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# **Laboratory Manual**

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1. Laboratory policy: Accuracy, Reliability, Speciality

#### 2. An overview:

- 2.1 The laboratory belongs to the quality department of SUPER INSTRUMENT LTD(SICC), it can meet the raw materials procurement acceptance, product development and mass production until the final product testing. We have advanced testing, testing equipment and professional inspection personnel, the test results are true and reliable by ISO9001 system.
- 2.2 Laboratory inspection personnel must be trained and qualified before operation.

## 3. Job responsibilities of laboratory personnel

- 3.1 Job responsibilities of the laboratory leader
  - 3.1.1 Implement international and domestic policies, regulations and decrees related to standard and quality.
  - 3.1.2 Ensure the equipment of laboratory equipment and instruments, the quality of personnel and the environmental conditions. Organize the training and assessment of personnel.
  - 3.1.3 Be fully responsible for the impartiality, independence and scientificity of laboratory tests.
  - 3.1.4 Responsible for the preparation of laboratory long-term plans.
  - 3.1.5 Responsible for reviewing the internal documents such as test specifications and operating procedures.
  - 3.1.6 Responsible for reviewing test reports.
  - 3.1.7 Responsible for labor protection and personnel safety in the laboratory.
  - 3.1.8 Coordinate all laboratory work to ensure test quality.
- 3.2 Job responsibilities of test technicians
  - 3.2.1 Seriously study the measurement technology and implement the new testing procedures.
  - 3.2.2 Responsible for the correctness of the test methods and results.
  - 3.2.3 Undertake to make technical appraisals and sign disposal opinions on degradation and scrapping of instruments.
  - 3.2.4 Responsible for submitting test instruments for inspection and keeping verification certificates.

#### 4. Testing and quality control

- 4.1 Test and quality control of thermocouples and resistance wires shall be carried out in accordance with SICC's product inspection procedures.
  - 4.1.1 The diameter and roundness of wires shall be measured in two vertical directions of the same section with a micrometer with a resolution of not less than 0.01mm. Each coil of wires shall measure at least three different points.
  - 4.1.2 The surface of the wires shall be uniform in color, bright and clean, without oil stain, folding, crack, burr and interlayer. Small scratches, depressions and individual dark spots not exceeding the diameter tolerance are allowed.
  - 4.1.3 When the temperature at the reference end is 0°,the relationship between temperature and thermoelectric EMF shall meet the standard of GB/T 16839.1-1997, the calibration tolerance of IEC584 and ASTM E230, check the parameters below:



Type K

	EMF(mv)										
Specification	100°C	200°C	400°C	600°C	800°C	1000°C					
NiCr(KP)	2.826~ 2.886	5.949~ 6.007	12.729~ 12.821	19.532~ 19.676	26.064~26.246	32.313~32.525					
NiSi(KN)	1.224~ 1.256	2.145- 2.175	3.600~ 3.644	5.271~ 5.331	7.080~ 7.160	8.807~8.907					
NiCr-NiSi	4.050~ 4.142	8.094~ 8.182	16.329~16.465	24.803~25.007	33.144~33.406	41.120~41.432					

Physical parameters

Constitution	Density	ND/9C	TC()	E 1 2 2 1 111 /0/\	Resistivity
Specification	(g/cm3)	MP(°C)	TS(mpa)	Extensibility(%)	(20°CΩ.m)
NiCr(KP)	8.6	1427	≥490	≥10	0.71
NiSi(KN)	8.6	1360	≥390	≥15	0.3

Type J

EMF(mv)										
Specification 100°C 200°C 300°C 400°C 600°C										
Fe-CuNi	5.207~ 5.331	10.717~ 10.841	16.244~16.410	21.760~21.936	32.956~33.236					

Physical parameters

Specification	Density (g/cm3)	MP (°C)	TS(mpa)	Extensibility	Resistivity (20°CμΩ.m)
Fe(JP)	7.8	1407	≥240	≥20	0.12
CuNi(JN)	8.8	1220	≥390	≥25	0.49

Type N

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Specification	100°C	200°C	400°C	600°C	800°C						
NiCrSi(NP)	CrSi(NP) 1.755~ 1.813 3.910~3.97		8.880~ 8.959	14.307~14.433	20.012~ 20.180						
NiCrMg(NN)	0.975~1.005	1.954~ 1.988	4.035~4.075	6.212~ 6.274	8.318~ 8.402						
NiCrSi-NiCrMg	2.730~2.818	5.864~5.962	12.915~13.033	20.519~20.707	28.330~28.582						

Physical parameters

Specification	Density (g/cm3)	MP(°C)	TS (mpa)	Extensibility	Resistivity (20°CμΩ.m)
NiCrSi(NP)	8.5	1410	≥620	≥25	0.97
NiCrMg(NN)	8.6	1340	≥550	≥30	0.33



Type E

EMF(mv)										
Specification	100°C	200°C	400°C	600°C	800°C					
NiCr(EP)	2.784~2.844	5.938~ 6.002	12.709~ 12.819	19.537~ 19.699	26.102~ 26.308					
CuNi(EN)	3.467~3.543	7.410~ 7.492	16.109~ 16.255	25.362~25.588	34.664~34.960					
NiCr-CuNi	6.251~6.387	13.348~13.494	28.818~29.074	44.899~45.287	60.766~ 61.268					

Physical parameters

Specification	Density (g/cm3)	MP(°C)	TS(mpa)	Extensibility	Resistivity (20°CμΩ.m)
NiCr(EP)	8.5	1427	≥490	≥10	0.71
CuNi(EN)	8.5	1220	≥390	≥25	0.50

Type T

EMF(mv)									
Specification	100°C	200°C	300°C						
Cu(TP)	0.763~ 0.783	1.826~ 1.846	3.136~ 3.146						
CuNi(TN)	3.466~ 3.542	7.412~ 7.488	11.655~ 11.771						
Cu-CuNi	4.229~ 4.325	9.238~ 9.334	14.790~ 14.930						

Physical parameters

Specification	Density (g/cm3)	MP(°C)	TS (mpa)	Extensibility	Resistivity (20°CμΩ.m)
Cu(TP)	9	1084	≥190	≥20	0.018
CuNi(TN)	8.9	1220	≥390	≥25	0.50

Resitance wirre 's physical parameters

Specification	Resistivity (20°CμΩ.m)	CE (α×10-6/°C)	MP(°C)	TCR(ppm/°C)	Max of T(°C)
Cr20Ni80	1.09 ± 0.05	14	1400	85	1000
Cr15Ni60	1.10±0.05	17	1390	140	950
Cr20Ni30	1.05±0.05	19	1390	280	950

Chemical composition(%)

Specification	Cr	Ni	Fe	Al	Si	С	Р	S	Mn
Cr20Ni80	20.0-23.00	BAL.	≤1.0	≤0.50	0.75-1.60	≤0.08	≤0.02	≤0.015	≤0.60
Cr15Ni60	15.0- 18.0	55.0-61.0	BAL.	≤0.50	0.75-1.60	≤0.08	≤0.02	≤0.015	≤0.60
Cr20Ni30	18.0- 21.00	30.0-34.0	BAL.		1.00-2.00	≤0.08	≤0.02	≤0.015	≤1.00



# 1.1. Test and inspection of mineral thermocouple cables

4.2.1Scope: This regulation is applicable to the inspection of thermocouple cables whose measuring range is from 80  $^{\circ}$ C to 1200 $^{\circ}$ C and the length is not less than 500mm.

4.2.2Check the geometry and appearance with steel tape measure, vernier caliper and visual inspection. The outer diameter wall thickness and coupling wire diameter of the product shall conform to the following table (size:mm).

Outer diameter D Wall thickness Minimum		Wire diameter Minimum	Insulation thickness Minimum
0.5 + / - 0. 025	0.05	0.08	0.04
1.0 + / - 0. 025	0.1	0.15	0.08
1.5+ / - 0. 025	0.15	0.23	0.12
2.0+ / - 0. 025	0.2	0.3	0.16
3.0 + / - 0. 030	0.3	0.45	0.24
4.5 + / - 0. 045	0.45	0.68	0.36
6.0+ / - 0. 060	0.6	0.9	0.48
8.0+ / - 0. 080	0.8	1.2	0.64

<sup>4.2.3</sup> Appearance inspection should meet the following requirements: the surface of the thermocouple should be smooth, no pores, no slag. Thermocouple electrode should be flat, no crack, diameter should be uniform.

The thermocouple qualified by the appearance inspection shall be sampled or calibrated at the temperature of the highest test point for 2 hours after annealing and calibrated with the furnace cooling to below  $250^{\circ}$ C before verifying the value indicated.

Thermocouple cable verification: by comparison method. Please refer to JJF 351-2017 for details.

Note: the welding of the measuring end of the thermocouple should be firm and spherical 4.2.5 The classification type of thermocouples are K, N, E, J, T. Different grades of thermocouple in the specified temperature range, its tolerance in accordance with the following table, and meet the requirements of class 1 as qualified products.

<sup>4.2.4</sup> Calibration of thermoelectric EMF and temperature deviation of thermocouple.



Tolerance values of the thermocouples per IEC 60584-2 / ASTM E230 (Reference temperature 0 °C)

Model	Thermocouple	Tolerance value per	Class	Temperature range	Tolerance value
	NiCr-NiAl (NiCr-Ni)	IEC 60584 part 2	1	-40 +1000 °C	±1.5 °C or 0.0040 •   t   1)2)
K			2	-40 +1200 °C	±2.5 °C or 0.0075 •   t
NiCrSi-NiSi	ACTIA F000	Special	0 +1260 °C	±1.1 °C or ±0.4 %	
		ASTM E230	Standard	0 +1260 °C	±2.2 °C or ±0.75 %
J Fe-CuNi		IEC 60584 part 2	1	-40 +750 °C	±1.5 °C or 0.0040 •   t
	Fe-CuNi		2	-40 +750 °C	±2.5 °C or 0.0075 •   t
		ASTM E230	Special	0 +760 °C	±1.1 °C or ±0.4 %
			Standard	0 +760 °C	±2.2 °C or ±0.75 %
E NiCr-CuNi		150 00504 10	1	-40 +800 °C	±1.5 °C or 0.0040 •   t
	NiCr-CuNi	IEC 60584 part 2	2	-40 +900 °C	±2.5 °C or 0.0075 •   t
	NICI-CUNI	ASTM E230	Special	0 +870 °C	±1.0 °C or ±0.4 %
			Standard	0 +870 °C	±1.7 °C or ±0.5 %
T Cu-CuNi			1	-40 +350 °C	±0.5 °C or 0.0040 •   t
		IEC 60584 part 2	2	-40 +350 °C	±1.0 °C or 0.0075 •   t
	Cu Cubii		3	-200 +40 °C	±1.0 °C or 0.015 •   t
	Cu-CuNi	ASTM E230	Special	0 +370 °C	±0.5 °C or ±0.4 %
			Standard	-200 0 °C	±1.0 °C or ±1.5 %
			Standard	0 +370 °C	±1.0 °C or ±0.75 %
		IEC 60584 part 2	1	0 +1600 °C	±1.0 °C or ±[1 + 0.003 (t - 1100)] °C
₹	Pt13%Rh-Pt Pt10%Rh-Pt		2	0 +1600 °C	±1.5 °C or ±0.0025 •   t
3		10TH F000	Special	0 +1480 °C	±0.6 °C or ±0.1 %
		ASTM E230	Standard	0 +1480 °C	±1.5 °C or ±0.25 %
B Pt30	Diggs/ Dis Digg/ Dis	IEC 60584 part 2	2	+600 +1700 °C	±0.0025 •   t
			3	+600 +1700 °C	±4.0 °C or ±0.005 •   t
	Pt30%Rh-Pt6%Rh	ASTM E230	Special	-	14
			Standard	+870 +1700 °C	±0.5 %

- 4.2.6 Measuring insulation resistance: The insulation resistance shall be measured between the wires and the wire and the casing. The measured voltage is  $500V \pm 50V$  for the outer diameter >1.5mm, and  $100V \pm 25V$  for the outer diameter  $\leq 1.5$ mm.
- 4.2 Package mark: manufacturer, order number, specification, batch number, accuracy, EMF, quantity, date.

### 5. Management of measuring instruments and testing equipment

- 5.1 Purchase, warehousing, distribution, circulation and ledger management of measuring instruments.
  - 5.1.1 Purchase of measuring instruments: the laboratory personnel shall submit the demand plan to the purchase department.
  - 5.1.2 Acceptance and warehousing of measuring instruments:before the new measure instruments are into storage, the purchase department shall check and accept the "certificate of measuring instruments" to the laboratory. The unqualified instruments can not be used and handled by the purchasing personnel.
  - 5.1.3 Distribution of measuring instruments: The laboratory shall issue measuring instruments according to the plan reported by each department, each user shall issue the material requisition with the signature of the department leader and the quality department shall issue it after review.
  - 5.1.4 Circulation of measuring instruments: When an employee is transferred with SICC, the measuring instruments for personal use and storage shall be left in the original department. When it is necessary to bring them to the new department, the employee shall submit a report to the quality department and obtain approval.
  - 5.1.5 Accounting management of measuring instruments: each department shall establish its own "Measuring Instrument Management Ledger", and the laboratory shall establish its own by classification. Standards of measurement equipment file should include manufacturer model, specification, attachment list, location and its changes, production date, date of record, verification or calibration, adjustment or repair, damage and loss situation, sequestration and disabled, demotion and discard records, such as the verification or calibration certificate, operation manual, schematic diagram, curriculum vitae and other materials.
  - 5.1.6 Degradation and scrapping of measuring instruments: 5.1.6.1 Measuring instruments for general work that ca
    - 5.1.6.1 Measuring instruments for general work that cannot restore original accuracy after adjustment and modification but can still be used for low accuracy shall be approved for use by the laboratory after degradation, but their technical indicators shall meet the requirements of degradation level. The laboratory shall strengthen the assessment of the technical indicators of the measuring instruments for grading down to ensure product quality and safe production.
    - 5.1.6.2 For the degradation of precision and valuable measuring instruments, the user shall write a report, detailing the reasons for degradation, and the degradation



shall be carried out after technical appraisal by the laboratory and approval by the laboratory.

- 5.1.6.3 When SICC's highest standard of measurement does not meet the requirements of value transfer, the laboratory shall issue a report on the basis of the notice of result issued by the verification institution, and with the consent of the competent leadership, the report shall be submitted to the superior competent metrology department for approval to reduce the use of the company's highest standard of measurement to work measuring instruments.
- 5.1.6.4 Where the measurement instruments approved to be degraded are used within a specified range, color "limited use" signs shall be pasted on the obvious position of the measuring instruments, and the range and point of limited use shall be indicated.
- 5.1.6.5 The degraded measuring instruments shall still be included in the normal weekly inspection management.
- 5.1.6.6 If the measuring instruments for general work are damaged due to long-term use or normal use and are found to be unqualified and unable to be repaired or have no repair value (including the measuring instruments declared to be eliminated by the state), the laboratory shall fill in the "Approval Form for the Scrapping of measuring instruments", and dispose of them after the approval number is obtained, and cancel the bookkeeping and account cards at all levels.
- 5.1.6.7 For the scrapping of precision and valuable measuring instruments, the user shall write a written report detailing the scrapping reason, make technical appraisal by the laboratory, confirm by the leader of the quality department, and report to the leader in charge for approval before scrapping.
- 5.1.6.8 The scrapping of the SICC's highest measuring standard shall be reported by the laboratory, reviewed by the leader of the quality department, and scrapped with the consent of the leader in charge.
- 5.1.6.9 If measuring instruments are lost or damaged and cannot be repaired or have no repair value, they shall be scrapped and all levels of account books and account cards shall be canceled.
- 5.1.6.10 All measuring instruments approved for scrapping shall be strictly stored to prevent them from mixing into the production site, and the laboratory shall be responsible for retrieving them and establishing a scrapping account for measuring instruments.
- 5.2 Management of measuring instrument identification
  - 5.2.1 Classification management and marking management of measuring instruments
    - 5.2. 1.1 classification
    - ① Class A measuring instruments
    - A. The company's highest standard of measurement, general standard and measuring instruments for value transmission and their main supporting measuring instruments.
    - B. Key metering devices used by the company for process, quality testing, energy and business management that require high metering data.
    - (2) Class B measuring instruments
    - A. Measuring instruments with data requirements for process control and quality testing.
    - B. Measuring instruments used in the company's operation and management.
    - 5.2.1.2 Classification Management Requirements
    - (1) Class A measuring instruments
    - A. The measures for compulsory verification of measuring instruments stipulated by the state shall be implemented in accordance with the provisions of the national metrological administrative department and the metrological administrative institution of the national defense industry system.
    - B.Strictly implement the verification regulations and verification cycle stipulated by
    - C. Measuring instrument users must designate special personnel for storage and maintenance, and establish strict use storage and verification adjustment

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D.Periodic inspection rate should reach 100%.



- (2) Class B measuring instruments
- A. The verification shall be carried out according to the period stipulated by the national verification regulations. In principle, the verification period shall not exceed the longest period stipulated by the verification regulations. In special cases, the period shall be extended.
- B. For measuring instruments that are difficult to disassemble on continuously running devices, verification cycles can be arranged synchronously according to verification regulations and reliability data according to equipment maintenance cycles, but strict supervision must be carried out.
- C. General measuring instruments as special measuring instruments, according to their actual use needs, can reduce the verification items or partial verification, but the verification certificate should indicate the limit of dosage, limit range and use place, in the obvious position of the measuring instruments paste limit sign.
- D.For measuring instruments with infrequent use, stable performance and low accuracy requirements, the verification cycle can be appropriately extended, and the length of the extension should be based on ensuring the reliability data of measuring instruments.
- 5.2.1.3 Logo management and requirements
- ① Instructions for the use of measuring color signs
- A. "Qualification certificate" mark: it means that the measuring instrument is qualified according to the national verification system or national defense industry verification system and inspection procedures
- B. "Forbidden" mark: the measurement that fails to be repaired temporarily or exceeds the verification period and fails to pass sampling inspection
- C. "Sealed" mark: used for long-term idle or temporarily not put into use, nor periodic verification of the meter, With the "seal" mark, prevent into production and management use
- 5.3 Quality control of measuring instruments
  - 5.3.1.1 Metrological verification shall be carried out in accordance with the national metrological verification system table and metrological verification regulations.
  - 5.3.1.2 Unverified measuring instruments shall be regarded as unqualified measuring instruments and shall not be used in production and business management.
  - 5.3.1.3 The periodic inspection rate of standard measuring instruments is 100%
  - 5.3.2 First test or calibration
    - 5.3.2.1 Before the measuring instruments are put into use, the laboratory shall entrust a qualified institution to carry out verification or



calibration and obtain inspection. After the certificate is fixed or calibrated, it shall be uniformly numbered, posted and included in the periodic verification plan.

5.3.2.2 The company's highest standards of measurement shall be timely sent by the metrology room to the superior metrology technical department for verification or verification according to the verification cycle. The superior metrological technical department shall come to the factory for verification, and the verification period shall be 6-12 months or the superior metrological technical department shall make other regulations.

5.3.2.3 Verification certificate of periodic verification of measuring instruments, notification of verification results, correction table, etc., and relevant original funds. Materials should be stored in equipment files at any time. If the verification certificate of the measurement instrument submitted for inspection is required, the original shall be kept.

- 5.3.3 In order to ensure that the measuring instruments are always qualified and effective within the verification cycle, the company will calibrate the measuring instruments during the test process of selecting high-end materials (specifically, according to the provisions of the company).
- 5.3.4 Use, storage and maintenance of measuring instruments
  - 5.3.4.1 Requirements for the use of measuring instruments
  - A. Users of each department shall be familiar with the basic principle, performance, operation method and maintenance knowledge of measuring instruments.
  - B. Check and confirm that the measuring instrument is in good condition before operation.
  - C. If there is any abnormality or misalignment in the use of measuring instruments, users should stop using them immediately and submit them to the measuring instrument keeper or instrument personnel of the unit in time for delivery or notification to the laboratory. For precision and valuable measuring instruments and equipment, report to the relevant leaders of the quality department, and the quality department is responsible for handling.

#### 6. Environment Control

- 6.1 Control of laboratory environmental conditions
- 6.1.1 Basic requirements for laboratory environmental conditions
- 6.1.1.1 The standard temperature of the laboratory is  $20^{\circ}$ C, and the temperature of the general testing room and test room should be  $20\pm5^{\circ}$ C.
- 6.1.1.2 The relative humidity in the laboratory should generally be maintained at 50-70%.



- 6.1.1.3 The environmental conditions of noise, shockproof, dustproof, anticorrosion, antimagnetic and shielding in the laboratory shall conform to the verification regulations of verification projects carried out indoors and the requirements of measurement standards and measurement and testing instruments for environmental conditions. Indoor lighting shall be conducive to verification and measurement and testing work.
- 6.1.2 When the environmental conditions of the laboratory are abnormal, such as temperature and humidity exceeding the specified range and obviously affecting the verification or testing results, the director of the laboratory shall be reported in time, and the leaders of the quality Department and relevant leaders shall be reported step by step. When the environmental conditions often appear abnormal or cannot meet the metrological verification and precision measurement work, it shall report to the relevant leaders in writing and take appropriate measures to solve the problem. Under existing conditions, the laboratory should take active measures to maintain sub-measuring equipment and measuring standards.
- 6.1.3 Daily control and management of laboratory environmental conditions
- 6.1.3.1 The laboratory shall be kept neat and clean. Necessary cleaning shall be carried out after work every day, and instruments and accessories shall be cleaned regularly. After use of instruments and equipment, instruments and accessories shall be neatly placed and covered with instrument cover or dustproof cloth. All electric instruments and equipment should be cut off after use.
- 6.1.3.2 Smoking, eating snacks, drinking water and storing food are strictly prohibited in the laboratory. Non-laboratory personnel are not allowed to enter the laboratory without permission. The number of people allowed to enter should be strictly controlled, so as not to cause fluctuations in indoor temperature and humidity.
- 6.1.3.3 The laboratory shall have special personnel responsible for the record of indoor temperature and humidity.Do not open doors and windows for air conditioning and dehumidification equipment. Designate someone to operate air conditioning equipment or dehumidifier. Records of indoor temperature and humidity shall be kept by each measuring and testing room for five years.
  - 6.2 Laboratory security and confidentiality
  - 6.2.1 The laboratory is the place where the company's highest measurement standards and precision and valuable instruments and equipment are stored. It is also an important place for metrological checking or calibration, precise measurement testing and various performance tests. All metrology and testing personnel shall ensure the public safety and technical safety of the laboratory work site, and keep the secrets of the state and the enterprise.
  - 6.2.2 Laboratory personnel shall strictly abide by safety production regulations when carrying out metrological verification or testing, adjustment or test.
  - 6.2.3 The laboratory shall do a good job of confidentiality, anti-theft and fire



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prevention, regularly check whether public safety facilities are in good condition and reliable, and keep doors, Windows, water and electricity well when off duty.

6.2.4 The laboratory shall keep confidential the relevant data required by the client.