivent être protégées et bloquées contre un support pendant le transport. Les opérations d'ébavurage des échantillons ne doivent pas plier, chauffer excessivement ni endommager les échantillons. Un ponçage léger avec du papier fin constitue une approche acceptable.

Il convient que les éprouvettes soient des barres rectangulaires ou circulaires de section transversale uniforme. Les grands côtés de l'éprouvette doivent être parallèles. Toute torsion ou courbure doit être évitée afin de n'appliquer aucune flexion ou torsion à l'éprouvette lors de son montage sur le mandrin ou l'embase. Les éprouvettes qui forment un arc ou un "U" sont acceptables à condition que toute la courbure puisse être supportée sur un plan, sans appliquer de torsion à l'éprouvette pliée.

L'éprouvette doit être propre et exempte de trace de résidus de fluides de coupe ou autres contaminants de surface. Les résidus sont de préférence nettoyés par dégraissage à l'aide de solvants, suivi d'un nettoyage aux ultrasons avec un détergent doux à base aqueuse et d'un rinçage à l'eau distillée ou ultra pure, puis du séchage à l'air. Il convient d'éviter tout décapage chimique pour nettoyer la surface au risque d'introduire des contaminants, en particulier de l'hydrogène et de l'oxygène. En général, il suffit de procéder à un polissage mécanique doux des régions de connexion des prises de réglage de tension et des conducteurs de courant pour éliminer les oxydes de surface. Le revêtement de ces régions avec une feuille d'indium ou un autre métal (par évaporation ou par pulvérisation cathodique, par exemple) est une méthode acceptable pour protéger des contacts polis à condition de ne pas revêtir l'intégralité de l'éprouvette.

L'éprouvette doit être constituée d'une seule pièce et ne doit présenter aucun joint ou épissure.

Une méthode mécanique doit être utilisée pour fixer l'éprouvette sur le mandrin ou l'embase. L'installation et l'instrumentation de l'éprouvette ne doivent pas appliquer de charge excessive, contrainte de flexion, contrainte de traction ou torsion sur l'éprouvette.

L'éprouvette doit comporter des contacts de courant à proximité de chacune de ses extrémités et une paire de contacts de tension sur la partie centrale entre les contacts de courant (c'est-à-dire une technique de mesure à 4 points). Une distance non inférieure à la plus grande dimension (largeur, épaisseur ou diamètre) perpendiculaire à la longueur de l'éprouvette doit séparer les contacts de tension des contacts de courant.

7 Acquisition et analyse des données

7.1 Matériel d'acquisition des données

Les alimentations électriques modernes peuvent être commandées par ordinateur et présentent une variété de caractéristiques qui favorisent la commande à distance de la sortie de courant. Il n'est pas exigé d'utiliser de telles alimentations bien qu'elles puissent largement faciliter l'automatisation de l'acquisition des données. Des modes pulsés permettent d'appliquer le courant seulement lorsque des signaux de tension sont en cours d'acquisition, éliminant ainsi la chaleur générée dans l'échantillon pendant le cycle d'arrêt. En cas d'application du courant pulsé, la durée d'impulsion doit inclure de longues périodes pour la stabilisation et le filtrage des signaux de tension.

Certaines alimentations incorporent un shunt interne pour réguler le courant de sortie. Si une telle alimentation est utilisée, le shunt interne doit être étalonné périodiquement avec un mesurage du shunt externe et de la tension.

Le montage d'essai peut fixer une tension de base arbitraire U_0, qui peut être détectée lorsque l'échantillon est dans l'état supraconducteur et que l'alimentation est coupée. La valeur U_0 peut dériver au fil du temps en raison des variations de l'environnement thermique et d'autres facteurs. Un matériel de conception avancée permet la compensation de la dérive et le retour à zéro automatique de sorte que la moyenne temporelle de U_0 soit égale à 0. Les tensiomètres numériques ne sont pas exigés, mais améliorent considérablement l'acquisition des données. Outre la compensation de la dérive et le retour à zéro de la tension, le filtrage et la compensation interne des tensions à induction thermique peuvent améliorer l'exactitude de mesure de la tension. Il convient que le filtrage couvre en moyenne les signaux de tension pendant une durée au moins égale à la constante de temps thermique de l'appareillage à basse température, généralement un intervalle de 0,1 s à 10 s. Il est important de comprendre la façon dont les tensions sont corrigées pour la dérive et les effets thermiques. Les tensiomètres sensibles, en particulier les nanovoltmètres, exigent un préamplificateur qui doit être à l'équilibre thermique et qui peut exiger plusieurs heures de fonctionnement avant le mesurage.

L'acquisition de données par ordinateur facilite considérablement l'enregistrement et la consignation des données.

7.2 Résistance (R_1) à la température ambiante

La température ambiante T_1 du laboratoire de mesure doit être mesurée. Un courant d'essai I_1 doit être appliqué conformément aux exigences de l'Article 4. La tension résultante U_1 doit être enregistrée avec I_1 et T_1. La résistance doit être déterminée par

$$R_1 = \frac{U_1}{I_1}\left[1 - 0{,}003\ 7\ \left(T_1 - 293\right)\right] \tag{2}$$

avec T_1 en unités de kelvin. Le coefficient 0,003 7 reflète le taux de variation expérimental de la résistance en fonction de la température mentionnée dans [7] [1] sur l'intervalle de 273 K à 300 K.

7.3 Résistance résiduelle (R_2) juste au-dessus de la transition supraconductrice

Le mesurage de R_2 doit être effectué avec l'échantillon toujours monté sur le mandrin ou l'embase de mesure de R_1.

L'éprouvette doit être placée dans un cryostat comme cela est spécifié en 5.2. L'éprouvette doit être lentement immergée dans un bain d'hélium liquide et refroidie à la température de l'hélium liquide. Pendant qu'une forte évaporation d'hélium liquide accompagne le refroidissement initial, l'élimination de la chaleur du mandrin, surtout s'il est blindé, peut exiger plus de 5 min. Le courant peut être appliqué et la tension surveillée pendant cette période, mais aucun mesurage ne doit être effectué avant la fin de la forte évaporation de l'hélium liquide.

Lorsque le taux d'évaporation convient aux mesurages, la tension U_0 doit être mesurée pendant que l'échantillon est immergé dans l'hélium liquide. L'échantillon est susceptible d'être à l'état supraconducteur dans ces conditions. Le courant I_2 doit ensuite être appliqué selon les exigences de l'Article 4 et les considérations de l'Article 5. Les tensions doivent être relevées pour la polarité de courant direct et de courant inverse, respectivement U_0^+ et U_0^-. Les différences entre U_0, U_0^+ et U_0^- doivent être enregistrées.

L'éprouvette doit ensuite être progressivement chauffée de façon à passer progressivement de l'état supraconducteur à l'état normal. Un appareillage conforme à l'Article 5 permet de réchauffer progressivement l'éprouvette en élevant par exemple le niveau du mandrin au-dessus du niveau du bain d'hélium liquide. Deux tensions U_2^+ et U_2^- doivent être mesurées presque simultanément en appliquant le même courant de mesure I_2 avec la polarité directe et inverse, respectivement. Le courant ne doit pas être appliqué lorsque les mesurages ne sont pas enregistrés. La tension U_2 doit être déterminée par la formule

$$U_2 = \frac{\left|U_2^+ - U_2^-\right|}{2} \tag{3}$$

[1] Les chiffres entre crochets renvoient à la Bibliographie.

pour laquelle il convient de noter que le signe de U_2^- est opposé à celui de U_2^+. Il s'ensuit que la Formule (3) indique une moyenne des deux nombres à peu près égaux en valeur. Une résistance R doit être déterminée à partir de la tension par la formule

$$R = \frac{U_2}{I_2} \tag{4}$$

Lors du réchauffement de l'échantillon, les valeurs de R doivent être enregistrées en fonction de la température T déterminée par le thermomètre fixé au mandrin ou à l'embase. Il est admis d'utiliser des outils de représentation graphique et des logiciels d'analyse de données pour tracer la courbe de la résistance en fonction de la température et pour effectuer des extrapolations.

Une courbe de la résistance en fonction de la température doit donc être obtenue (voir la Figure 1). La courbe de la résistance en fonction de la température doit être enregistrée en continu jusqu'à atteindre une température d'au moins 15 K. La courbe de la résistance en fonction de la température doit être analysée en traçant une ligne dans la région de plus forte pente près du point médian de montée en résistance (ligne (a) de la Figure 1) et en extrapolant cette ligne suffisamment au-dessus de la valeur de R enregistrée à 15 K. Une seconde ligne doit être tracée dans la région de la courbe de la résistance en fonction de la température au-dessus de la transition (ligne (b) de la Figure 1), et cette ligne doit être extrapolée à une température suffisamment basse pour couper la ligne (a). L'intersection est désignée comme le point A de la Figure 1. La valeur de la résistance R_2 qui correspond au point d'intersection A doit être enregistrée, ainsi que la valeur de la température T_c^* qui correspond au point d'intersection A.

7.4 Validation du mesurage de la résistance résiduelle

La détermination de R_2 doit être valide si tous les critères suivants sont remplis.

Les tensions parasites doivent être telles que

$$\frac{\left|U_0^+ - U_0^-\right|}{I_2 R_2} < 3\ \% \tag{5}$$

La dérive ou la dispersion thermique doit être telle que, pour des valeurs consécutives U_2^+ et U_2^- enregistrées avec une température proche de T_c^*,

$$\frac{\left|U_2^+ + U_2^-\right|}{I_2 R_2} < 3\ \% \tag{6}$$

La température ambiante doit être telle que

$$283\,\mathrm{K} < T_1 < 303\,\mathrm{K} \tag{7}$$

7.5 Rapport de résistance résiduelle (RRR)

Le RRR doit être calculé au moyen de la Formule (1) et la valeur obtenue doit être enregistrée.

8 Incertitude de la méthode d'essai

D'après les résultats de la comparaison interlaboratoire, récapitulés à l'Article C.2, une incertitude type de 0,3 % à 1,3 % a été obtenue entre laboratoires.

9 Rapport d'essai

9.1 Généralités

Un rapport d'essai qui récapitule les résultats de la méthode d'essai de RRR doit être fourni.

9.2 Informations d'essai

Les informations d'essai enregistrées doivent comprendre les éléments suivants:

a) la date et l'heure du mesurage;
b) le nom de l'opérateur;
c) l'édition de l'IEC 61788-23 suivie.

9.3 Informations sur l'éprouvette

Les informations suivantes relatives à l'éprouvette doivent être consignées dans le rapport d'essai:

a) les informations de traitement thermique du fournisseur, de fabrication ou toute autre information de suivi, comme un numéro de bon de commande;
b) le numéro d'identification de la feuille ou de la pièce, le cas échéant;
c) la forme et l'orientation de l'éprouvette par rapport au bain d'hélium.

9.4 Conditions d'essai

Le rapport d'essai doit inclure les conditions d'essai suivantes:

a) la température ambiante T_1;
b) les courants de transport I_1 et I_2;
c) les tensions U_1 et U_2, en notant que U_2 varie en fonction de la température et exige donc d'être consignée sous forme de tableau ou de graphique;
d) les résistances R_1 et R_2;
e) les tensions U_0, U_0^+, U_0^- ou la validation, Formule (5).

Les informations supplémentaires suivantes peuvent être incluses dans le rapport d'essai:

f) la distance de la prise de réglage de tension L;

g) les dimensions de l'éprouvette et la surface de la section A;

h) la résistivité $\rho_1 = R_1 A L^{-1}$ et $\rho_2 = R_2 A L^{-1}$.

9.5 Valeur de RRR

La valeur de RRR doit être exprimée comme $r_{RRR} \pm u_{RRR}$, par exemple, 300 ± 19,2 ($k = 2$), où u_{RRR} est l'incertitude type composée conformément à l'Annexe C. En variante, r_{RRR} peut être exprimée comme une valeur minimale (par exemple, la valeur minimale 285) pour indiquer la limite inférieure de l'intervalle de confiance représenté par l'incertitude. Il n'est pas nécessaire de consigner l'incertitude pour un seul mesurage. Sauf spécification contraire, il convient d'exprimer les résultats sous forme de trois chiffres significatifs.

L'Annexe A fournit des informations supplémentaires relatives au mesurage du RRR. L'Annexe B donne les définitions et un exemple de l'incertitude de mesure. L'Annexe C spécifie l'évaluation de l'incertitude dans la méthode d'essai de référence du RRR pour les supraconducteurs Nb.