

INTERNATIONAL STANDARD



**Superconductivity –
Part 4: Residual resistance ratio measurement – Residual resistance ratio of
Nb-Ti and Nb₃Sn composite superconductors**

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Nb-Ti and Nb₃Sn composite superconductors**

INTERNATIONAL
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INTERNATIONAL ELECTROTECHNICAL COMMISSION

SUPERCONDUCTIVITY –**Part 4: Residual resistance ratio measurement –
Residual resistance ratio of Nb-Ti and Nb₃Sn
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International Standard IEC 61788-4 has been prepared by IEC technical committee 90: Superconductivity.

This fifth edition cancels and replaces the fourth edition published in 2016. This edition constitutes a technical revision.

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

- a) change in the suitable distance of voltage taps on the specimen for reliable measurement,
- b) new report on the result of the round robin test of the residual resistance ratio of Nb₃Sn superconductors that proves the validity of the measurement method in this standard,
- c) revision of the confusing definitions of the copper ratio and copper fraction.

The text of this standard is based on the following documents:

FDIS	Report on voting
90/448/FDIS	90/451/RVD

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

This publication has been drafted in accordance with the ISO/IEC Directives, Part 2.

A list of all parts of 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 publication will remain unchanged until the stability date indicated on the IEC website under "<http://webstore.iec.ch>" in the data related to the specific publication. At this date, the publication will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

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INTRODUCTION

Copper, Cu/Cu-Ni or aluminium is used as matrix material in ~~Nb-Ti~~ Ni-Ti and Nb₃Sn composite superconductors and works as an electrical shunt when the superconductivity is interrupted. It also contributes to recovery of the superconductivity by conducting heat generated in the superconductor to the surrounding coolant. The cryogenic-temperature resistivity of copper is an important quantity, which influences the stability and AC losses of the superconductor. The residual resistance ratio is defined as a ratio of the resistance of the superconductor at room temperature to that just above the superconducting transition.

This document specifies the test method for residual resistance ratio of Nb-Ti and Nb₃Sn composite superconductors. The curve method is employed for the measurement of the resistance just above the superconducting transition. Other methods are described in Clause A.3.

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SUPERCONDUCTIVITY –

Part 4: Residual resistance ratio measurement – Residual resistance ratio of Nb-Ti and Nb₃Sn composite superconductors

1 Scope

This part of IEC 61788 specifies a test method for the determination of the residual resistance ratio (RRR) of Nb-Ti and Nb₃Sn composite superconductors with Cu, Cu-Ni, Cu/Cu-Ni and Al matrix in a strain-free condition and zero external magnetic field. This method is intended for use with superconductor specimens that have a monolithic structure with rectangular or round cross-section, RRR value less than 350, and cross-sectional area less than 3 mm². In the case of Nb₃Sn, the specimens have received a reaction heat-treatment.

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 (IEV) – Part 815: Superconductivity* (available at: 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 terminological databases for use in standardization at the following addresses:

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

3.1

residual resistance ratio

RRR

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

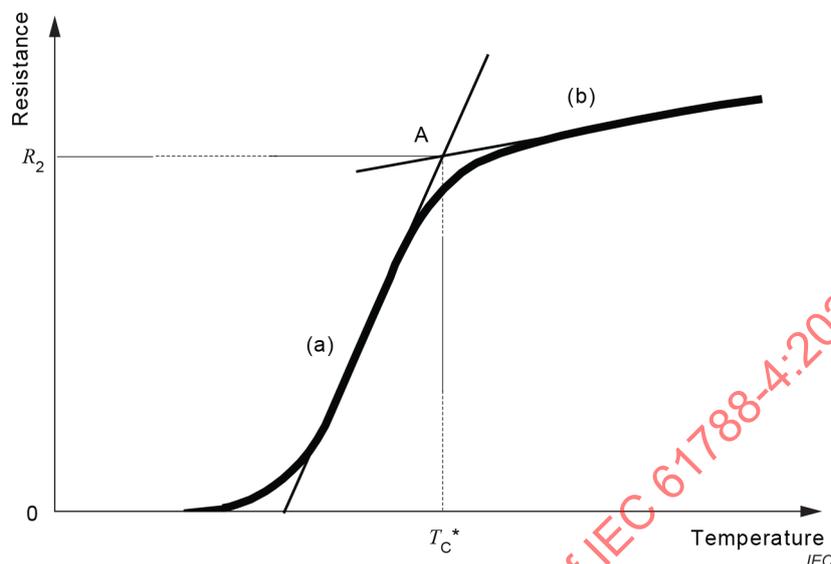
Note 1 to entry: This note applies to the French language only.

Note 2 to entry: In this document for Nb-Ti and Nb₃Sn composite superconductors, the room temperature is defined as 293 K (20 °C), and the residual resistance ratio is obtained in Formula (1), where the resistance (R_1) at 293 K is divided by the resistance (R_2) just above the superconducting transition.

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

Here r_{RRR} is a value of the residual resistance ratio, R_2 is a value of the resistance measured in a strain-free condition and zero external magnetic field.

Figure 1 shows schematically a resistance versus temperature curve acquired on a specimen while measuring the cryogenic resistance.–



The cryogenic resistance, R_2 , is determined by the intersection, A, of two straight lines (a) and (b) at temperature T_c^* .

Figure 1 – Relationship between temperature and resistance

4 Principle

The resistance measurement both at room and cryogenic temperatures shall be performed with the four-terminal technique. All measurements are done without an applied magnetic field.

The target relative combined standard uncertainty of this method is defined as an expanded uncertainty ($k=2$) not to exceed 5 %.

The maximum bending strain induced during mounting and cooling the Nb-Ti specimen shall not exceed 2 %. The measurement shall be conducted in a strain-free condition or in a condition with allowable thermal strain for the Nb₃Sn specimen.

5 Apparatus

5.1 Material of measurement mandrel or of measurement base plate

Material of the measurement mandrel for a coiled Nb-Ti specimen or of the measurement base plate for a straight Nb-Ti or Nb₃Sn specimen shall be copper, aluminium, silver, or the like whose thermal conductivity is equal to or better than 100 W/(m·K) at liquid helium temperature (4,2 K). The surface of the material shall be covered with an insulating layer (tape or a layer made of polyethylene terephthalate, polyester, polytetrafluoroethylene, etc.) whose thickness is 0,1 mm or less.

5.2 Diameter of the measurement mandrel and length of the measurement base plate

The diameter of the measurement mandrel shall be large enough to keep the bending strain of the specimen less than or equal to 2 % for the Nb-Ti specimen. The Nb₃Sn specimen on a base plate shall be measured in a strain-free condition or a condition with allowable thermal strain.

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

5.3 Cryostat for the resistance (R_2) measurement

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

6 Specimen preparation

The test specimen shall have no joints or splices with a length of 30 mm or longer. The specimen shall be instrumented with current contacts near each of its ends and a pair of voltage contacts over its central portion. The distance between two voltage taps (L) shall be ~~25~~ 15 mm or longer. A thermometer for measuring cryogenic temperature shall be attached near the specimen.

Some mechanical method shall be used to hold the specimen against the insulated layer of the measurement mandrel or base plate. Special care should be taken during instrumentation and installation of the specimen on the measurement mandrel or base plate so that no excessive force, which may cause undesired bending strain or tensile strain, would be applied to the specimen. Ideally, the Nb₃Sn specimen is intended to 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.

The specimen shall be mounted on a measurement mandrel or on a measurement base plate for these measurements. Both resistance measurements, R_1 and R_2 , shall be made on the same specimen and the same mounting.

7 Data acquisition and analysis

7.1 Resistance (R_1) at room temperature

The mounted specimen shall be measured at room temperature (T_m (K)), where T_m satisfies the following condition: $273 \text{ K} \leq T_m \leq 308 \text{ K}$. A specimen current (I_1 (A)) shall be applied so that the current density is in the range of 0,1 A/mm² to ~~1~~ 2 A/mm² based on the total wire cross-sectional area, and the resulting voltage (U_1 (V)), I_1 and T_m shall be recorded. Formula (2) below shall be used to calculate the resistance (R_m) at room temperature. The resistance (R_1) at 293 K (20 °C) shall be calculated using Formula (3) for a wire with Cu matrix. For wires that do not contain a pure Cu component, the value of R_1 shall be set equal to R_m , without any temperature correction.

$$R_m = \frac{U_1}{I_1} \quad (2)$$

$$R_1 = \frac{R_m}{\left[1 + 0,00393 \times (T_m - 293)\right]} \quad (3)$$

7.2 Resistance (R_2 or R_2^*) just above the superconducting transition

7.2.1 Correction of strain effect

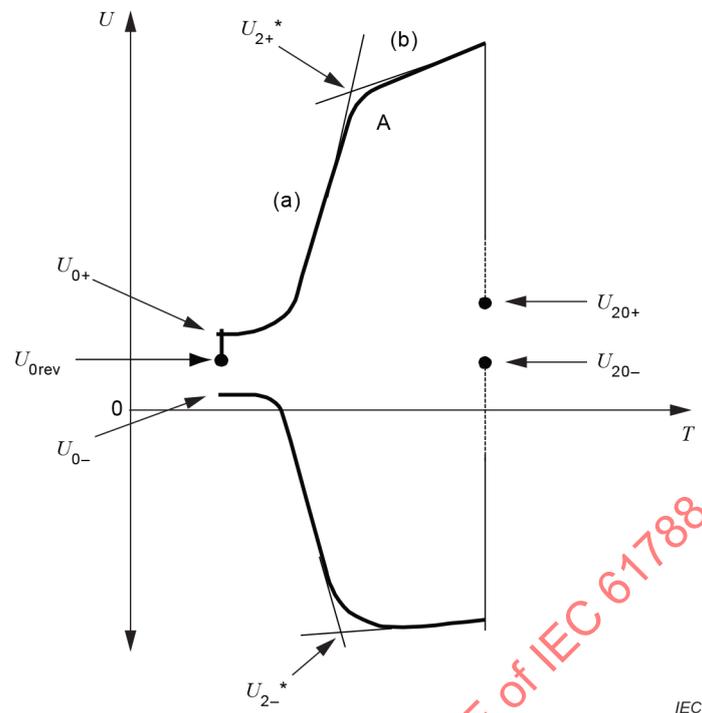
Under a strained condition of the Nb-Ti specimen, the measured cryogenic resistance, R_2^* , is not a correct value for R_2 . The corresponding correction of the strain effect is described in 7.3.

7.2.2 Data acquisition of cryogenic resistance

The specimen, which is still mounted as it was for the room temperature measurement, shall be placed in the cryostat for electrical measurement specified in 5.3. Horizontal mounting of the specimen is recommended in Clause A.1. Alternative cryostats that employ a heating element to sweep the specimen temperature are described in Clause A.2. The specimen shall be slowly lowered into the liquid helium bath and cooled to liquid helium temperature over a time period of at least 5 min.

During the acquisition phases of the low-temperature R_2^* measurements, a specimen current (I_2) shall be applied so that the current density is in the range 0,1 A/mm² to 10 A/mm² based on the total wire cross-sectional area, and the resulting voltage (U (V)), I_2 (A), and specimen temperature (T (K)) shall be recorded. In order to keep the ratio of signal to noise high enough, the measurement shall be carried out under the condition that the absolute value of the resulting voltage above the superconducting transition exceeds 10 μ V. An illustration of the data to be acquired and its analysis is shown in Figure 2.

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NOTE Voltages with subscripts + and – are those obtained in the first and second measurements under positive and negative currents, respectively, and U_{20+} and U_{20-} are those obtained at zero current. For clarity, U_{0rev} , measured at zero current is not shown coincident with U_{0-} . Straight line (a) is drawn in the transition region with a sharp increase in the voltage with temperature and straight line (b) is drawn in the region with a nearly constant voltage.

Figure 2 – Voltage versus temperature curves and definitions of each voltage

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

$$\frac{|U_{0+} - U_{0rev}|}{\bar{U}_2} < 1\% \quad (4)$$

where \bar{U}_2 is the average voltage for the specimen in the normal state at cryogenic temperature, which is defined by Formula (5).

The specimen shall be gradually warmed so that it changes to the normal state completely. When the cryostat for the resistance measurement specified in 5.3 is used, this can be achieved simply by raising the specimen to an appropriate position above the liquid helium level. The specimen voltage versus temperature curve shall be acquired with the rate of temperature increase maintained between 0,1 K/min and 10 K/min. The voltage versus temperature curve shall continue to be recorded during the transition into the normal state, up to a temperature somewhat less than 15 K for the Nb-Ti specimen and less than 25 K for the Nb₃Sn specimen. Then, the specimen current shall be decreased to zero and the corresponding voltage, U_{20+} ,

shall be recorded at a temperature below 15 K for the Nb-Ti specimen and below 25 K for the Nb₃Sn specimen.

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

Each of the two voltage versus temperature curves shall be analysed by drawing a line (a) through the data where the absolute value of voltage sharply increases with temperature (see Figure 2) and drawing a second line (b) through the data above the transition where the voltage is nearly constant for Nb-Ti or raised gradually and almost linearly for Nb₃Sn with temperature increase. U_{2+}^* and U_{2-}^* in Figure 2 shall be determined at the intersection of these two lines for the positive and negative polarity curves, respectively.

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

$$\bar{U}_2 = \frac{|U_{2+} - U_{2-}|}{2} \quad (5)$$

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

$$\frac{|\Delta_+ - \Delta_-|}{\bar{U}_2} < 3 \% \quad (6)$$

where Δ_+ and Δ_- are defined as $\Delta_+ = U_{20+} - U_{0+}$ and $\Delta_- = U_{20-} - U_{0-}$. If the R_2^* measurement does not meet the validity requirements in 7.2.2, specifically either in Formula (4) or (6), then improvement steps either in hardware or experimental operation shall be taken to meet these requirements before results are reported.

Formula (7) shall be used to calculate the measured resistance (R_2^*) just above the superconducting transition.

$$R_2^* = \frac{\bar{U}_2}{I_2} \quad (7)$$

7.2.3 Optional acquisition methods

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

7.3 Correction on measured R_2^* of Nb-Ti composite superconductor for bending strain

If there is no pure Cu component in the superconductor, then R_2 shall be set equal to R_2^* .

For a specimen with a pure Cu component, the bending strain shall be defined by $\varepsilon_b = 100 \times (h/r)$ (%), where h is a half of the specimen thickness for rectangular wires or a radius for round wires and r is the bending radius. If the bending strain is less than 0,3 %, then no correction is necessary, and R_2 shall be set equal to R_2^* .

If neither of the above two situations applies, then the resistance R_2 just above the superconducting transition under the strain-free condition shall be estimated by

$$R_2 = R_2^* - \Delta\rho \times \frac{L}{S_{\text{Cu}}} \quad (8)$$

where $\Delta\rho$ is defined below and S_{Cu} and L are defined in 8.4. The increase in the resistivity of pure copper at 4,2 K due to tensile strain, ε (%), is expressed by

$$\Delta\rho \text{ (}\Omega\text{m)} = 6,24 \times 10^{-12} \varepsilon - 5,11 \times 10^{-14} \varepsilon^2; \varepsilon \leq 2 \text{ \%} \quad (9)$$

The calculation of Formula (9) shall be carried out assuming that the equivalent tensile strain ε is $(1/2)\varepsilon_b$ and $(4/3 \pi)\varepsilon_b$ for rectangular and round wires, respectively. The bending strain dependency of residual resistance ratio for pure copper is described in Clause A.4.

7.4 Residual resistance ratio (RRR)

The RRR value shall be calculated using Formula (1).

8 Uncertainty and stability of the test method

8.1 Temperature

The room temperature shall be determined with a standard uncertainty not exceeding 0,6 K, while holding the specimen, which is mounted on the measurement mandrel or on the measurement base plate, at room temperature.

8.2 Voltage measurement

For the resistance measurement, the voltage signal shall be measured with a relative standard uncertainty not exceeding 0,3 %.

8.3 Current

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

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

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

8.4 Dimension

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

For correction of the bending strain effect in the case of the wire with pure Cu matrix, the cross-sectional area of Cu matrix (S_{Cu}) shall be determined using a nominal value of copper to non-copper ratio and nominal dimensions of the specimen. The wire diameter (d) and mandrel radius (R_d) shall be determined with relative standard uncertainty not exceeding 1 % and 3 %, respectively.

9 Test report

9.1 RRR value

The obtained RRR value (r_{RRR}) shall be reported as

$$r_{RRR} (1 \pm U_{re}) \quad (n = \dots), \quad (10)$$

where

U_{re} is the expanded relative uncertainty:

$$U_{re} = 2u_r \quad (k = 2)$$

where

u_r denotes the relative combined standard uncertainty,

k is a coverage factor, and

n is the sampling number.

It is desired that n be larger than 4 so that the normal distribution can be assumed for observed results to estimate the standard deviation. If n is not sufficiently large, a rectangular distribution shall be assumed.

9.2 Specimen

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

- a) manufacturer;
- b) classification and/or symbol;
- c) shape and area of the cross-section;
- d) dimensions of the cross-sectional area;
- e) number of filaments or subelements;
- f) diameter of the filaments or subelements;
- ~~g) Cu to Nb-Ti volume ratio, Cu-Ni to Nb-Ti volume ratio, or Cu, Cu-Ni to Nb-Ti volume ratio, or Al, Cu to Nb-Ti volume ratio or volume ratio among Cu-Ni, Cu, and Nb-Ti or among Al, Cu, and Nb-Ti for Nb-Ti specimen;~~
- g) for Nb-Ti specimen, the volume ratio of the following material to Nb-Ti:
 - Cu,
 - Cu-Ni,
 - Cu and Cu-Ni,
 - Al and Cu,
 or the following volume ratio:
 - Cu-Ni: Cu: Nb-Ti,

Al: Cu: Nb-Ti;

- h) Cu to non-Cu volume ratio for Nb₃Sn specimen;
- i) cross-sectional area of the Cu matrix (S_{Cu}).

9.3 Test conditions

9.3.1 Measurements of R_1 and R_2

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

- a) total length of the specimen;
- b) distance between the voltage measurement taps (L);
- c) length of each current contact;
- d) transport currents (I_1 and I_2);
- e) current densities (I_1 and I_2 divided by the nominal total wire cross-sectional area);
- f) voltages (U_1 , U_{0+} , U_{0rev} , U_{2+}^* , U_{20+} , U_{0-} , U_{2-}^* , U_{20-} and \bar{U}_2);
- g) resistances (R_m , R_1 , R_2^* and R_2);
- h) resistivities ($\rho_1 = (R_1 \times S_{Cu}) / L$ and $\rho_2 = (R_2 \times S_{Cu}) / L$);
- i) material, shape, and dimensions of the mandrel or the base plate;
- j) installation method of the specimen in the mandrel or the base plate;
- k) insulating material of the mandrel or the base plate.

9.3.2 Measurement of R_1

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

- a) temperature setting and holding method of the specimen;
- b) T_m : Temperature for measurement of R_m .

9.3.3 Measurement of R_2

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

- a) rate of increasing temperature;
- b) method of cooling down and heating up.

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 superconductors is given in Annex C.

Annex A (informative)

Additional information relating to the measurement of RRR

A.1 Recommendation on specimen mounting orientation

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

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

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

a) Heater method

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

b) ~~Adiabatic~~ Controlled methods

1) Adiabatic method

In this method, the cryostat holds a chamber in which the specimen, a sample holder, a heater and so on are contained. Before the chamber is immersed in the liquid helium bath, air inside the chamber is pumped out and helium gas is filled. Then, the chamber is immersed in the liquid helium bath and the specimen is cooled to a temperature ~~of 5 K or lower~~ 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.

2) Quasi-adiabatic method

In this method, the cryostat holds the specimen a certain distance above the liquid helium bath for the entire cryogenic measurement. A thermal anchor from the measurement mandrel or the measurement base plate to the liquid helium bath allows the specimen to be cooled to a temperature ~~of 5 K or lower~~ 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.

3) Refrigerator method

In this method, an electromechanical apparatus (a refrigerator) is used to cool the specimen, which is mounted on a measurement mandrel or a measurement base plate, to a temperature ~~of 6 K or lower~~ below the critical temperature. The specimen can be heated above the superconducting transition by a heater or by controlling the refrigerator power.

A.3 Alternative measurement methods of R_2 or R_2^*

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

a) Modified reference method

This is a simplified method with acquisition of only one voltage-temperature curve and is used only for Nb-Ti composite superconductors. The voltage of the specimen is measured in the superconducting state under a desired direction of current (I_2) and then with current in the opposite direction. These values are U_{0+} and U_{0rev} as shown in Figure A.1. The current is then changed back to the initial direction. After the transition to the normal state, the voltage is measured as U'_{2+} in a plateau region of the curve within about 4 K above the transition. Then the voltage is read under a zero current (U_{20}). The current direction is then reversed and the voltage is measured again (U'_{2-}). The cryogenic resistance is obtained from

$$R_2^* = \frac{\bar{U}_2}{I_2} \quad (\text{A.1})$$

with

$$\bar{U}_2 = \frac{|U'_{2+} - U'_{2-}|}{2} \quad (\text{A.2})$$

This approximately compensates for the effect of thermoelectric voltage. The following conditions should be fulfilled to ensure that the influence of the interfering voltage and the thermoelectric voltage shift on R_2^* measurement is not appreciably large:

$$\frac{|U_{0+} - U_{0rev}|}{\bar{U}_2} < 1\% \quad (\text{A.3})$$

$$\frac{|\Delta_{2+} - \Delta_{2-}|}{\bar{U}_2} < 3\% \quad (\text{A.4})$$

where Δ_{2+} and Δ_{2-} are defined by $\Delta_{2+} = |U'_{2+} - U_{20}|$ and $\Delta_{2-} = |U'_{2-} - U_{20}|$, respectively.

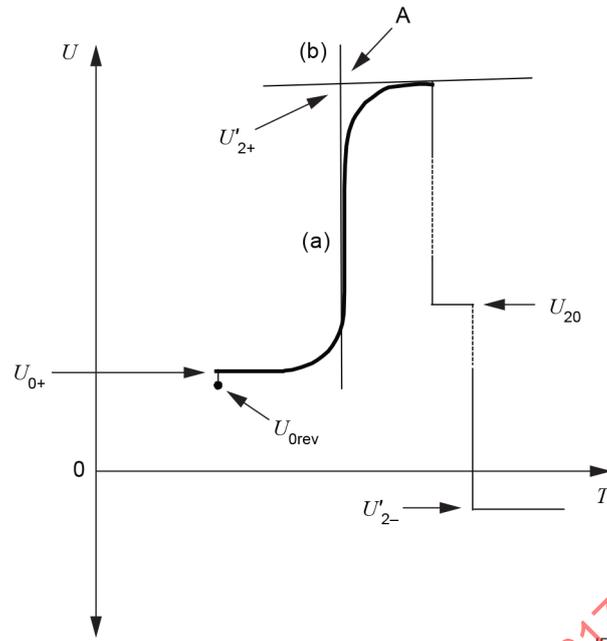


Figure A.1 – Definition of voltages

b) Measurement of voltage vs. time

Instead of measuring the voltage as a function of temperature, one can determine the cryogenic resistance from a voltage-versus-time curve that is continuously recorded both below and above the transition. Care should be taken not to re-cool the specimen without re-starting the acquisition of voltage-versus-time. The characteristic voltages, such as U_{2+}^* , can be similarly obtained from the intersection of the two straight lines drawn on the region of steepest slope during the transition and on the relatively flat region sufficiently above the transition in the voltage-versus-time curve. The analysis afterward to determine the cryogenic resistance is the same as in the reference method.

c) Fixed temperature method

In this method R_2 or R_2^* is directly determined at a fixed temperature in a plateau region within about 4 K above the transition for Nb-Ti composite superconductors, and R_2 is directly determined at 20 K for Nb₃Sn composite superconductors, instead of using the method described in 7.2. In this case it is desirable to check that the whole specimen is at a uniform and fixed temperature. In the measurement of Nb₃Sn composite superconductor the fixed temperature of 20 K should be determined with a combined standard uncertainty not exceeding 0,6 K. The fixed temperature and the combined standard uncertainty should be noted in the test report. Also the U_{0+} and U_{0-} , which are defined in 7.2.2, should be recorded as the zero voltage level in the fixed temperature method. In order to eliminate the influence of thermoelectric voltage, two voltage signals of the specimen, say U_{2+} and U_{2-} , should be acquired nearly simultaneously by reversal of the test current. For the fixed temperature method the effect of thermoelectric voltage on determination of cryogenic resistance can be eliminated.

d) Computer-based method

A computer can be used to control the current direction and warming of the specimen and to measure the voltage-temperature curve. Changes in current direction by periodic current reversals or periodic current on and off cycles are used to correct for offset voltages in order that the measurements can be made during one cycle of changing the specimen temperature. The effect of thermoelectric voltage should also be checked.

e) Other simplified methods with periodic checks

Simplified methods without temperature measurement might also be accepted, if an operator with sufficient experience performs the measurement using a given apparatus and if the

following condition is satisfied. If a simplified laboratory practice can be shown, through periodic checks, to achieve the same result as the method in this part of IEC 61788, within its stated uncertainty, then the simplified practice may be used in place of this reference method. These periodic checks could be accomplished by doing one of the following:

- 1) an interlaboratory comparison where one laboratory uses the reference method and another laboratory uses its own simplified method;
- 2) a single laboratory comparison where one laboratory "checks" their simplified method against the reference method;
- 3) periodic measurement of a small set of reference samples with well-known RRR values using the simplified method;
- 4) regular/frequent measurements with multiple specimens, one of which is a reference sample that would not be mounted/dismounted and would be measured every time as a calibrator.

A.4 Bending strain dependency of RRR for Nb-Ti composite superconductor

In general, the resistivity (ρ) of a pure metal such as copper at a very-low temperature increases as its applied strain increases. In general, a lower ρ wire has a larger percentage change in ρ than a higher ρ wire. There is almost no effect of strain on the room temperature resistivity of a metal. This means that the change in r_{RRR} with strain is more significant for a material whose r_{RRR} is high. According to the result of the intercomparison tests [1]¹, the dependency on bending strain was low for a specimen of low r_{RRR} . Bending strain is applied when the specimen is mounted on the measurement mandrel. Since the bending strain is inversely proportional to a radius of bent curvature, the smaller is the diameter of the measurement mandrel, the larger is the bending strain being applied to the specimen.

The increase in resistivity, $\Delta\rho$, at 4 K as a function of cold working ratio, r_{CW} [%], for pure copper is shown in Chapter 8 of reference [2]. Since the value of r_{CW} is approximately equal to the value of tensile strain, ε , when ε is small, the result is expressed as in Formula (9). The dependence of the copper resistivity increase on bending strain can be obtained by replacing the bending strain by an equivalent tensile strain.

Figure A.2 shows the relationship between r_{RRR} and bending strain for Nb-Ti composite superconductors with pure Cu matrix, obtained from the measured values of the intercomparison test performed in 1993 and 1994. The lines in the figure are the relationships calculated according to Formula (9) for each specimen. The measured values basically agree with the calculated values, and high r_{RRR} materials are sensitive to bending strain. Using Formula (9), Figure A.3 shows the dependency of round Cu wires where r_{RRR} with zero strain ($r_{RRR}(0)$) varies from 50 to 350. Figure A.4 shows bending strain dependency of r_{RRR} normalized by the value at zero strain. A similar dependency of rectangular Cu wires is shown in Figures A.5 and A.6. For copper with r_{RRR} of 350, which is the highest limit of r_{RRR} in this document, the r_{RRR} decreases by about 10 % for a bending strain of 2 %, with respect to the zero strain value.

¹ Numbers in square brackets refer to the Bibliography.

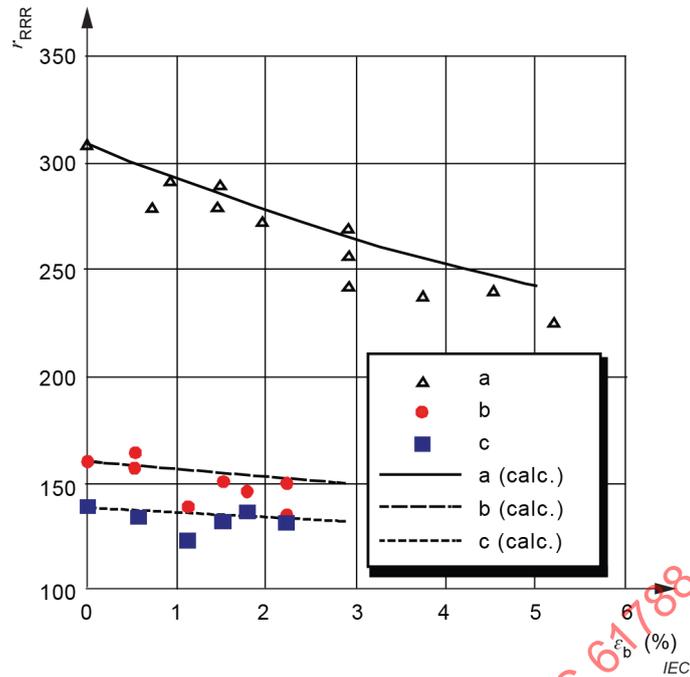


Figure A.2 – Bending strain dependency of RRR value for pure Cu matrix of Nb-Ti composite superconductors (comparison between measured values and calculated values)

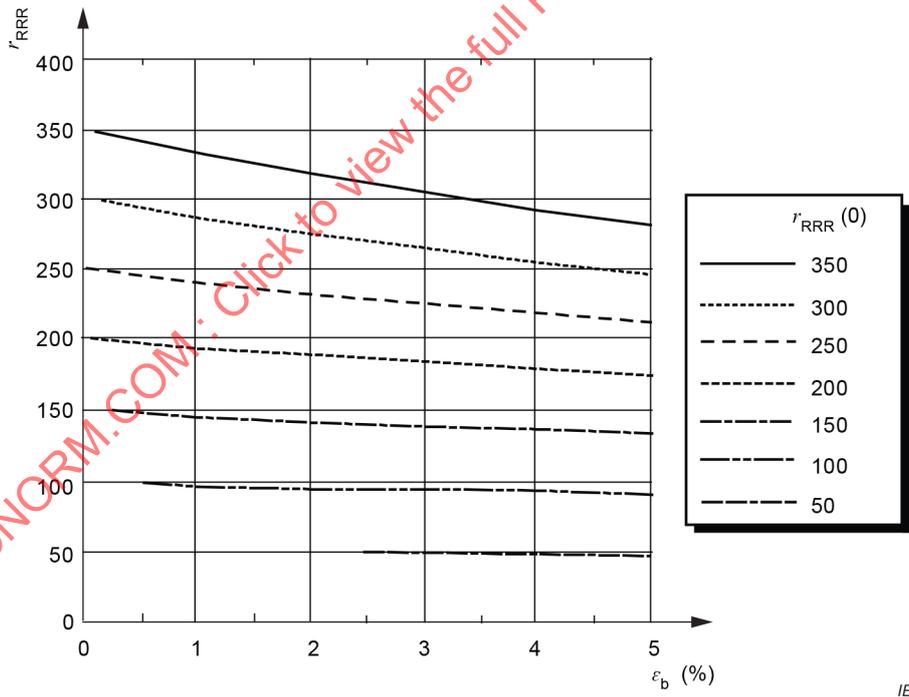


Figure A.3 – Bending strain dependency of RRR value for round Cu wires

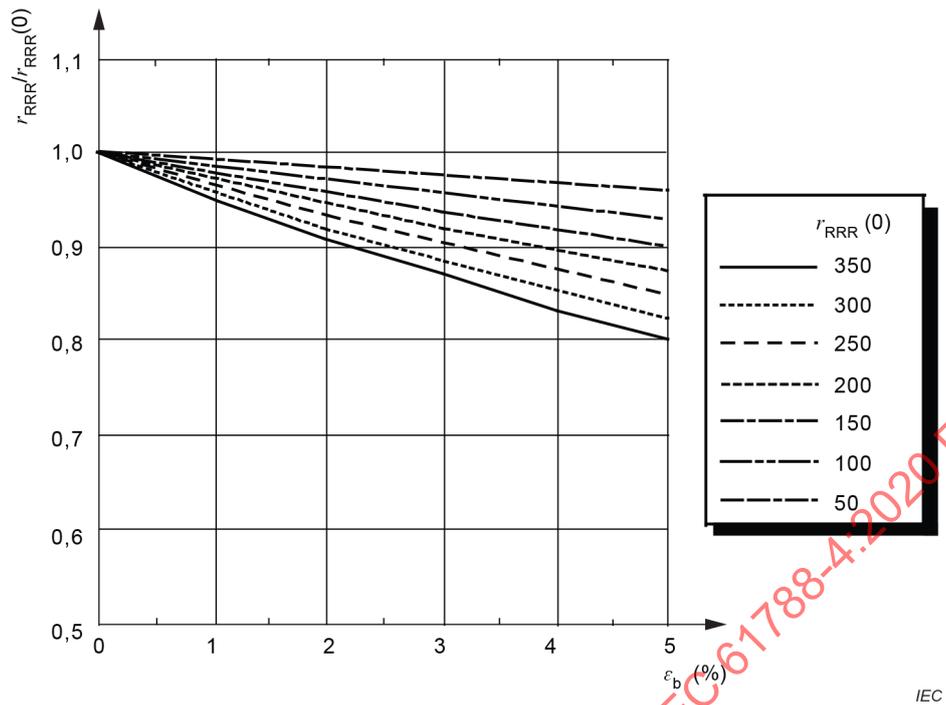


Figure A.4 – Bending strain dependency of normalized RRR value for round Cu wires

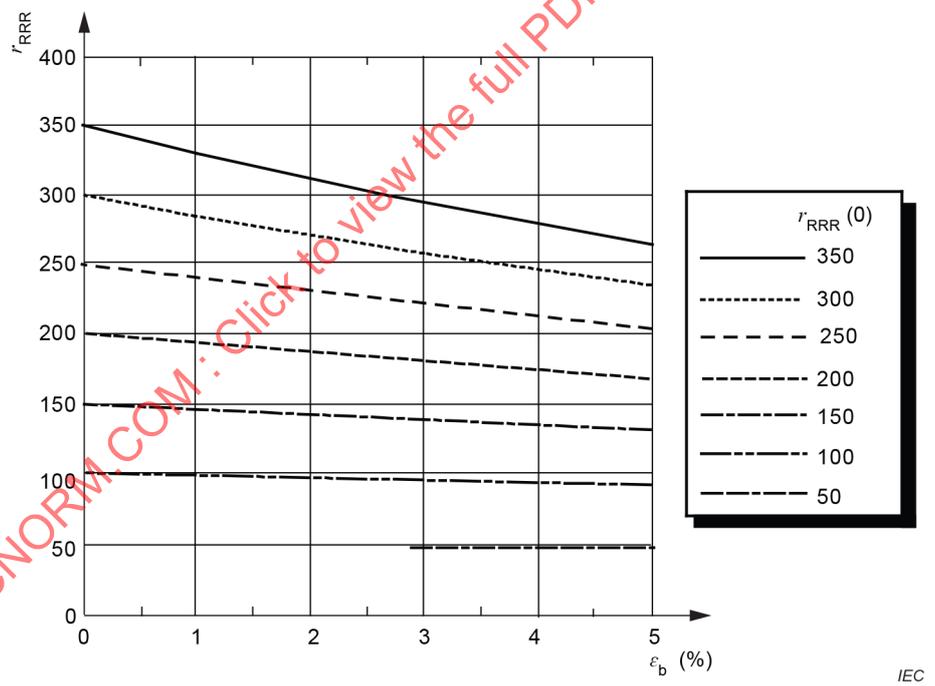


Figure A.5 – Bending strain dependency of RRR value for rectangular Cu wires

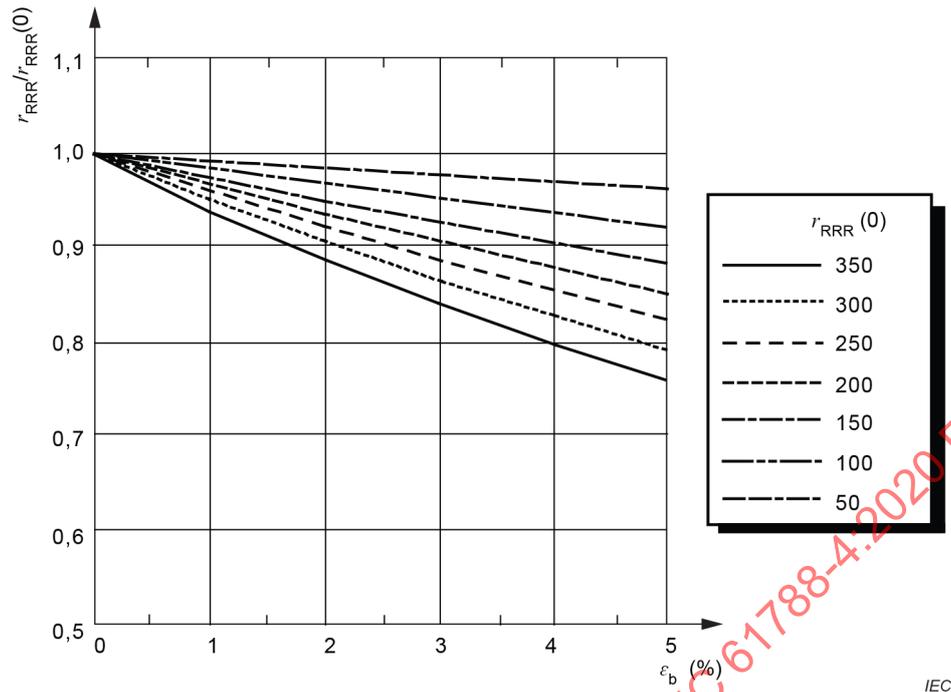


Figure A.6 – Bending strain dependency of normalized RRR value for rectangular Cu wires

To evaluate a high- r_{RRR} material, it is therefore desirable to use a straight base plate or a mandrel with a large coil diameter so that the evaluation can be performed with the least possible bending strain being applied. In addition to this, special care should be taken with the specimen so that there is no significant strain applied to it during handling.

The minimum diameters, $D_m d_{min}$, of the measurement mandrel for round and rectangular wires are listed in Table A.1 and Table A.2, respectively.

Table A.1 – Minimum diameter of the measurement mandrel for round wires

Wire diameter d [mm]	0,50	0,75	1,00	1,25	1,50
Minimum diameter $D_m d_{min}$ [mm]	10,6	15,9	21,2	26,5	31,8

Table A.2 – Minimum diameter of the measurement mandrel for rectangular wires

Thickness t [mm]	0,25	0,50	0,75	1,00
Minimum diameter $D_m d_{min}$ [mm]	6,3	12,5	18,8	25,0

A.5 Procedure of correction of bending strain effect

Clause A.5 describes the procedure of correction of bending strain effect on the resistance at low temperature given in 7.3. For a specimen of thickness $2h$ mounted on a mandrel of radius R_d , the bending strain is given by

$$\varepsilon_b = 100 \times (h / R_d) \% . \tag{A.5}$$

Then, the equivalent tensile strain is

$$\varepsilon = (1/2)\varepsilon_b \quad (\text{A.6})$$

for a rectangular wire and

$$\varepsilon = [4/(3\pi)]\varepsilon_b \quad (\text{A.7})$$

for a round wire. The increase in the resistivity of pure copper at 4,2 K is calculated by substituting this ε value into Formula (9). Then, the corrected resistance at low temperature is calculated using Formula (8).

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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 [3].

It was left to each Technical Committee to decide if they were going to change existing and future standards to be consistent with the new unified approach. Such change is not easy and creates additional confusion, especially for those who are not familiar with statistics and the term uncertainty. At the June 2006 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 have been 1, 2, 3, or some other number. A manufacturer’s specification that can sometimes be described by a rectangular distribution will lead to a conversion number of $1/\sqrt{3}$. The appropriate coverage factor was used when converting the original number to the equivalent standard uncertainty. The conversion process is not something that the user of the standard needs to address for compliance to 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 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 interlaboratory comparison.

B.2 Definitions

Statistical definitions can be found in three sources: ISO/IEC Guide 98-3:2008, ISO/IEC Guide 99:2007 [4], and the NIST Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results (NIST) [5]. 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 (5.1.6, Annex J), but they are not formally defined in ISO/IEC Guide 98-3:2008 (see [53]).

B.3 Consideration of the uncertainty concept

Statistical evaluations in the past frequently used the coefficient of variation (COV), which is the ratio of the standard deviation and the mean (N.B. the COV is often called the relative standard deviation). Such evaluations have been used to assess the precision of the measurements and give the closeness of repeated tests. The standard uncertainty (SU) 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. Tables B.2, B.3, B.4 and B.5 are 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 (\bar{X}) [V]	
E_1	E_2
0,001 190 19	2,334 411 62

$$\bar{X} = \frac{\sum_{i=1}^n X_i}{n} \quad [\text{V}] \quad (\text{B.1})$$

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

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

$$\sigma = \sqrt{\frac{1}{n-1} \cdot \sum_{i=1}^n (X_i - \bar{X})^2} \quad [\text{V}] \quad (\text{B.2})$$

Table B.4 – Standard uncertainties of two output signals

Standard uncertainty (<i>u</i>) [V]	
<i>E</i> ₁	<i>E</i> ₂
0,000 095 97	0,000 067 48

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

Table B.5 – COV values of two output signals

COV value [%]	
<i>E</i> ₁	<i>E</i> ₂
25,498 2	0,009 1

$$X_{\text{cov}} = \frac{\sigma}{X} \tag{B.4}$$

The standard uncertainty is very similar for the two extensometer deflections. In contrast, the COV value (X_{cov}) is nearly a factor of 2 800 different between the two data sets. This shows the advantage of using the standard uncertainty, which is independent of the mean value.

B.4 Uncertainty evaluation example for 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 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) The user derives in the first step a mathematical measurement model in the form of identified measurand as a function of all input quantities. A simple example of such model is given for the uncertainty of a force, F_{LC} measurement using a load cell:

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

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 mass/day and the reproducibility of checks on different days, respectively.

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

- b) The user should identify the type of distribution for each input quantity (e.g. Gaussian distributions for Type A measurements and rectangular distributions for Type B measurements).
- c) Evaluate the standard uncertainty of the Type A measurements:

$$u_A = \frac{\sigma}{\sqrt{n}}$$

where σ is the experimental standard deviation and n is the total number of measured data points.

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

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

where d_w is the range of rectangular distributed values.

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

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

In this case, it has been assumed that there is no correlation between input quantities. If the model equation has terms with products or quotients, the combined standard uncertainty is evaluated using partial derivatives and the relationship becomes more complex due to the sensitivity coefficients [6], [7].

- f) Optional – the combined standard uncertainty of the estimate of the referred measurand can be multiplied by a coverage factor (e.g. 1 for 68 % or 2 for 95 % or 3 for 99 %) to increase the probability that the measurand can be expected to lie within the interval.
- g) Report the result as the estimate of the measurand \pm the expanded uncertainty, together with the unit of measurement, and, at a minimum, state the coverage factor used to compute the expanded uncertainty and the estimated coverage probability.

To facilitate the computation and standardize the procedure, use of appropriate certified commercial software is a straightforward method that reduces the amount of routine work [8], [9]². In particular, the indicated partial derivatives can be easily obtained when such a software tool is used. Further references for the guidelines of measurement uncertainties are given in [5], [10], and [11].

² References [8] and [9] give example(s) of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by IEC of these products.

Annex C
(informative)

**Uncertainty evaluation in test method of RRR for
Nb-Ti and Nb₃Sn composite superconductors**

C.1 Evaluation of uncertainty

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

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

$$\frac{\Delta r_{RRR}}{r_{RRR}} = \frac{\Delta R_1}{R_1} - \frac{\Delta R_2}{R_2} \quad (C.1)$$

Hence, the relative standard uncertainty of r_{RRR} is

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

Since the room temperature resistance is given by

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

the deviation of R_1 is

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

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

$$\Delta T_m = \Delta T_{m1} + \Delta T_{m2} \quad [K] \quad (C.5)$$

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

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

where

u_{U1} [V] is the Type B uncertainty in the room temperature voltage ($u_{U1}/U_1 = 0,005/\sqrt{3}$),

u_{I1} [A] is the Type B uncertainty in the room temperature current ($u_{I1}/I_1 = 0,005/\sqrt{3}$),

u_{Tm2} [K] is the Type B uncertainty in the room temperature measurement using a bolometer ($u_{Tm2} = 1/\sqrt{3}$ [K]).

The u_{RTm1} [Ω] is the type B uncertainty in R_1 due to the difference of the room temperature from the specimen temperature and is formally expressed as $u_{RTm1} = 0,00393 I_1 u_{Tm1}$. However, u_{Tm1} is not obtained from a mathematical model but u_{Tm1} is directly estimated as $\pm 1,7\%$ of R_1 from the results of round robin testing on RRR of Nb-Ti [12]. Assuming a similar situation, it can also be assumed as $u_{RTm1}/R_1 = 0,017/\sqrt{3}$.

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

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

where u_{U2} [V] is the Type B uncertainty due to the voltmeter, and u_{I2} [A] is the Type B uncertainty in the current. In the above, $u_{U2}/U_2 = 0,005/\sqrt{3}$ and $u_{I2}/I_2 = 0,005/\sqrt{3}$. The first and second terms are doubled because the measurements are done twice. Hence, when the sample is measured in a bending-free condition, the relative combined standard uncertainty is given by

$$u_r = \left[1,43 \times 10^{-4} + 2 \left(\frac{b}{R_2} \right)^2 \right]^{1/2}. \quad (C.8)$$

When the sample current is measured using a voltmeter and a standard resistor, the uncertainties of the voltage and resistance affect the uncertainty of measurement. If the value of the voltage and its standard uncertainty are U and u_U , and if the value of the resistance and its standard uncertainty are R and u_R , $(U_1/I_1^2)^2 u_{I1}^2$ in Formula (C.6) and $(U_2/I_2^2)^2 u_{I2}^2$ in Formula (C.7) are respectively replaced by

$$\left(\frac{U_1}{I_1} \right)^2 \left(\frac{u_U^2}{U^2} + \frac{u_R^2}{R^2} \right), \quad \left(\frac{U_2}{I_2} \right)^2 \left(\frac{u_U^2}{U^2} + \frac{u_R^2}{R^2} \right). \quad (C.9)$$

When the cryogenic resistance is measured in a bent condition, the result needs to be compensated for the strain effect using the given equation with the distance between the two voltage taps (L), the diameter (d), copper ~~ratio~~ fraction (r_{Cu}) and the radius of a mandrel (R_d) used for the measurement. We assume that a round wire of diameter d is wound on a measurement mandrel of radius R_d . With the aid of Formulae (8) and (9) the value of the compensated cryogenic resistance is given by

$$\begin{aligned} R_2 &= R_2^* - 6,24 \times 10^{-12} \frac{8}{3\pi^2} \frac{L}{d r_{Cu} R_d} \\ &= R_2^* - 1,69 \times 10^{-12} \frac{L}{d r_{Cu} R_d} \quad [\Omega], \end{aligned} \quad (C.10)$$

where we have used $\varepsilon = (4/3\pi)(d/2R_d)$ and $S_{Cu} = \pi(d/2)^2 r_{Cu}$, and the small second term in Formula (9) was neglected. The quantity r_{Cu} is ~~a ratio that copper occupies in a cross-sectional area of the wire and can be~~ given by $r_{Cu} = c/(1+c)$ using the copper ratio, c . If the second term in Formula (C.10) is denoted by δR_2 , the contribution to the combined standard uncertainty of u_{R2} from the uncertainties of L , d , r_{Cu} and R_d is estimated as

$$u_{R2}^* = |\delta R_2| \left[\left(\frac{u_L}{L} \right)^2 + \left(\frac{u_d}{d} \right)^2 + \left(\frac{u_{rCu}}{r_{Cu}} \right)^2 + \left(\frac{u_{Rd}}{R_d} \right)^2 \right]^{1/2} \quad [\Omega], \quad (C.11)$$

where u_L [m], u_d [m], u_{rCu} and u_{Rd} [m] are the Type B standard uncertainties of distance between voltage taps, diameter, copper ~~ratio~~ fraction and radius of mandrel, respectively. L is required to be measured within the uncertainty $u_L/L = 0,05/\sqrt{3}$. It is assumed that the uncertainty of d is $u_d/d = 0,02/\sqrt{3}$. The relative uncertainties of r_{Cu} and R_d are required to be smaller than $0,05/\sqrt{3}$. The maximum compensation is about $\delta R_2/R_2 = 0,10$ when the bending strain is 2 % for $r_{RRR} = 350$. Hence, the relative combined standard uncertainty of cryogenic resistance due to the bending strain correction is estimated at most to be

$$\frac{u_{R2}^*}{R_2} = 0,513 \times 10^{-2}. \quad (C.12)$$

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

$$u_r = \frac{u}{(R_1/R_2)} = \left[\left(\frac{u_{R1}}{R_1} \right)^2 + \left(\frac{u_{R2}}{R_2} \right)^2 + \left(\frac{u_{R2}^*}{R_2} \right)^2 \right]^{1/2} = \left[1,69 \times 10^{-4} + 2 \left(\frac{b}{R_2} \right)^2 \right]^{1/2}. \quad (C.13)$$

According to the round robin test shown in Clause C.2, u_r was estimated as $2,44 \times 10^{-2}$. Thus, b/R_2 is estimated as

$$\frac{b}{R_2} = 1,46 \times 10^{-2}. \quad (C.14)$$

The type and target value of uncertainty are listed for each measurement in Table C.1.

Table C.1 – Uncertainty of each measurement

Uncertainty	Type	Value	Remarks
u_{U_1}/U_1	B	$0,005/\sqrt{3}$	$ \Delta U_1 /U_1 < 0,005$
u_{I_1}/I_1	B	$0,005/\sqrt{3}$	$ \Delta I_1 /I_1 < 0,005$
$u_{T_{m2}}$	B	$1/\sqrt{3}$ K	$ \Delta T_m < 1$ K
u_{U_2}/U_2	B	$0,005/\sqrt{3}$	$ \Delta U_2 /U_2 < 0,005$
u_{I_2}/I_2	B	$0,005/\sqrt{3}$	$ \Delta I_2 /I_2 < 0,005$
u_L/L	B	$0,05/\sqrt{3}$	$ \Delta L /L < 0,05$
u_d/d	B	$0,02/\sqrt{3}$	$ \Delta d /d < 0,02$
$u_{r_{Cu}}/r_{Cu}$	B	$0,05/\sqrt{3}$	$ \Delta r_{Cu} /r_{Cu} < 0,05$
u_{R_d}/R_d	B	$0,05/\sqrt{3}$	$ \Delta R_d /R_d < 0,05$

C.2 Summary of round robin test of RRR of a Nb-Ti composite superconductor

The round robin test of RRR was carried out on a Cu/Nb-Ti composite superconductor. The specifications of the test superconductor are:

- diameter: 0,80 mm, 0,86 mm including insulating layer;
- Cu/Nb-Ti ratio: 6,5;
- mean filament diameter: about 70 μm ;
- number of filaments: 16;
- twist pitch: 30 mm;
- critical current: more than 185 A (3 T, 4,2 K);
- r_{RRR} : more than 150.

Participating institutes were provided with specimens that were nearly straight. Some specimens were measured in the as-received condition and some were measured wound on a bobbin under a strained condition. The number of participating institutes was 13 from five countries and the number of determinations was 77. R_2 was measured following the method defined in 7.2 and 7.3, and those in Clause A.3. The details of the measurements are described in reference [12]. The effect of the strain was corrected using Formulae (8) and (9). The distribution of the measured r_{RRR} is shown in Figure C.1. Almost all of the data, except for three, were concentrated fairly sharply. The average was 178,5, the standard deviation was 4,4 and the COV value was 2,44 %. If the three extraordinary data are omitted, the average was 178,2, the standard deviation was 3,1 and the COV value was 1,73 %.

Hence, it is reasonable to define the target relative combined standard uncertainty of this method not to exceed 2,5 % based on the COV value in the round robin test.

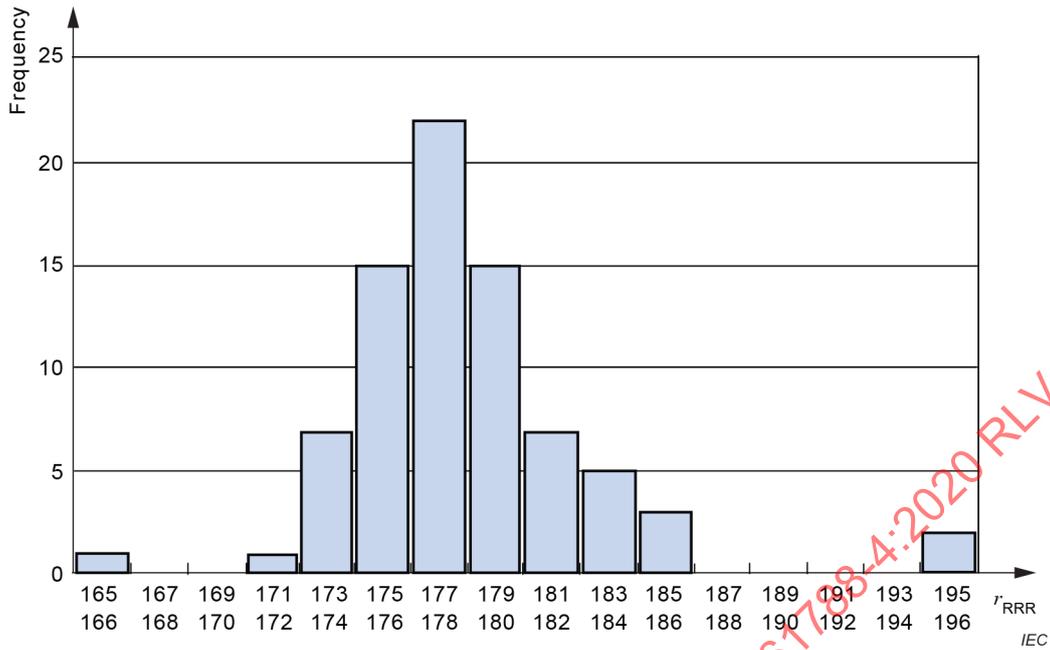


Figure C.1 – Distribution of observed r_{RRR} of Cu/Nb-Ti composite superconductor

C.3 Reason for large COV value in the intercomparison test on Nb₃Sn composite superconductor

The COV value of the intercomparison test for Nb₃Sn samples was 6,07 % [13]. This value is much larger than that for Nb-Ti (2,44 %), although there is no contribution from additional uncertainty in correction of the strain effect. For clarification of this reason an intercomparison test was performed between two laboratories for three Nb₃Sn samples, two of which were cut from the same batch of heat treatment. The r_{RRR} obtained using the reference method agreed within 1 % between the two laboratories for the three samples as shown in Table C.2, while the r_{RRR} values were different between the two samples obtained from the same batch [14]. This indicates that the large COV value in the former intercomparison test originated from inhomogeneity of samples, while the test method itself was fairly accurate. This inhomogeneity may be due to the high sensitivity to heat treatment conditions or due to defects of the diffusion barrier. Since a r_{RRR} value is commonly required to be greater than a minimum value in order to pass, the existence of inhomogeneities may require that several specimens of a given wire be measured and reported.

Table C.2 – Obtained values of R_1 , R_2 and r_{RRR} for three Nb₃Sn samples

Sample	Laboratory 1			Laboratory 2		
	$R_1(293\text{K}) [\Omega]$	$R_2(T_e^*) [\Omega]$	r_{RRR}	$R_1(293\text{K}) [\Omega]$	$R_2(T_e^*) [\Omega]$	r_{RRR}
B	$1,593 \times 10^{-3}$	$1,49 \times 10^{-5}$	107	$1,61 \times 10^{-3}$	$1,49 \times 10^{-5}$	108
C	$1,719 \times 10^{-3}$	$1,66 \times 10^{-5}$	104	$1,74 \times 10^{-3}$	$1,66 \times 10^{-5}$	105
D	$1,619 \times 10^{-3}$	$1,61 \times 10^{-5}$	100	$1,65 \times 10^{-3}$	$1,62 \times 10^{-5}$	101

For this reason the uncertainty in the test method of RRR for Nb₃Sn is expected to be as low as that for Nb-Ti. Therefore, the value of $b/R_2 = 1,46 \times 10^{-2}$ obtained in the intercomparison test for RRR measurement in Nb-Ti can also be used to estimate the uncertainty of r_{RRR} in Nb₃Sn with Formula (C.8). In addition, the result shown in Table C.2 indicates that the main

~~difference between the measurements in the two laboratories comes from the observed values of R_1 . This is considered to be caused by the uncertainty in the room temperature.~~

For clarification of the reason for the large COV a round robin test was performed. Since inhomogeneity in Nb₃Sn samples is anticipated as one of the reasons, real round robin tests were examined by measuring one specimen successively by participating institutions. The measured wires were two internal tin-processed Nb₃Sn composite superconductors. Three samples were cut from different positions of each wire. The number of participating institutions was six and the measurement method described in this document was employed. Each sample was successively measured seven times including one measurement C^a using the voltage vs. time method described in Clause A.3 b). All measured RRR values for six samples are shown in four digits for the analysis of the uncertainty in Table C.2, whereas significant digits are three. Table C.3 shows the average, standard deviation and COV.

Table C.2 – Obtained values of RRR for six Nb₃Sn specimens

Institution	r_{RRR}					
	#1-1	#1-2	#1-3	#2-1	#2-2	#2-3
A	152,7	155,7	158,2	182,2	177,7	181,3
B	152,5	159,4	158,0	189,1	173,4	183,0
C	148,6	153,4	154,3	175,8	168,1	177,9
D	150,0	153,4	150,6	174,8	156,7	173,0
E	143,2	153,4	153,3	178,6	164,8	177,8
F	148,0	156,3	157,3	181,6	171,3	181,6
C ^a	144,6	151,4	153,8	178,6	168,3	178,1

^a The voltage-time curve is used for determination of the cryogenic resistance. Although the significant digits of observed RRR value are three, four digit numbers are listed for the analysis of uncertainty.

Table C.3 – Average, standard deviation and coefficient of variation for six specimens

	#1-1	#1-2	#1-3	#2-1	#2-2	#2-3
Average	148,5	154,7	155,1	180,1	168,6	179,0
σ	3,6	2,6	2,8	4,8	6,7	3,4
COV (%)	2,5	1,7	1,8	2,7	4,0	1,9

The obtained COV for Nb₃Sn is ranged 1,70 % to 3,98 % and comparable to that for Nb-Ti, 2,44 %. If the result on specimen #2-2 with the largest COV value is disregarded, it is comparable to or even better than that for Nb-Ti. The uncertainty of RRR in Nb₃Sn is mostly attributed to that of measurement of the cryogenic resistance [14], which is largely different from the case of Nb-Ti. The difficulty to fit a straight line to non-linear voltage vs. temperature curves may be a reason for the large uncertainty. This round robin test clarified that Nb₃Sn composite superconductors are somewhat inhomogeneous along their length. This may be caused by the high sensitivity to heat treatment conditions or to random pores or breaks in the diffusion barrier. For this reason it is recommended to measure several specimens of a given superconductor to evaluate the distribution of the RRR value.

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INTERNATIONAL STANDARD

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**Superconductivity –
Part 4: Residual resistance ratio measurement – Residual resistance ratio of
Nb-Ti and Nb₃Sn composite superconductors**

**Supraconductivité –
Partie 4: Mesurage du rapport de résistance résiduelle – Rapport de résistance
résiduelle des composites supraconducteurs de Nb-Ti et de Nb₃Sn**

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

SUPERCONDUCTIVITY –**Part 4: Residual resistance ratio measurement –
Residual resistance ratio of Nb-Ti and Nb₃Sn
composite superconductors**

FOREWORD

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International Standard IEC 61788-4 has been prepared by IEC technical committee 90: Superconductivity.

This fifth edition cancels and replaces the fourth edition published in 2016. This edition constitutes a technical revision.

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

- a) change in the suitable distance of voltage taps on the specimen for reliable measurement,
- b) new report on the result of the round robin test of the residual resistance ratio of Nb₃Sn superconductors that proves the validity of the measurement method in this standard,
- c) revision of the confusing definitions of the copper ratio and copper fraction.

The text of this standard is based on the following documents:

FDIS	Report on voting
90/448/FDIS	90/451/RVD

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

This publication has been drafted in accordance with the ISO/IEC Directives, Part 2.

A list of all parts of 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 publication will remain unchanged until the stability date indicated on the IEC website under "<http://webstore.iec.ch>" in the data related to the specific publication. At this date, the publication will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

IMPORTANT – The 'colour inside' logo on the cover page of this publication indicates that it contains colours which are considered to be useful for the correct understanding of its contents. Users should therefore print this document using a colour printer.

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INTRODUCTION

Copper, Cu/Cu-Ni or aluminium is used as matrix material in Ni-Ti and Nb₃Sn composite superconductors and works as an electrical shunt when the superconductivity is interrupted. It also contributes to recovery of the superconductivity by conducting heat generated in the superconductor to the surrounding coolant. The cryogenic-temperature resistivity of copper is an important quantity, which influences the stability and AC losses of the superconductor. The residual resistance ratio is defined as a ratio of the resistance of the superconductor at room temperature to that just above the superconducting transition.

This document specifies the test method for residual resistance ratio of Nb-Ti and Nb₃Sn composite superconductors. The curve method is employed for the measurement of the resistance just above the superconducting transition. Other methods are described in Clause A.3.

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SUPERCONDUCTIVITY –

Part 4: Residual resistance ratio measurement – Residual resistance ratio of Nb-Ti and Nb₃Sn composite superconductors

1 Scope

This part of IEC 61788 specifies a test method for the determination of the residual resistance ratio (RRR) of Nb-Ti and Nb₃Sn composite superconductors with Cu, Cu-Ni, Cu/Cu-Ni and Al matrix in a strain-free condition and zero external magnetic field. This method is intended for use with superconductor specimens that have a monolithic structure with rectangular or round cross-section, RRR value less than 350, and cross-sectional area less than 3 mm². In the case of Nb₃Sn, the specimens have received a reaction heat-treatment.

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 (IEV) – Part 815: Superconductivity* (available at: 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 terminological databases for use in standardization at the following addresses:

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

3.1 residual resistance ratio RRR

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

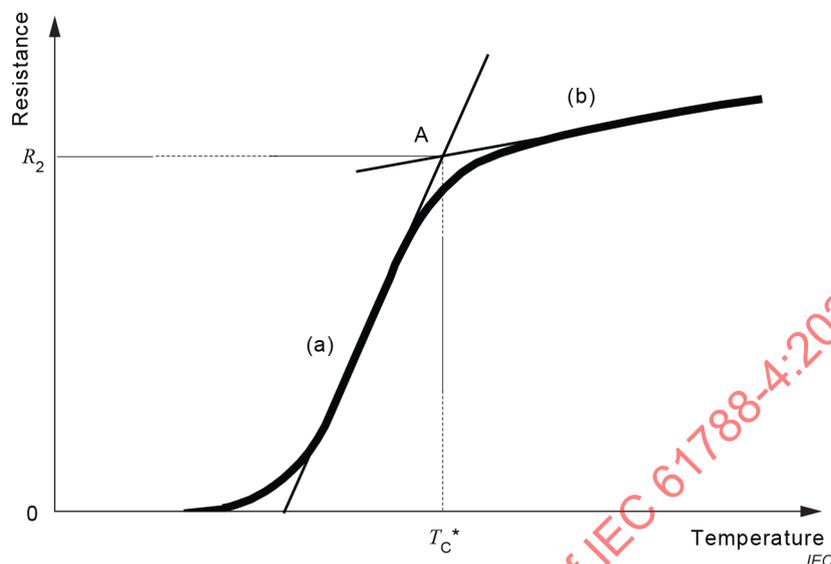
Note 1 to entry: This note applies to the French language only.

Note 2 to entry: In this document for Nb-Ti and Nb₃Sn composite superconductors, the room temperature is defined as 293 K (20 °C), and the residual resistance ratio is obtained in Formula (1), where the resistance (R_1) at 293 K is divided by the resistance (R_2) just above the superconducting transition.

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

Here r_{RRR} is a value of the residual resistance ratio, R_2 is a value of the resistance measured in a strain-free condition and zero external magnetic field.

Figure 1 shows schematically a resistance versus temperature curve acquired on a specimen while measuring the cryogenic resistance.–



The cryogenic resistance, R_2 , is determined by the intersection, A, of two straight lines (a) and (b) at temperature T_c^* .

Figure 1 – Relationship between temperature and resistance

4 Principle

The resistance measurement both at room and cryogenic temperatures shall be performed with the four-terminal technique. All measurements are done without an applied magnetic field.

The target relative combined standard uncertainty of this method is defined as an expanded uncertainty ($k = 2$) not to exceed 5 %.

The maximum bending strain induced during mounting and cooling the Nb-Ti specimen shall not exceed 2 %. The measurement shall be conducted in a strain-free condition or in a condition with allowable thermal strain for the Nb₃Sn specimen.

5 Apparatus

5.1 Material of measurement mandrel or of measurement base plate

Material of the measurement mandrel for a coiled Nb-Ti specimen or of the measurement base plate for a straight Nb-Ti or Nb₃Sn specimen shall be copper, aluminium, silver, or the like whose thermal conductivity is equal to or better than 100 W/(m·K) at liquid helium temperature (4,2 K). The surface of the material shall be covered with an insulating layer (tape or a layer made of polyethylene terephthalate, polyester, polytetrafluoroethylene, etc.) whose thickness is 0,1 mm or less.

5.2 Diameter of the measurement mandrel and length of the measurement base plate

The diameter of the measurement mandrel shall be large enough to keep the bending strain of the specimen less than or equal to 2 % for the Nb-Ti specimen. The Nb₃Sn specimen on a base plate shall be measured in a strain-free condition or a condition with allowable thermal strain.

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

5.3 Cryostat for the resistance (R_2) measurement

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

6 Specimen preparation

The test specimen shall have no joints or splices with a length of 30 mm or longer. The specimen shall be instrumented with current contacts near each of its ends and a pair of voltage contacts over its central portion. The distance between two voltage taps (L) shall be 15 mm or longer. A thermometer for measuring cryogenic temperature shall be attached near the specimen.

Some mechanical method shall be used to hold the specimen against the insulated layer of the measurement mandrel or base plate. Special care should be taken during instrumentation and installation of the specimen on the measurement mandrel or base plate so that no excessive force, which may cause undesired bending strain or tensile strain, would be applied to the specimen. Ideally, the Nb₃Sn specimen is intended to 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.

The specimen shall be mounted on a measurement mandrel or on a measurement base plate for these measurements. Both resistance measurements, R_1 and R_2 , shall be made on the same specimen and the same mounting.

7 Data acquisition and analysis

7.1 Resistance (R_1) at room temperature

The mounted specimen shall be measured at room temperature (T_m (K)), where T_m satisfies the following condition: $273 \text{ K} \leq T_m \leq 308 \text{ K}$. A specimen current (I_1 (A)) shall be applied so that the current density is in the range of 0,1 A/mm² to 2 A/mm² based on the total wire cross-sectional area, and the resulting voltage (U_1 (V)), I_1 and T_m shall be recorded. Formula (2) below shall be used to calculate the resistance (R_m) at room temperature. The resistance (R_1) at 293 K (20 °C) shall be calculated using Formula (3) for a wire with Cu matrix. For wires that do not contain a pure Cu component, the value of R_1 shall be set equal to R_m , without any temperature correction.

$$R_m = \frac{U_1}{I_1} \quad (2)$$

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

7.2 Resistance (R_2 or R_2^*) just above the superconducting transition

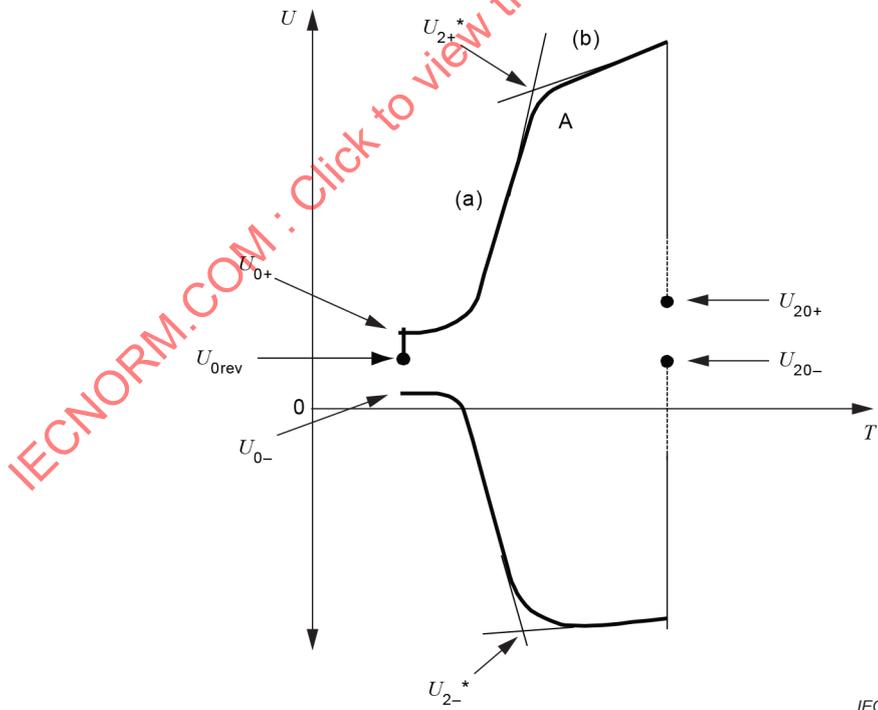
7.2.1 Correction of strain effect

Under a strained condition of the Nb-Ti specimen, the measured cryogenic resistance, R_2^* , is not a correct value for R_2 . The corresponding correction of the strain effect is described in 7.3.

7.2.2 Data acquisition of cryogenic resistance

The specimen, which is still mounted as it was for the room temperature measurement, shall be placed in the cryostat for electrical measurement specified in 5.3. Horizontal mounting of the specimen is recommended in Clause A.1. Alternative cryostats that employ a heating element to sweep the specimen temperature are described in Clause A.2. The specimen shall be slowly lowered into the liquid helium bath and cooled to liquid helium temperature over a time period of at least 5 min.

During the acquisition phases of the low-temperature R_2^* measurements, a specimen current (I_2) shall be applied so that the current density is in the range 0,1 A/mm² to 10 A/mm² based on the total wire cross-sectional area, and the resulting voltage (U (V)), I_2 (A), and specimen temperature (T (K)) shall be recorded. In order to keep the ratio of signal to noise high enough, the measurement shall be carried out under the condition that the absolute value of the resulting voltage above the superconducting transition exceeds 10 μ V. An illustration of the data to be acquired and its analysis is shown in Figure 2.



NOTE Voltages with subscripts + and - are those obtained in the first and second measurements under positive and negative currents, respectively, and U_{20+} and U_{20-} are those obtained at zero current. For clarity, U_{0rev} , measured at zero current is not shown coincident with U_{0-} . Straight line (a) is drawn in the transition region with a sharp increase in the voltage with temperature and straight line (b) is drawn in the region with a nearly constant voltage.

Figure 2 – Voltage versus temperature curves and definitions of each voltage

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

$$\frac{|U_{0+} - U_{0rev}|}{\bar{U}_2} < 1\% \quad (4)$$

where \bar{U}_2 is the average voltage for the specimen in the normal state at cryogenic temperature, which is defined by Formula (5).

The specimen shall be gradually warmed so that it changes to the normal state completely. When the cryostat for the resistance measurement specified in 5.3 is used, this can be achieved simply by raising the specimen to an appropriate position above the liquid helium level. The specimen voltage versus temperature curve shall be acquired with the rate of temperature increase maintained between 0,1 K/min and 10 K/min. The voltage versus temperature curve shall continue to be recorded during the transition into the normal state, up to a temperature somewhat less than 15 K for the Nb-Ti specimen and less than 25 K for the Nb₃Sn specimen. Then, the specimen current shall be decreased to zero and the corresponding voltage, U_{20+} , shall be recorded at a temperature below 15 K for the Nb-Ti specimen and below 25 K for the Nb₃Sn specimen.

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

Each of the two voltage versus temperature curves shall be analysed by drawing a line (a) through the data where the absolute value of voltage sharply increases with temperature (see Figure 2) and drawing a second line (b) through the data above the transition where the voltage is nearly constant for Nb-Ti or raised gradually and almost linearly for Nb₃Sn with temperature increase. U_{2+}^* and U_{2-}^* in Figure 2 shall be determined at the intersection of these two lines for the positive and negative polarity curves, respectively.

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

$$\bar{U}_2 = \frac{|U_{2+} - U_{2-}|}{2} \quad (5)$$

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

$$\frac{|\Delta_+ - \Delta_-|}{\bar{U}_2} < 3 \% \quad (6)$$

where Δ_+ and Δ_- are defined as $\Delta_+ = U_{20+} - U_{0+}$ and $\Delta_- = U_{20-} - U_{0-}$. If the R_2^* measurement does not meet the validity requirements in 7.2.2, specifically either in Formula (4) or (6), then improvement steps either in hardware or experimental operation shall be taken to meet these requirements before results are reported.

Formula (7) shall be used to calculate the measured resistance (R_2^*) just above the superconducting transition.

$$R_2^* = \frac{\bar{U}_2}{I_2} \quad (7)$$

7.2.3 Optional acquisition methods

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

7.3 Correction on measured R_2^* of Nb-Ti composite superconductor for bending strain

If there is no pure Cu component in the superconductor, then R_2 shall be set equal to R_2^* .

For a specimen with a pure Cu component, the bending strain shall be defined by $\varepsilon_b = 100 \times (h/r)$ (%), where h is a half of the specimen thickness for rectangular wires or a radius for round wires and r is the bending radius. If the bending strain is less than 0,3 %, then no correction is necessary, and R_2 shall be set equal to R_2^* .

If neither of the above two situations applies, then the resistance R_2 just above the superconducting transition under the strain-free condition shall be estimated by

$$R_2 = R_2^* - \Delta\rho \times \frac{L}{S_{Cu}} \quad (8)$$

where $\Delta\rho$ is defined below and S_{Cu} and L are defined in 8.4. The increase in the resistivity of pure copper at 4,2 K due to tensile strain, ε (%), is expressed by

$$\Delta\rho (\Omega m) = 6,24 \times 10^{-12} \varepsilon - 5,11 \times 10^{-14} \varepsilon^2; \varepsilon \leq 2 \% \quad (9)$$

The calculation of Formula (9) shall be carried out assuming that the equivalent tensile strain ε is $(1/2)\varepsilon_b$ and $(4/3 \pi)\varepsilon_b$ for rectangular and round wires, respectively. The bending strain dependency of residual resistance ratio for pure copper is described in Clause A.4.

7.4 Residual resistance ratio (RRR)

The RRR value shall be calculated using Formula (1).

8 Uncertainty and stability of the test method

8.1 Temperature

The room temperature shall be determined with a standard uncertainty not exceeding 0,6 K, while holding the specimen, which is mounted on the measurement mandrel or on the measurement base plate, at room temperature.

8.2 Voltage

For the resistance measurement, the voltage signal shall be measured with a relative standard uncertainty not exceeding 0,3 %.

8.3 Current

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

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

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

8.4 Dimension

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

For correction of the bending strain effect in the case of the wire with pure Cu matrix, the cross-sectional area of Cu matrix (S_{Cu}) shall be determined using a nominal value of copper to non-copper ratio and nominal dimensions of the specimen. The wire diameter (d) and mandrel radius (R_d) shall be determined with relative standard uncertainty not exceeding 1 % and 3 %, respectively.

9 Test report

9.1 RRR value

The obtained RRR value (r_{RRR}) shall be reported as

$$r_{RRR} (1 \pm U_{re}) \quad (n = \dots), \quad (10)$$

where

U_{re} is the expanded relative uncertainty:

$$U_{re} = 2u_r \quad (k = 2)$$

where

u_r denotes the relative combined standard uncertainty,

k is a coverage factor, and

n is the sampling number.

It is desired that n be larger than 4 so that the normal distribution can be assumed for observed results to estimate the standard deviation. If n is not sufficiently large, a rectangular distribution shall be assumed.

9.2 Specimen

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

- a) manufacturer;
- b) classification and/or symbol;
- c) shape and area of the cross-section;
- d) dimensions of the cross-sectional area;
- e) number of filaments or subelements;
- f) diameter of the filaments or subelements;
- g) for Nb-Ti specimen, the volume ratio of the following material to Nb-Ti:
 - Cu,
 - Cu-Ni,
 - Cu and Cu-Ni,
 - Al and Cu,
 or the following volume ratio:
 - Cu-Ni: Cu: Nb-Ti,
 - Al: Cu: Nb-Ti;
- h) Cu to non-Cu volume ratio for Nb₃Sn specimen;
- i) cross-sectional area of the Cu matrix (S_{Cu}).

9.3 Test conditions

9.3.1 Measurements of R_1 and R_2

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

- a) total length of the specimen;
- b) distance between the voltage measurement taps (L);
- c) length of each current contact;
- d) transport currents (I_1 and I_2);
- e) current densities (I_1 and I_2 divided by the nominal total wire cross-sectional area);
- f) voltages (U_1 , U_{0+} , U_{0rev} , U_{2+}^* , U_{20+} , U_{0-} , U_{2-}^* , U_{20-} and \bar{U}_2);
- g) resistances (R_m , R_1 , R_2^* and R_2);
- h) resistivities ($\rho_1 = (R_1 \times S_{Cu}) / L$ and $\rho_2 = (R_2 \times S_{Cu}) / L$);
- i) material, shape, and dimensions of the mandrel or the base plate;
- j) installation method of the specimen in the mandrel or the base plate;
- k) insulating material of the mandrel or the base plate.

9.3.2 Measurement of R_1

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

- a) temperature setting and holding method of the specimen;
- b) T_m : Temperature for measurement of R_m .

9.3.3 Measurement of R_2

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

- a) rate of increasing temperature;
- b) method of cooling down and heating up.

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 superconductors is given in Annex C.

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Annex A (informative)

Additional information relating to the measurement of RRR

A.1 Recommendation on specimen mounting orientation

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

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

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

a) Heater method

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

b) Controlled methods

1) Adiabatic method

In this method, the cryostat holds a chamber in which the specimen, a sample holder, a heater and so on are contained. Before the chamber is immersed in the liquid helium bath, air inside the chamber is pumped out and helium gas is filled. Then, the chamber is immersed in the liquid helium bath and the specimen 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.

2) Quasi-adiabatic method

In this method, the cryostat holds the specimen a certain distance above the liquid helium bath for the entire cryogenic measurement. A thermal anchor from the 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.

3) Refrigerator method

In this method, an electromechanical apparatus (a refrigerator) is used to cool the specimen, which is mounted on 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.3 Alternative measurement methods of R_2 or R_2^*

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

a) Modified reference method

This is a simplified method with acquisition of only one voltage-temperature curve and is used only for Nb-Ti composite superconductors. The voltage of the specimen is measured in the superconducting state under a desired direction of current (I_2) and then with current in the opposite direction. These values are U_{0+} and U_{0rev} as shown in Figure A.1. The current is then changed back to the initial direction. After the transition to the normal state, the voltage is measured as U'_{2+} in a plateau region of the curve within about 4 K above the transition. Then the voltage is read under a zero current (U_{20}). The current direction is then reversed and the voltage is measured again (U'_{2-}). The cryogenic resistance is obtained from

$$R_2^* = \frac{\bar{U}_2}{I_2} \quad (\text{A.1})$$

with

$$\bar{U}_2 = \frac{|U'_{2+} - U'_{2-}|}{2} \quad (\text{A.2})$$

This approximately compensates for the effect of thermoelectric voltage. The following conditions should be fulfilled to ensure that the influence of the interfering voltage and the thermoelectric voltage shift on R_2^* measurement is not appreciably large:

$$\frac{|U_{0+} - U_{0rev}|}{\bar{U}_2} < 1\% \quad (\text{A.3})$$

$$\frac{|\Delta_{2+} - \Delta_{2-}|}{\bar{U}_2} < 3\% \quad (\text{A.4})$$

where Δ_{2+} and Δ_{2-} are defined by $\Delta_{2+} = |U'_{2+} - U_{20}|$ and $\Delta_{2-} = |U'_{2-} - U_{20}|$, respectively.

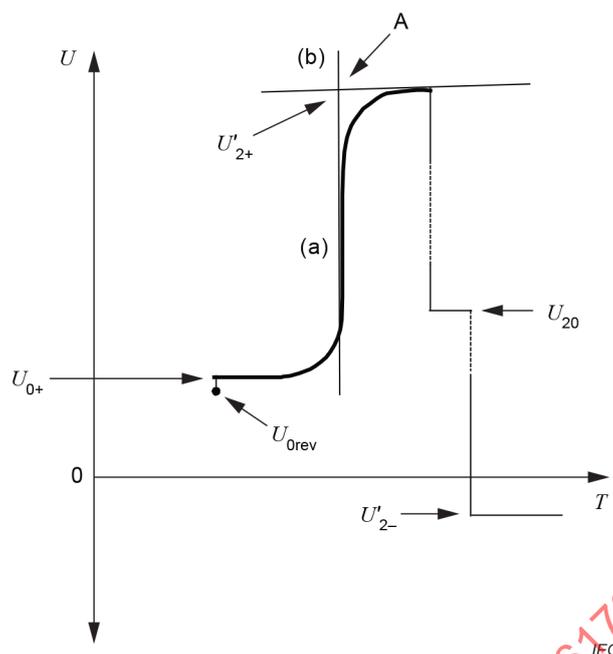


Figure A.1 – Definition of voltages

b) Measurement of voltage vs. time

Instead of measuring the voltage as a function of temperature, one can determine the cryogenic resistance from a voltage-versus-time curve that is continuously recorded both below and above the transition. Care should be taken not to re-cool the specimen without re-starting the acquisition of voltage-versus-time. The characteristic voltages, such as U_{2+}^* , can be similarly obtained from the intersection of the two straight lines drawn on the region of steepest slope during the transition and on the relatively flat region sufficiently above the transition in the voltage-versus-time curve. The analysis afterward to determine the cryogenic resistance is the same as in the reference method.

c) Fixed temperature method

In this method R_2 or R_2^* is directly determined at a fixed temperature in a plateau region within about 4 K above the transition for Nb-Ti composite superconductors, and R_2 is directly determined at 20 K for Nb₃Sn composite superconductors, instead of using the method described in 7.2. In this case it is desirable to check that the whole specimen is at a uniform and fixed temperature. In the measurement of Nb₃Sn composite superconductor the fixed temperature of 20 K should be determined with a combined standard uncertainty not exceeding 0,6 K. The fixed temperature and the combined standard uncertainty should be noted in the test report. Also the U_{0+} and U_{0-} , which are defined in 7.2.2, should be recorded as the zero voltage level in the fixed temperature method. In order to eliminate the influence of thermoelectric voltage, two voltage signals of the specimen, say U_{2+} and U_{2-} , should be acquired nearly simultaneously by reversal of the test current. For the fixed temperature method the effect of thermoelectric voltage on determination of cryogenic resistance can be eliminated.

d) Computer-based method

A computer can be used to control the current direction and warming of the specimen and to measure the voltage-temperature curve. Changes in current direction by periodic current reversals or periodic current on and off cycles are used to correct for offset voltages in order that the measurements can be made during one cycle of changing the specimen temperature. The effect of thermoelectric voltage should also be checked.

e) Other simplified methods with periodic checks

Simplified methods without temperature measurement might also be accepted, if an operator with sufficient experience performs the measurement using a given apparatus and if the

following condition is satisfied. If a simplified laboratory practice can be shown, through periodic checks, to achieve the same result as the method in this part of IEC 61788, within its stated uncertainty, then the simplified practice may be used in place of this reference method. These periodic checks could be accomplished by doing one of the following:

- 1) an interlaboratory comparison where one laboratory uses the reference method and another laboratory uses its own simplified method;
- 2) a single laboratory comparison where one laboratory "checks" their simplified method against the reference method;
- 3) periodic measurement of a small set of reference samples with well-known RRR values using the simplified method;
- 4) regular/frequent measurements with multiple specimens, one of which is a reference sample that would not be mounted/dismounted and would be measured every time as a calibrator.

A.4 Bending strain dependency of RRR for Nb-Ti composite superconductor

In general, the resistivity (ρ) of a pure metal such as copper at a very-low temperature increases as its applied strain increases. In general, a lower ρ wire has a larger percentage change in ρ than a higher ρ wire. There is almost no effect of strain on the room temperature resistivity of a metal. This means that the change in r_{RRR} with strain is more significant for a material whose r_{RRR} is high. According to the result of the intercomparison tests [1]¹, the dependency on bending strain was low for a specimen of low r_{RRR} . Bending strain is applied when the specimen is mounted on the measurement mandrel. Since the bending strain is inversely proportional to a radius of bent curvature, the smaller is the diameter of the measurement mandrel, the larger is the bending strain being applied to the specimen.

The increase in resistivity, $\Delta\rho$, at 4 K as a function of cold working ratio, r_{CW} [%], for pure copper is shown in Chapter 8 of reference [2]. Since the value of r_{CW} is approximately equal to the value of tensile strain, ε , when ε is small, the result is expressed as in Formula (9). The dependence of the copper resistivity increase on bending strain can be obtained by replacing the bending strain by an equivalent tensile strain.

Figure A.2 shows the relationship between r_{RRR} and bending strain for Nb-Ti composite superconductors with pure Cu matrix, obtained from the measured values of the intercomparison test performed in 1993 and 1994. The lines in the figure are the relationships calculated according to Formula (9) for each specimen. The measured values basically agree with the calculated values, and high r_{RRR} materials are sensitive to bending strain. Using Formula (9), Figure A.3 shows the dependency of round Cu wires where r_{RRR} with zero strain ($r_{RRR}(0)$) varies from 50 to 350. Figure A.4 shows bending strain dependency of r_{RRR} normalized by the value at zero strain. A similar dependency of rectangular Cu wires is shown in Figures A.5 and A.6. For copper with r_{RRR} of 350, which is the highest limit of r_{RRR} in this document, the r_{RRR} decreases by about 10 % for a bending strain of 2 %, with respect to the zero strain value.

¹ Numbers in square brackets refer to the Bibliography.

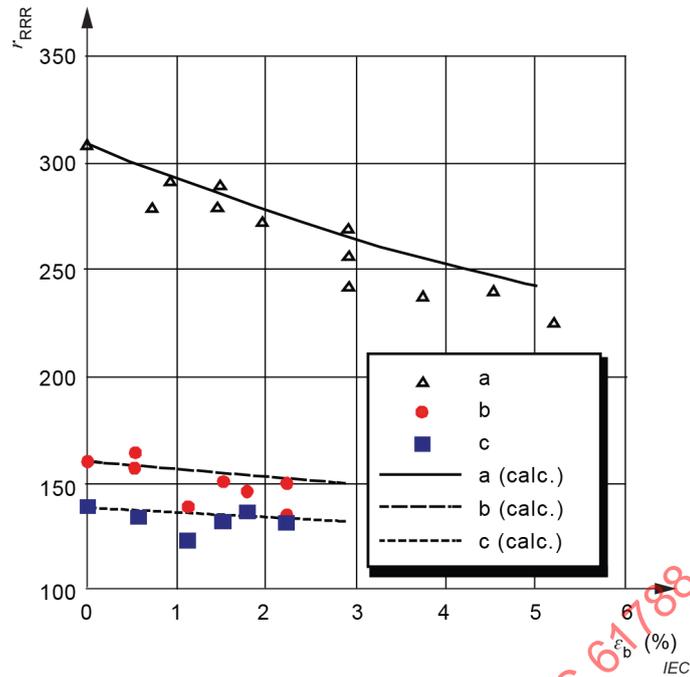


Figure A.2 – Bending strain dependency of RRR value for pure Cu matrix of Nb-Ti composite superconductors (comparison between measured values and calculated values)

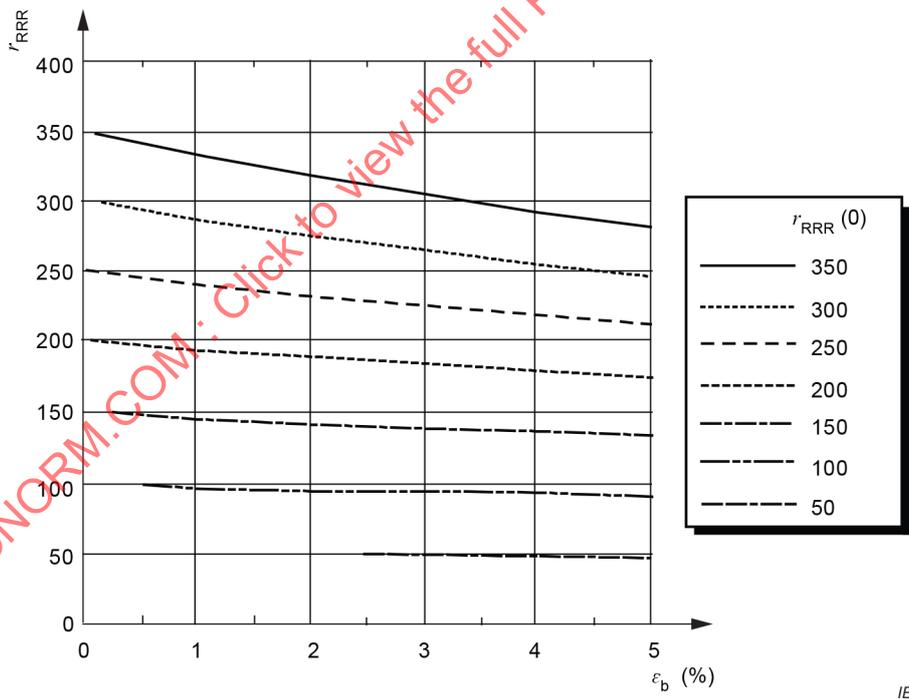


Figure A.3 – Bending strain dependency of RRR value for round Cu wires

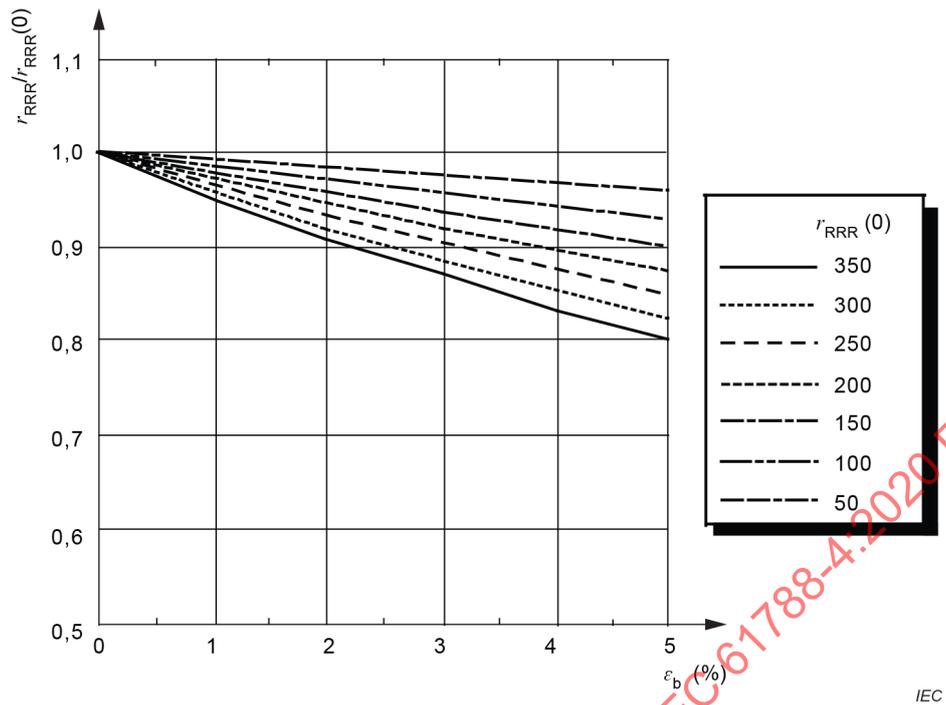


Figure A.4 – Bending strain dependency of normalized RRR value for round Cu wires

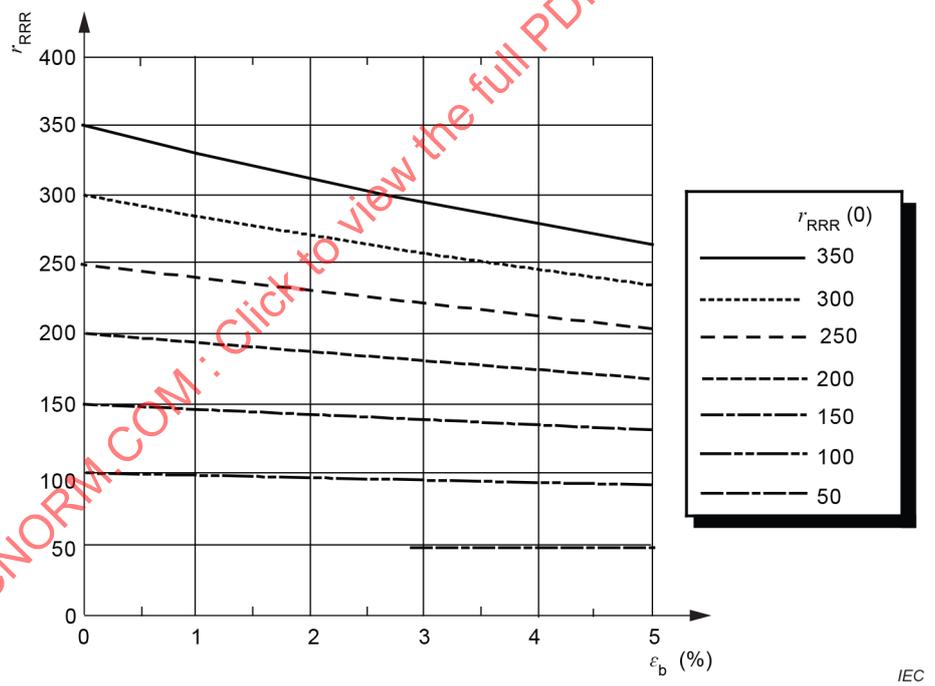


Figure A.5 – Bending strain dependency of RRR value for rectangular Cu wires

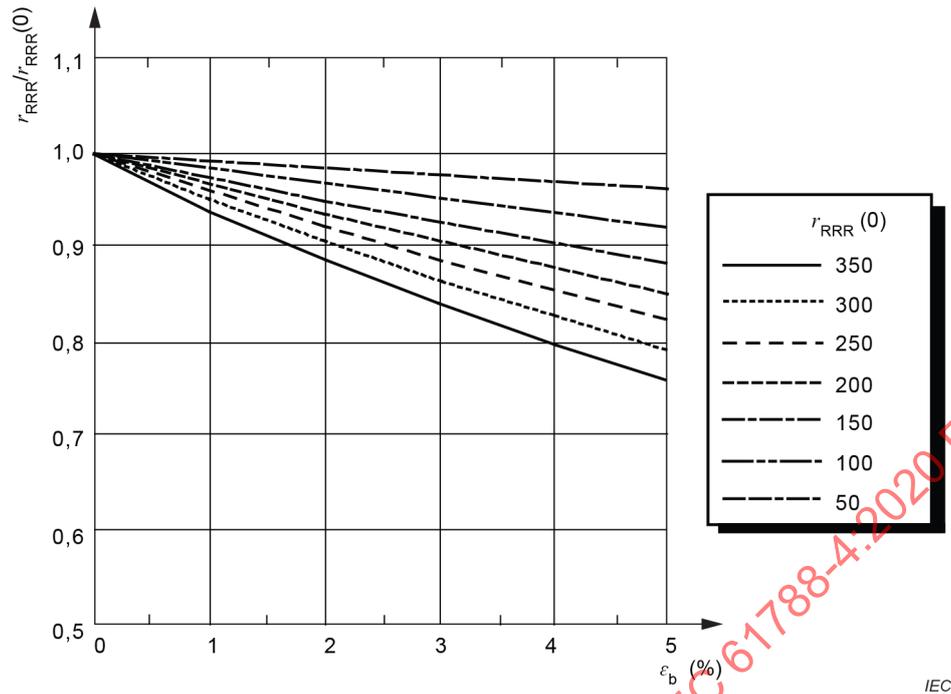


Figure A.6 – Bending strain dependency of normalized RRR value for rectangular Cu wires

To evaluate a high- r_{RRR} material, it is therefore desirable to use a straight base plate or a mandrel with a large coil diameter so that the evaluation can be performed with the least possible bending strain being applied. In addition to this, special care should be taken with the specimen so that there is no significant strain applied to it during handling.

The minimum diameters, D_m , of the measurement mandrel for round and rectangular wires are listed in Table A.1 and Table A.2, respectively.

Table A.1 – Minimum diameter of the measurement mandrel for round wires

Wire diameter d [mm]	0,50	0,75	1,00	1,25	1,50
Minimum diameter D_m [mm]	10,6	15,9	21,2	26,5	31,8

Table A.2 – Minimum diameter of the measurement mandrel for rectangular wires

Thickness t [mm]	0,25	0,50	0,75	1,00
Minimum diameter D_m [mm]	6,3	12,5	18,8	25,0

A.5 Procedure of correction of bending strain effect

Clause A.5 describes the procedure of correction of bending strain effect on the resistance at low temperature given in 7.3. For a specimen of thickness $2h$ mounted on a mandrel of radius R_d , the bending strain is given by

$$\varepsilon_b = 100 \times (h / R_d) \% . \tag{A.5}$$

Then, the equivalent tensile strain is

$$\varepsilon = (1/2)\varepsilon_b \quad (\text{A.6})$$

for a rectangular wire and

$$\varepsilon = [4/(3\pi)]\varepsilon_b \quad (\text{A.7})$$

for a round wire. The increase in the resistivity of pure copper at 4,2 K is calculated by substituting this ε value into Formula (9). Then, the corrected resistance at low temperature is calculated using Formula (8).

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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 [3].

It was left to each Technical Committee to decide if they were going to change existing and future standards to be consistent with the new unified approach. Such change is not easy and creates additional confusion, especially for those who are not familiar with statistics and the term uncertainty. At the June 2006 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 have been 1, 2, 3, or some other number. A manufacturer’s specification that can sometimes be described by a rectangular distribution will lead to a conversion number of $1/\sqrt{3}$. The appropriate coverage factor was used when converting the original number to the equivalent standard uncertainty. The conversion process is not something that the user of the standard needs to address for compliance to 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 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 interlaboratory comparison.

B.2 Definitions

Statistical definitions can be found in three sources: ISO/IEC Guide 98-3:2008, ISO/IEC Guide 99:2007 [4], and the NIST Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results (NIST) [5]. 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 (5.1.6, Annex J), but they are not formally defined in ISO/IEC Guide 98-3:2008 (see [3]).

B.3 Consideration of the uncertainty concept

Statistical evaluations in the past frequently used the coefficient of variation (COV), which is the ratio of the standard deviation and the mean (N.B. the COV is often called the relative standard deviation). Such evaluations have been used to assess the precision of the measurements and give the closeness of repeated tests. The standard uncertainty (SU) 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. Tables B.2, B.3, B.4 and B.5 are 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 (\bar{X}) [V]	
E_1	E_2
0,001 190 19	2,334 411 62

$$\bar{X} = \frac{\sum_{i=1}^n X_i}{n} \quad [\text{V}] \quad (\text{B.1})$$

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

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

$$\sigma = \sqrt{\frac{1}{n-1} \cdot \sum_{i=1}^n (X_i - \bar{X})^2} \quad [\text{V}] \quad (\text{B.2})$$

Table B.4 – Standard uncertainties of two output signals

Standard uncertainty (<i>u</i>) [V]	
<i>E</i> ₁	<i>E</i> ₂
0,000 095 97	0,000 067 48

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

Table B.5 – COV values of two output signals

COV value [%]	
<i>E</i> ₁	<i>E</i> ₂
25,498 2	0,009 1

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

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

B.4 Uncertainty evaluation example for 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 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) The user derives in the first step a mathematical measurement model in the form of identified measurand as a function of all input quantities. A simple example of such model is given for the uncertainty of a force, F_{LC} measurement using a load cell:

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

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 mass/day and the reproducibility of checks on different days, respectively.

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

- b) The user should identify the type of distribution for each input quantity (e.g. Gaussian distributions for Type A measurements and rectangular distributions for Type B measurements).
- c) Evaluate the standard uncertainty of the Type A measurements:

$$u_A = \frac{\sigma}{\sqrt{n}}$$

where σ is the experimental standard deviation and n is the total number of measured data points.

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

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

where d_w is the range of rectangular distributed values.

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

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

In this case, it has been assumed that there is no correlation between input quantities. If the model equation has terms with products or quotients, the combined standard uncertainty is evaluated using partial derivatives and the relationship becomes more complex due to the sensitivity coefficients [6], [7].

- f) Optional – the combined standard uncertainty of the estimate of the referred measurand can be multiplied by a coverage factor (e.g. 1 for 68 % or 2 for 95 % or 3 for 99 %) to increase the probability that the measurand can be expected to lie within the interval.
- g) Report the result as the estimate of the measurand \pm the expanded uncertainty, together with the unit of measurement, and, at a minimum, state the coverage factor used to compute the expanded uncertainty and the estimated coverage probability.

To facilitate the computation and standardize the procedure, use of appropriate certified commercial software is a straightforward method that reduces the amount of routine work [8], [9]². In particular, the indicated partial derivatives can be easily obtained when such a software tool is used. Further references for the guidelines of measurement uncertainties are given in [5], [10], and [11].

² References [8] and [9] give example(s) of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by IEC of these products.

Annex C (informative)

Uncertainty evaluation in test method of RRR for Nb-Ti and Nb₃Sn composite superconductors

C.1 Evaluation of uncertainty

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

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

$$\frac{\Delta r_{RRR}}{r_{RRR}} = \frac{\Delta R_1}{R_1} - \frac{\Delta R_2}{R_2} \quad (C.1)$$

Hence, the relative standard uncertainty of r_{RRR} is

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

Since the room temperature resistance is given by

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

the deviation of R_1 is

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

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

$$\Delta T_m = \Delta T_{m1} + \Delta T_{m2} \quad [K] \quad (C.5)$$

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

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

where

u_{U1} [V] is the Type B uncertainty in the room temperature voltage ($u_{U1}/U_1 = 0,005/\sqrt{3}$),

u_{I1} [A] is the Type B uncertainty in the room temperature current ($u_{I1}/I_1 = 0,005/\sqrt{3}$),

u_{Tm2} [K] is the Type B uncertainty in the room temperature measurement using a bolometer ($u_{Tm2} = 1/\sqrt{3}$ [K]).

The u_{RTm1} [Ω] is the type B uncertainty in R_1 due to the difference of the room temperature from the specimen temperature and is formally expressed as $u_{RTm1} = 0,00393 I_1 \Delta T_{m1}$. However, u_{Tm1} is not obtained from a mathematical model but u_{Tm1} is directly estimated as $\pm 1,7\%$ of R_1 from the results of round robin testing on RRR of Nb-Ti [12]. Assuming a similar situation, it can also be assumed as $u_{RTm1}/R_1 = 0,017/\sqrt{3}$.

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

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

where u_{U2} [V] is the Type B uncertainty due to the voltmeter, and u_{I2} [A] is the Type B uncertainty in the current. In the above, $u_{U2}/U_2 = 0,005/\sqrt{3}$ and $u_{I2}/I_2 = 0,005/\sqrt{3}$. The first and second terms are doubled because the measurements are done twice. Hence, when the sample is measured in a bending-free condition, the relative combined standard uncertainty is given by

$$u_r = \left[1,43 \times 10^{-4} + 2 \left(\frac{b}{R_2} \right)^2 \right]^{1/2}. \quad (C.8)$$

When the sample current is measured using a voltmeter and a standard resistor, the uncertainties of the voltage and resistance affect the uncertainty of measurement. If the value of the voltage and its standard uncertainty are U and u_U , and if the value of the resistance and its standard uncertainty are R and u_R , $(U_1/I_1^2)^2 u_{I1}^2$ in Formula (C.6) and $(U_2/I_2^2)^2 u_{I2}^2$ in Formula (C.7) are respectively replaced by

$$\left(\frac{U_1}{I_1} \right)^2 \left(\frac{u_U^2}{U^2} + \frac{u_R^2}{R^2} \right), \quad \left(\frac{U_2}{I_2} \right)^2 \left(\frac{u_U^2}{U^2} + \frac{u_R^2}{R^2} \right). \quad (C.9)$$

When the cryogenic resistance is measured in a bent condition, the result needs to be compensated for the strain effect using the given equation with the distance between the two voltage taps (L), the diameter (d), copper fraction (r_{Cu}) and the radius of a mandrel (R_d) used for the measurement. We assume that a round wire of diameter d is wound on a measurement mandrel of radius R_d . With the aid of Formulae (8) and (9) the value of the compensated cryogenic resistance is given by

$$\begin{aligned} R_2 &= R_2^* - 6,24 \times 10^{-12} \frac{8}{3\pi^2} \frac{L}{d r_{Cu} R_d} \\ &= R_2^* - 1,69 \times 10^{-12} \frac{L}{d r_{Cu} R_d} \quad [\Omega], \end{aligned} \quad (C.10)$$

where we have used $\varepsilon = (4/3\pi)(d/2R_d)$ and $S_{Cu} = \pi(d/2)^2 r_{Cu}$, and the small second term in Formula (9) was neglected. The quantity r_{Cu} is given by $r_{Cu} = c/(1+c)$ using the copper ratio, c . If the second term in Formula (C.10) is denoted by δR_2 , the contribution to the combined standard uncertainty of u_{R2} from the uncertainties of L , d , r_{Cu} and R_d is estimated as

$$u_{R2}^* = |\delta R_2| \left[\left(\frac{u_L}{L} \right)^2 + \left(\frac{u_d}{d} \right)^2 + \left(\frac{u_{rCu}}{r_{Cu}} \right)^2 + \left(\frac{u_{Rd}}{R_d} \right)^2 \right]^{1/2} \quad [\Omega], \quad (C.11)$$

where u_L [m], u_d [m], u_{rCu} and u_{Rd} [m] are the Type B standard uncertainties of distance between voltage taps, diameter, copper fraction and radius of mandrel, respectively. L is required to be measured within the uncertainty $u_L/L = 0,05/\sqrt{3}$. It is assumed that the uncertainty of d is $u_d/d = 0,02/\sqrt{3}$. The relative uncertainties of r_{Cu} and R_d are required to be smaller than $0,05/\sqrt{3}$. The maximum compensation is about $\delta R_2/R_2 = 0,10$ when the bending strain is 2 % for $r_{RRR} = 350$. Hence, the relative combined standard uncertainty of cryogenic resistance due to the bending strain correction is estimated at most to be

$$\frac{u_{R2}^*}{R_2} = 0,513 \times 10^{-2}. \quad (C.12)$$

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

$$u_r = \frac{u}{(R_1/R_2)} = \left[\left(\frac{u_{R1}}{R_1} \right)^2 + \left(\frac{u_{R2}}{R_2} \right)^2 + \left(\frac{u_{R2}^*}{R_2} \right)^2 \right]^{1/2} = \left[1,69 \times 10^{-4} + 2 \left(\frac{b}{R_2} \right)^2 \right]^{1/2}. \quad (C.13)$$

According to the round robin test shown in Clause C.2, u_r was estimated as $2,44 \times 10^{-2}$. Thus, b/R_2 is estimated as

$$\frac{b}{R_2} = 1,46 \times 10^{-2}. \quad (C.14)$$

The type and target value of uncertainty are listed for each measurement in Table C.1.

Table C.1 – Uncertainty of each measurement

Uncertainty	Type	Value	Remarks
u_{U_1}/U_1	B	$0,005/\sqrt{3}$	$ \Delta U_1 /U_1 < 0,005$
u_{I_1}/I_1	B	$0,005/\sqrt{3}$	$ \Delta I_1 /I_1 < 0,005$
u_{Tm2}	B	$1/\sqrt{3}$ K	$ \Delta T_m < 1$ K
u_{U_2}/U_2	B	$0,005/\sqrt{3}$	$ \Delta U_2 /U_2 < 0,005$
u_{I_2}/I_2	B	$0,005/\sqrt{3}$	$ \Delta I_2 /I_2 < 0,005$
u_L/L	B	$0,05/\sqrt{3}$	$ \Delta L /L < 0,05$
u_d/d	B	$0,02/\sqrt{3}$	$ \Delta d /d < 0,02$
$u_{r_{Cu}}/r_{Cu}$	B	$0,05/\sqrt{3}$	$ \Delta r_{Cu} /r_{Cu} < 0,05$
u_{R_d}/R_d	B	$0,05/\sqrt{3}$	$ \Delta R_d /R_d < 0,05$

C.2 Summary of round robin test of RRR of a Nb-Ti composite superconductor

The round robin test of RRR was carried out on a Cu/Nb-Ti composite superconductor. The specifications of the test superconductor are:

- diameter: 0,80 mm, 0,86 mm including insulating layer;
- Cu/Nb-Ti ratio: 6,5;
- mean filament diameter: about 70 μm ;
- number of filaments: 16;
- twist pitch: 30 mm;
- critical current: more than 185 A (3 T, 4,2 K);
- r_{RRR} : more than 150.

Participating institutes were provided with specimens that were nearly straight. Some specimens were measured in the as-received condition and some were measured wound on a bobbin under a strained condition. The number of participating institutes was 13 from five countries and the number of determinations was 77. R_2 was measured following the method defined in 7.2 and 7.3, and those in Clause A.3. The details of the measurements are described in reference [12]. The effect of the strain was corrected using Formulae (8) and (9). The distribution of the measured r_{RRR} is shown in Figure C.1. Almost all of the data, except for three, were concentrated fairly sharply. The average was 178,5, the standard deviation was 4,4 and the COV value was 2,44 %. If the three extraordinary data are omitted, the average was 178,2, the standard deviation was 3,1 and the COV value was 1,73 %.

Hence, it is reasonable to define the target relative combined standard uncertainty of this method not to exceed 2,5 % based on the COV value in the round robin test.

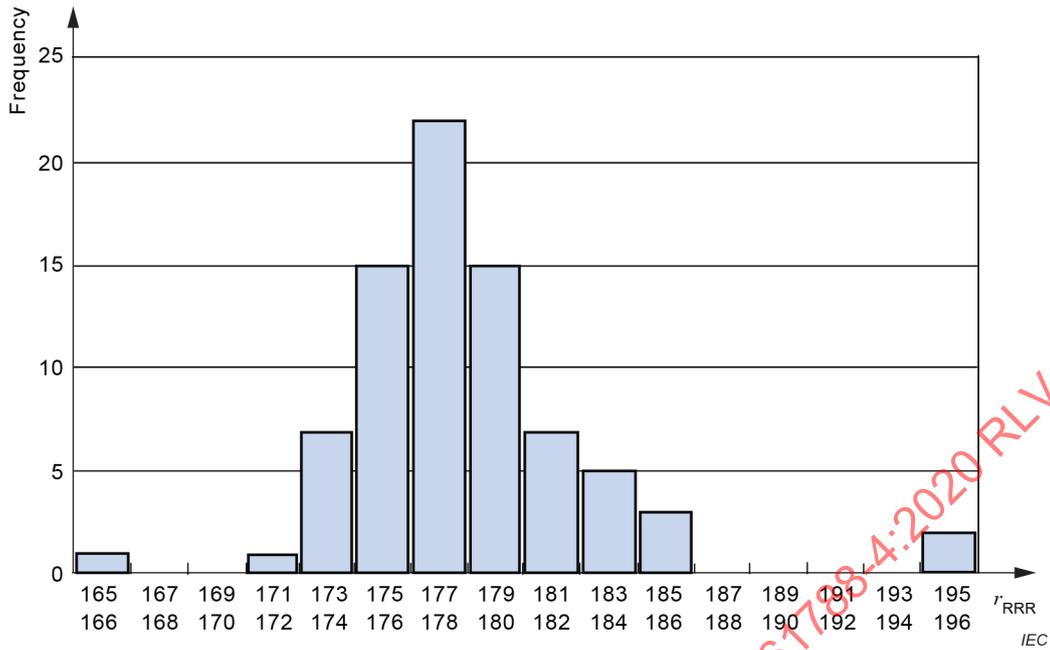


Figure C.1 – Distribution of observed r_{RRR} of Cu/Nb-Ti composite superconductor

C.3 Reason for large COV value in the intercomparison test on Nb₃Sn composite superconductor

The COV value of the intercomparison test for Nb₃Sn samples was 6,07 % [13]. This value is much larger than that for Nb-Ti (2,44 %), although there is no contribution from additional uncertainty in correction of the strain effect. For clarification of the reason for the large COV a round robin test was performed. Since inhomogeneity in Nb₃Sn samples is anticipated as one of the reasons, real round robin tests were examined by measuring one specimen successively by participating institutions. The measured wires were two internal tin-processed Nb₃Sn composite superconductors. Three samples were cut from different positions of each wire. The number of participating institutions was six and the measurement method described in this document was employed. Each sample was successively measured seven times including one measurement C^a using the voltage vs. time method described in Clause A.3 b). All measured RRR values for six samples are shown in four digits for the analysis of the uncertainty in Table C.2, whereas significant digits are three. Table C.3 shows the average, standard deviation and COV.

Table C.2 – Obtained values of RRR for six Nb₃Sn specimens

Institution	r_{RRR}					
	#1-1	#1-2	#1-3	#2-1	#2-2	#2-3
A	152,7	155,7	158,2	182,2	177,7	181,3
B	152,5	159,4	158,0	189,1	173,4	183,0
C	148,6	153,4	154,3	175,8	168,1	177,9
D	150,0	153,4	150,6	174,8	156,7	173,0
E	143,2	153,4	153,3	178,6	164,8	177,8
F	148,0	156,3	157,3	181,6	171,3	181,6
C ^a	144,6	151,4	153,8	178,6	168,3	178,1

^a The voltage-time curve is used for determination of the cryogenic resistance. Although the significant digits of observed RRR value are three, four digit numbers are listed for the analysis of uncertainty.

Table C.3 – Average, standard deviation and coefficient of variation for six specimens

	#1-1	#1-2	#1-3	#2-1	#2-2	#2-3
Average	148,5	154,7	155,1	180,1	168,6	179,0
σ	3,6	2,6	2,8	4,8	6,7	3,4
COV(%)	2,5	1,7	1,8	2,7	4,0	1,9

The obtained COV for Nb₃Sn is ranged 1,70 % to 3,98 % and comparable to that for Nb-Ti, 2,44 %. If the result on specimen #2-2 with the largest COV value is disregarded, it is comparable to or even better than that for Nb-Ti. The uncertainty of RRR in Nb₃Sn is mostly attributed to that of measurement of the cryogenic resistance [14], which is largely different from the case of Nb-Ti. The difficulty to fit a straight line to non-linear voltage vs. temperature curves may be a reason for the large uncertainty. This round robin test clarified that Nb₃Sn composite superconductors are somewhat inhomogeneous along their length. This may be caused by the high sensitivity to heat treatment conditions or to random pores or breaks in the diffusion barrier. For this reason it is recommended to measure several specimens of a given superconductor to evaluate the distribution of the RRR value.

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COMMISSION ÉLECTROTECHNIQUE INTERNATIONALE

SUPRACONDUCTIVITÉ –

**Partie 4: Mesurage du rapport de résistance résiduelle –
Rapport de résistance résiduelle des composites
supraconducteurs de Nb-Ti et de Nb₃Sn**

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La Norme internationale IEC 61788-4 a été établie par le comité d'études 90 de l'IEC: Supraconductivité.

Cette cinquième édition annule et remplace la quatrième édition parue en 2016. Cette édition constitue une révision technique.

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

- a) modification de la distance adéquate des prises de tension sur les éprouvettes pour un mesurage fiable,

- b) nouveau rapport du résultat de l'essai interlaboratoire du rapport de résistance résiduelle des supraconducteurs Nb₃Sn qui atteste de la validité de la méthode de mesure utilisée dans la présente norme,
- c) révision des définitions prêtant à confusion concernant le rapport entre le cuivre et la fraction de cuivre.

Le texte de cette norme est issu des documents suivants:

FDIS	Rapport de vote
90/448/FDIS	90/451/RVD

Le rapport de vote indiqué dans le tableau ci-dessus donne toute information sur le vote ayant abouti à l'approbation de la présente norme.

Cette publication a été rédigée selon les Directives ISO/IEC, Partie 2.

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

Le comité a décidé que le contenu de cette publication ne sera pas modifié avant la date de stabilité indiquée sur le site web de l'IEC sous «<http://webstore.iec.ch>» dans les données relatives à la publication recherchée. À cette date, la publication sera

- reconduite,
- supprimée,
- remplacée par une édition révisée, ou
- amendée.

IMPORTANT – Le logo "colour inside" qui se trouve sur la page de couverture de cette publication indique qu'elle contient des couleurs qui sont considérées comme utiles à une bonne compréhension de son contenu. Les utilisateurs devraient, par conséquent, imprimer cette publication en utilisant une imprimante couleur.

INTRODUCTION

Le cuivre, Cu/Cu-Ni ou l'aluminium est utilisé comme matériau de matrice dans les composites supraconducteurs de Ni-Ti et de Nb₃Sn et fonctionne comme un shunt électrique lorsque la supraconductivité est interrompue. Il contribue également à la reprise de la supraconductivité en dirigeant la chaleur générée dans le supraconducteur vers le fluide de refroidissement environnant. La résistivité à la température cryogénique du cuivre est une grandeur importante qui influe sur la stabilité et les pertes en courant alternatif du supraconducteur. Le rapport de résistance résiduelle est défini comme le rapport de la résistance du supraconducteur à température ambiante à celle juste au-dessus de la transition supraconductrice.

Le présent document spécifie la méthode d'essai relative au rapport de résistance résiduelle des composites supraconducteurs de Nb-Ti et de Nb₃Sn. La méthode des courbes est utilisée pour mesurer la résistance juste au-dessus de la transition supraconductrice. D'autres méthodes sont décrites en A.3.

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SUPRACONDUCTIVITÉ –

Partie 4: Mesurage du rapport de résistance résiduelle – Rapport de résistance résiduelle des composites supraconducteurs de Nb-Ti et de Nb₃Sn

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) des composites supraconducteurs de Nb-Ti et de Nb₃Sn avec une matrice de Cu, Cu-Ni, Cu/Cu-Ni et Al dans une condition sans contrainte et dans un champ magnétique externe nul. Cette méthode est destinée à être utilisée avec des éprouvettes de supraconducteurs présentant une structure monolithique avec une section rectangulaire ou circulaire, un RRR de valeur inférieure à 350 et une surface de section inférieure à 3 mm². Dans le cas de Nb₃Sn, les éprouvettes ont subi un traitement thermique de réaction.

2 Références normatives

Les documents suivants cités dans le texte 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 Electrotechnique International (IEV) – Partie 815: Supraconductivité* (disponible sous: www.electropedia.org)

3 Termes et définitions

Pour les besoins du présent document, les termes et définitions de l'IEC 60050-815 et 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 <http://www.electropedia.org/>
- ISO Online browsing platform: disponible à l'adresse <http://www.iso.org/obp>

3.1 rapport de résistance résiduelle RRR

rapport de la résistance à la température ambiante à la résistance juste au-dessus de la transition supraconductrice

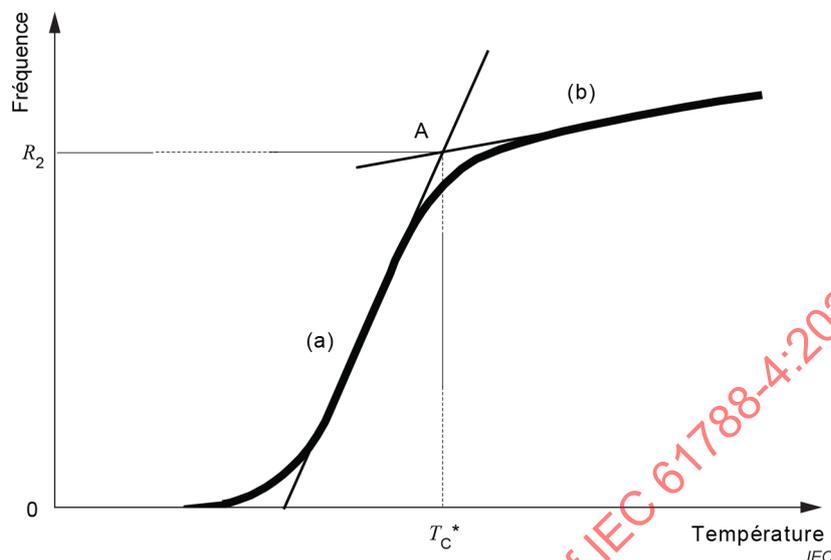
Note 1 à l'article: Cette note ne s'applique qu'à la langue anglaise.

Note 2 à l'article: Dans le présent document relatif aux composites supraconducteurs de Nb-Ti et de Nb₃Sn, la température ambiante est définie à 293 K (20 °C), et le rapport de résistance résiduelle est obtenu au moyen de la Formule (1), où la résistance (R_1) à 293 K est divisée par la résistance (R_2) juste au-dessus de la transition supraconductrice.

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

Ici r_{RRR} est une valeur du rapport de résistance résiduelle, R_2 est une valeur de la résistance mesurée dans une condition sans contrainte et dans un champ magnétique externe nul.

La Figure 1 représente de manière schématique une courbe de la résistance en fonction de la température obtenue sur une éprouvette en mesurant la résistance cryogénique.–



La résistance cryogénique, R_2 , est déterminée par l'intersection, A, de deux lignes droites (a) et (b) à la température T_C^* .

Figure 1 – Rapport entre la température et la résistance

4 Principe

Le mesurage de la résistance à température ambiante comme à température cryogénique doit être effectué au moyen de la technique des quatre bornes. Aucun champ magnétique n'est appliqué pour la réalisation de tous les mesurages.

L'incertitude type composée relative à la cible de cette méthode est définie comme une incertitude élargie ($k=2$) ne devant pas dépasser 5 %.

La flexion maximale induite durant le montage et le refroidissement de l'éprouvette de Nb-Ti ne doit pas dépasser 2 %. En ce qui concerne l'éprouvette de Nb₃Sn, le mesurage doit être effectué dans une condition sans contrainte ou avec une contrainte thermique admissible.

5 Appareillage

5.1 Matériau du mandrin de mesure ou de l'embase de mesure

Le mandrin de mesure pour une éprouvette de Nb-Ti enroulée ou l'embase de mesure pour une éprouvette de Nb-Ti ou de Nb₃Sn rectiligne doit être en cuivre, en aluminium, en argent ou matériau équivalent dont la conductivité thermique est supérieure ou égale à 100 W/(m·K) à la température de l'hélium liquide (4,2 K). La surface du matériau doit être recouverte d'une couche de matériau isolant (bande ou couche de polyéthylène téréphtalate, de polyester, de polytétrafluoroéthylène, etc.) d'une épaisseur maximale de 0,1 mm.

5.2 Diamètre du mandrin de mesure et longueur de l'embase de mesure

Le diamètre du mandrin de mesure doit être suffisamment grand pour maintenir la flexion inférieure ou égale à 2 % pour l'éprouvette de Nb-Ti. L'éprouvette de Nb₃Sn sur une embase doit être mesurée dans une condition sans contrainte ou avec une contrainte thermique admissible.

L'une des dimensions de l'embase de mesure doit être d'au moins 30 mm de longueur.

5.3 Cryostat pour le mesurage de la résistance (R_2)

Le cryostat doit comprendre une structure de support de l'éprouvette et un réservoir à hélium liquide pour le mesurage de la résistance R_2 . La structure de support de l'éprouvette doit permettre d'immerger et de retirer l'éprouvette, qui est montée sur un mandrin de mesure ou une embase de mesure, dans et hors du bain d'hélium liquide. La structure de support de l'éprouvette doit en outre être réalisée de façon qu'un courant puisse traverser l'éprouvette et que la tension résultante générée le long de l'éprouvette puisse être mesurée.

6 Préparation de l'éprouvette

L'éprouvette d'essai ne doit avoir aucun joint ou aucune épissure d'une longueur de 30 mm ou plus. Elle doit comporter des contacts de courant à proximité de chacune de ses extrémités et d'une paire de contacts de tension sur sa partie centrale. La distance entre les deux prises de réglage de tension (L) doit être supérieure ou égale à 15 mm. Un thermomètre de mesure de la température cryogénique doit être fixé à proximité de l'éprouvette.

Une méthode mécanique doit être utilisée pour maintenir l'éprouvette contre la couche de matériau isolant du mandrin ou de l'embase de mesure. Il convient de réaliser avec soin l'instrumentation et l'installation de l'éprouvette sur le mandrin de mesure ou l'embase de mesure, de manière à éviter des flexions ou des déformations de traction indésirables. Dans l'absolu, il est prévu que l'éprouvette de Nb₃Sn soit la plus rectiligne possible; or, ce n'est pas toujours le cas, c'est pourquoi il convient de veiller à bien mesurer l'éprouvette à l'état de livraison.

L'éprouvette doit être montée sur un mandrin de mesure ou sur une embase de mesure pour effectuer ces mesurages. Les deux mesurages de résistance, R_1 et R_2 , doivent être effectués sur la même éprouvette et le même montage.

7 Acquisition et analyse des données

7.1 Résistance (R_1) à température ambiante

L'éprouvette fixée doit être mesurée à température ambiante (T_m (K)), où T_m remplit la condition suivante: $273 \text{ K} \leq T_m \leq 308 \text{ K}$. Un courant (I_1 (A)) doit traverser l'éprouvette de sorte que la densité du courant se situe entre $0,1 \text{ A/mm}^2$ et 2 A/mm^2 sur la base de la surface totale de la section du fil, et la tension résultante (U_1 (V)), I_1 et T_m doivent être enregistrées. La Formule (2) ci-dessous doit être utilisée pour calculer la résistance (R_m) à température ambiante. La résistance (R_1) à 293 K (20 °C) doit être calculée au moyen de la Formule (3) pour un fil avec matrice en cuivre. Pour les fils qui ne contiennent pas de composant en cuivre pur, la valeur fixée de R_1 doit être égale à R_m , sans aucune correction de température.

$$R_m = \frac{U_1}{I_1} \quad (2)$$

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

7.2 Résistance (R_2 ou R_2^*) juste au-dessus de la transition supraconductrice

7.2.1 Correction de l'effet contrainte/déformation

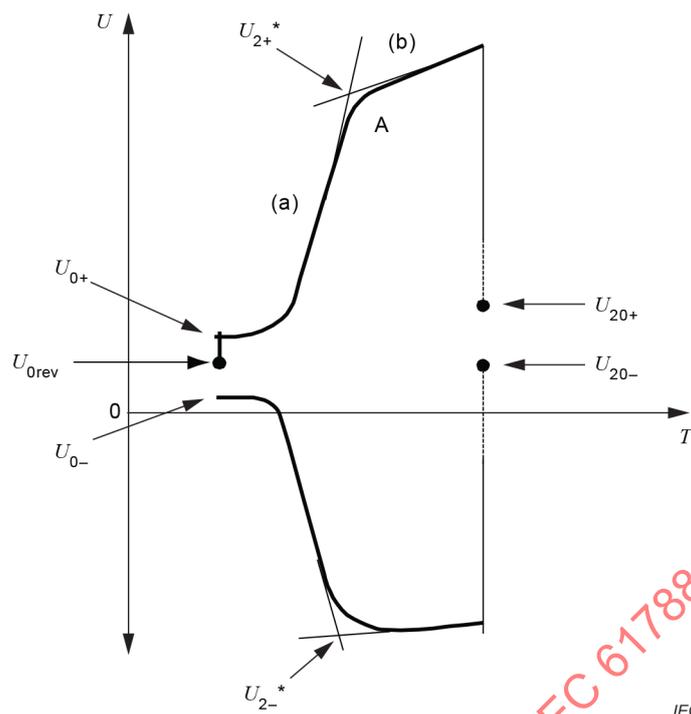
Dans l'état contraint de l'éprouvette de Nb-Ti, la résistance cryogénique mesurée, R_2^* , n'est pas une valeur correcte de R_2 . La correction correspondante de l'effet contrainte/déformation est décrite en 7.3.

7.2.2 Acquisition des données de la résistance cryogénique

L'éprouvette, toujours montée comme pour le mesurage à température ambiante, doit être placée dans le cryostat pour le mesurage électrique spécifié en 5.3. Un montage horizontal de l'éprouvette est recommandé en A.1. D'autres cryostats qui utilisent un élément chauffant pour faire varier la température de l'éprouvette sont décrits en A.2. L'éprouvette doit être lentement immergée dans le bain d'hélium liquide et refroidie à la température de l'hélium liquide pendant au moins 5 min.

Pendant les phases de réalisation des mesurages de R_2^* à basse température, un courant (I_2) de l'éprouvette doit être appliqué de sorte que la densité du courant se situe entre 0,1 A/mm² et 10 A/mm² sur la base de la surface totale de la section du fil, et la tension résultante (U (V)), I_2 (A), et la température de l'éprouvette (T (K)) doivent être enregistrées. Pour conserver un rapport signal/bruit suffisamment élevé, le mesurage doit être effectué en respectant la condition selon laquelle la valeur absolue de la tension résultante supérieure à la transition supraconductrice dépasse 10 μ V. Les données à obtenir et leur analyse sont représentées à la Figure 2.

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NOTE Les tensions indiquées + et - sont celles qui sont obtenues au premier et au deuxième mesurage avec, respectivement, des courants positif et négatif, et U_{20+} et U_{20-} sont les tensions obtenues avec un courant nul. Pour des raisons de clarté, U_{0rev} mesurée avec un courant nul et U_{0-} ne coïncident pas. Une droite (a) est tracée dans la région de transition avec une forte augmentation de la tension avec la température et une droite (b) est tracée dans la région avec une tension presque constante.

Figure 2 – Courbes de la tension en fonction de la température et définitions de chaque tension

Lorsque l'éprouvette est à l'état supraconducteur et que le courant d'essai (I_2) est appliqué, deux tensions doivent être mesurées presque simultanément: U_{0+} (la tension initiale enregistrée avec une polarité de courant positive) et U_{0rev} (la tension enregistrée pendant une courte variation de polarité du courant appliqué). Un mesurage valide de R_2^* exige l'absence de tensions de perturbation excessives et implique que l'éprouvette soit initialement à l'état supraconducteur. La formule suivante doit alors être satisfaite pour effectuer un mesurage valide:

$$\frac{|U_{0+} - U_{0rev}|}{\bar{U}_2} < 1\% \quad (4)$$

où \bar{U}_2 est la tension moyenne pour l'éprouvette à l'état normal à température cryogénique, définie par la Formule (5).

L'éprouvette doit être progressivement chauffée de sorte qu'elle passe entièrement à l'état normal. Lorsque le cryostat pour le mesurage de la résistance spécifié en 5.3 est utilisé, cela peut être effectué en soulevant simplement l'éprouvette jusqu'à une position appropriée au-dessus du niveau de l'hélium liquide. La courbe de la tension en fonction de la température de l'éprouvette doit être obtenue en maintenant la vitesse de montée en température entre 0,1 K/min et 10 K/min. La courbe de la tension en fonction de la température doit être enregistrée en continu pendant la transition vers l'état normal, jusqu'à une température inférieure à 15 K pour l'éprouvette de Nb-Ti et inférieure à 25 K pour l'éprouvette de Nb₃Sn. Le courant de l'éprouvette doit ensuite être ramené à zéro et la tension correspondante, U_{20+} , doit

être enregistrée à une température inférieure à 15 K pour l'éprouvette de Nb-Ti et inférieure à 25 K pour l'éprouvette de Nb₃Sn.

L'éprouvette doit ensuite être lentement immergée dans le bain d'hélium liquide et refroidie à ±1 K près de la température à laquelle le signal de tension initial U_{0+} a été enregistré. Un courant de l'éprouvette, I_2 , ayant la même intensité mais une polarité négative (polarité inverse par rapport à celle utilisée pour la courbe initiale) doit être appliqué, et la tension U_{0-} doit être enregistrée à cette température. Les étapes du mode opératoire doivent être répétées pour enregistrer la courbe du rapport entre la tension et la température avec ce courant négatif. En outre, lorsque le courant de mesure, I_2 , diminue jusqu'à 0, l'enregistrement de U_{20-} doit être effectué à ±1 K près de la température à laquelle U_{20+} a été enregistrée.

Chacune des deux courbes du rapport entre la tension et la température doit être analysée en traçant une droite (a) sur la courbe des données au niveau où la valeur absolue de la tension augmente fortement avec la température (voir la Figure 2) et en traçant une seconde droite (b) sur la courbe des données au-dessus de la transition, au niveau où la tension est presque constante pour Nb-Ti ou a augmenté progressivement et presque linéairement pour Nb₃Sn par rapport à la montée en température. U_{2+}^* et U_{2-}^* sur la Figure 2 doivent être déterminées à l'intersection de ces deux droites, respectivement pour les courbes de polarité positive et négative.

Les tensions corrigées, U_{2+} et U_{2-} , doivent être calculées au moyen des équations suivantes:

$U_{2+} = U_{2+}^* - U_{0+}$ et $U_{2-} = U_{2-}^* - U_{0-}$. La tension moyenne, \bar{U}_2 , doit être définie comme suit:

$$\bar{U}_2 = \frac{|U_{2+} - U_{2-}|}{2} \quad (5)$$

Un mesurage valide de R_2^* implique d'effectuer la variation de tension thermoélectrique dans des limites acceptables pendant les mesurages de U_{2+} et U_{2-} . La condition suivante doit alors être remplie pour effectuer un mesurage valide:

$$\frac{|\Delta_+ - \Delta_-|}{\bar{U}_2} < 3 \% \quad (6)$$

où Δ_+ et Δ_- sont définis par $\Delta_+ = U_{20+} - U_{0+}$ et $\Delta_- = U_{20-} - U_{0-}$. Si le mesurage de R_2^* ne respecte pas les exigences de validité en 7.2.2, particulièrement de la Formule (4) ou (6), des mesures pour l'amélioration du matériel ou du mode opératoire expérimental doivent être prises pour respecter ces exigences avant de consigner les résultats.

La Formule (7) doit être utilisée pour calculer la résistance mesurée (R_2^*) juste au-dessus de la transition supraconductrice.

$$R_2^* = \frac{\bar{U}_2}{I_2} \quad (7)$$

7.2.3 Méthodes d'acquisition facultatives

La méthode de «référence» est celle décrite dans le corps du présent document tandis que les méthodes d'acquisition facultatives sont décrites en A.3.

7.3 Correction de R_2^* mesurée d'un composite supraconducteur de Nb-Ti pour une flexion

S'il n'y a pas de composant en cuivre pur dans le supraconducteur, R_2 doit alors être égale à R_2^* .

Pour une éprouvette avec un composant en cuivre pur, la flexion doit être définie par $\varepsilon_b = 100 \times (h/r)$ (%), où h est égale à la moitié de l'épaisseur de l'éprouvette pour les fils rectangulaires ou à un rayon pour les fils circulaires et r est le rayon de courbure. Si la flexion est inférieure à 0,3 %, aucune correction n'est alors nécessaire, et R_2 doit être égale à R_2^* .

Si aucune des deux situations ci-dessus ne s'applique, la résistance R_2 juste au-dessus de la transition supraconductrice dans une condition sans contrainte doit alors être estimée comme suit:

$$R_2 = R_2^* - \Delta\rho \times \frac{L}{S_{\text{Cu}}} \quad (8)$$

où $\Delta\rho$ est défini ci-dessous et S_{Cu} et L sont définis en 8.4. L'augmentation de résistivité du cuivre pur à 4,2 K due à la déformation de traction, ε (%), s'exprime comme suit:

$$\Delta\rho \text{ (}\Omega\text{m)} = 6,24 \times 10^{-12} \varepsilon - 5,11 \times 10^{-14} \varepsilon^2; \varepsilon \leq 2 \% \quad (9)$$

Le calcul de la Formule (9) doit être effectué en partant du principe que la déformation de traction équivalente ε est respectivement de $(1/2)\varepsilon_b$ et $(4/3\pi)\varepsilon_b$ pour des fils rectangulaires et circulaires. La dépendance du rapport de résistance résiduelle en fonction de la flexion pour le cuivre pur est décrite en A.4.

7.4 Rapport de résistance résiduelle (RRR)

La valeur du RRR doit être calculée en utilisant la Formule (1).

8 Incertitude et stabilité de la méthode d'essai

8.1 Température

La température ambiante doit être déterminée avec une incertitude type ne dépassant pas 0,6 K, en maintenant à température ambiante l'éprouvette, qui est montée sur le mandrin de mesure ou sur l'embase de mesure.

8.2 Tension

Pour le mesurage de résistance, le signal de tension doit être mesuré avec une incertitude type relative ne dépassant pas 0,3 %.

8.3 Courant

Lorsque le courant est appliqué directement à l'éprouvette avec une source de courant continu programmable, le courant d'essai de l'éprouvette doit être déterminé avec une incertitude type relative ne dépassant pas 0,3 %.

Lorsque le courant d'essai de l'éprouvette est déterminé à partir de la caractéristique tension-courant d'une résistance étalon par la technique à quatre bornes, la résistance étalon, avec une incertitude type composée relative ne dépassant pas 0,3 %, doit être utilisée.

La fluctuation du courant d'essai continu de l'éprouvette, fourni par une alimentation en courant continu, doit être inférieure à 0,5 % pendant chaque mesurage de résistance.

8.4 Dimension

La distance le long de l'éprouvette entre les deux prises de tension (L) doit être déterminée avec une incertitude type composée relative ne dépassant pas 5 %.

Pour la correction de l'effet contrainte/déformation de flexion dans le cas du fil avec une matrice en cuivre pur, la surface de section de la matrice de cuivre (S_{Cu}) doit être déterminée en utilisant une valeur nominale du rapport entre le cuivre et l'absence de cuivre et les dimensions nominales de l'éprouvette. Le diamètre du fil (d) et le rayon du mandrin (R_d) doivent être déterminés avec une incertitude type relative ne dépassant respectivement pas 1 % et 3 %.

9 Rapport d'essai

9.1 Valeur du RRR

La valeur du RRR (r_{RRR}) obtenue doit être consignée comme suit

$$r_{RRR} (1 \pm U_{re}) \quad (n = \dots), \quad (10)$$

où

U_{re} est l'incertitude élargie relative:

$$U_{re} = 2u_r \quad (k = 2)$$

où

u_r représente l'incertitude type composée relative,

k est un facteur d'élargissement, et

n est le nombre d'échantillonnages.

Il est souhaitable que n soit supérieur à 4 de façon à pouvoir considérer une distribution normale pour les résultats observés afin d'estimer l'écart type. Si n n'est pas suffisamment grand, une distribution rectangulaire doit être considérée.

9.2 Éprouvette

Le rapport d'essai des résultats des mesurages doit également comprendre les éléments suivants, s'ils sont connus:

- a) fabricant;
- b) classification et/ou symbole;
- c) forme et surface de la section;
- d) dimensions de la surface de section;
- e) nombre de filaments ou de sous-éléments;
- f) diamètre des filaments ou des sous-éléments;
- g) pour l'éprouvette Nb-Ti, le rapport volumique du matériau suivant sur Nb-Ti:
 - Cu,
 - Cu-Ni,

Cu et Cu-Ni,

Al et Cu,

ou le rapport volumique suivant:

Cu-Ni: Cu: Nb-Ti,

Al: Cu: Nb-Ti;

- h) rapport volumique cuivre/non-cuivre pour l'éprouvette de Nb₃Sn;
- i) surface de section de la matrice de cuivre (S_{Cu}).

9.3 Conditions d'essai

9.3.1 Mesurage de R_1 et R_2

Les conditions d'essai suivantes doivent être consignées pour le mesurage de R_1 et R_2 :

- a) longueur totale de l'éprouvette;
- b) distance entre les prises de mesure de tension (L);
- c) longueur de chaque contact de courant;
- d) courants de transport (I_1 et I_2);
- e) densités de courant (I_1 et I_2 divisées par la surface nominale totale de la section du fil);
- f) tensions (U_1 , U_{0+} , U_{0rev} , U_{2+}^* , U_{20+} , U_{0-} , U_{2-}^* , U_{20-} et \bar{U}_2);
- g) résistances (R_m , R_1 , R_2^* et R_2);
- h) résistivités ($\rho_1 = (R_1 \times S_{Cu}) / L$ et $\rho_2 = (R_2 \times S_{Cu}) / L$);
- i) matériau, forme et dimensions du mandrin ou de l'embase;
- j) méthode d'installation de l'éprouvette sur le mandrin ou l'embase;
- k) matériau isolant du mandrin ou de l'embase.

9.3.2 Mesurage de R_1

Les conditions d'essai suivantes doivent être consignées pour le mesurage de R_1 :

- a) réglage de la température et méthode de fixation de l'éprouvette;
- b) T_m : Température pour le mesurage de R_m .

9.3.3 Mesurage de R_2

Les conditions d'essai suivantes doivent être consignées pour le mesurage de R_2 :

- a) vitesse de montée en température;
- b) méthode de refroidissement et de chauffage.

L'Annexe A fournit des informations supplémentaires concernant le mesurage du RRR. L'Annexe B présente 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 composites supraconducteurs.

Annexe A (informative)

Informations supplémentaires concernant le mesurage du RRR

A.1 Recommandation concernant l'orientation du montage de l'éprouvette

Lorsque l'éprouvette se présente sous la forme d'un fil rectiligne, un montage horizontal du fil sur l'embase est recommandé, car cette orientation du montage peut diminuer le gradient thermique possible le long du fil par comparaison à l'orientation verticale du montage. Dans le cas présent, orientation horizontale du montage signifie que l'axe du fil est parallèle à la surface de l'hélium liquide.

A.2 Autres méthodes pour augmenter la température d'une éprouvette au-dessus de la température de transition supraconductrice

Les méthodes suivantes sont également recommandées pour augmenter la température au-dessus de la valeur de transition supraconductrice de l'éprouvette. Pour ces méthodes, il convient d'appliquer une vitesse de montée en température de l'ensemble de l'éprouvette comprise entre 0,1 K/min et 10 K/min. Pour ralentir la vitesse de montée en température et éviter un gradient de température élevé, il convient de choisir avec soin la puissance et la capacité thermiques (éprouvette avec mandrin de mesure ou embase de mesure), ainsi que la distance entre le réchauffeur et l'éprouvette.

a) Méthode du réchauffeur

L'éprouvette peut être chauffée au-dessus de la valeur de transition supraconductrice par un réchauffeur monté dans le mandrin de mesure ou l'embase de mesure après avoir retiré l'éprouvette du bain d'hélium liquide dans le cryostat.

b) Méthodes contrôlées

1) Méthode adiabatique

Dans cette méthode, le cryostat maintient une chambre qui contient l'éprouvette, un support d'échantillon, un réchauffeur, etc. Avant d'immerger la chambre dans le bain d'hélium liquide, l'air de la chambre est expulsé pour la remplir ensuite d'hélium. La chambre est ensuite immergée dans le bain d'hélium liquide et l'éprouvette est refroidie à une température inférieure à la température critique. Après l'expulsion de l'hélium, l'éprouvette peut être chauffée au-dessus de la valeur de transition supraconductrice par le réchauffeur sous condition adiabatique.

2) Méthode quasi adiabatique

Dans cette méthode, le cryostat maintient l'éprouvette à une certaine distance au-dessus du bain d'hélium liquide tout au long du mesurage à température cryogénique. Une pièce d'ancrage thermique allant du mandrin de mesure ou de l'embase de mesure au bain d'hélium liquide permet de refroidir l'éprouvette à une température inférieure à la température critique. L'éprouvette peut être chauffée au-dessus de la valeur de transition supraconductrice par un réchauffeur situé dans le mandrin de mesure ou l'embase de mesure sous condition quasi adiabatique.

3) Méthode du réfrigérateur

Dans cette méthode, un dispositif électromécanique (réfrigérateur) est utilisé pour refroidir l'éprouvette, qui est montée sur un mandrin de mesure ou une embase de mesure, à une température inférieure à la température critique. L'éprouvette peut être chauffée au-dessus de la valeur de transition supraconductrice au moyen d'un réchauffeur ou en contrôlant la puissance du réfrigérateur.