

INTERNATIONAL
STANDARD

ISO
9950

First edition
1995-05-01

**Industrial quenching oils — Determination
of cooling characteristics — Nickel-alloy
probe test method**

*Huiles industrielles pour trempe — Détermination des caractéristiques de
refroidissement. — Méthode d'essai à la sonde en alliage de nickel*



Reference number
ISO 9950:1995(E)

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International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 9950 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

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Introduction

When hardening steel, quenching is normally the most critical phase of the hardening process. For the quenching, the heat treater has to select among different types of quenching media, such as oils, water-based polymer solutions, emulsions, etc. In addition, a large variety of quenchants is available commercially for each type of medium. Moreover, the cooling characteristics of each quenching medium can change in service due to thermal degradation, contamination, drag-out, etc.

A number of methods are used to evaluate quenching media. Indirect testing, involving hardening of test pieces of a certain steel, gives only limited information about the cooling characteristics. The most common method for direct testing is the so-called silver ball method, where a silver ball (probe) with a thermocouple at its centre is heated and quenched in the quenching medium in question. The temperature is recorded as a function of time, and often also the cooling rate as a function of temperature (or time).

Due mainly to difficulties concerning the silver ball probe manufacture and the assessment of test results, several modified probes have been used but with the same basic method. The probes have been made of various materials and different sizes, the shape normally being cylindrical.

In order to make comparison possible among test results received from different laboratories and among technical descriptions of quenching media provided from different suppliers, it is necessary to use a standard method of testing. Therefore, the International Federation for the Heat Treatment of Materials (IFHT) Technical Committee, *Scientific and Technological Aspects of Quenching*, has evaluated different existing methods and arranged tests in several countries in order to arrive at an agreement about a method to be recommended as a standard. The specification in this International Standard is the result of the work within this committee.

NOTE 1 This standard method is based on, and is identical in all principal details with, a method drawn up by a working party of the Wolfson Heat Treatment Centre Engineering Group, Aston University, Birmingham, UK. The deviation between the two methods concerns the measurement system, where a computer-based system for recording and differentiation of the thermocouple signal has been added as an alternative in the method described in this International Standard.

Industrial quenching oils — Determination of cooling characteristics — Nickel-alloy probe test method

1 Scope

This International Standard specifies a laboratory test using a nickel-alloy probe for the determination of the cooling characteristics of industrial quenching oils. The test is conducted in non-agitated oils and thus is able to rank the cooling characteristics of the different oils under standard conditions. No correlation can be made between the results of this test and the quench rates in industrial quench installations in which varying degrees of agitation are present.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 2719:1988, *Petroleum products and lubricants — Determination of flash point — Pensky-Martens closed cup method.*

ISO 2909:1981, *Petroleum products — Calculation of viscosity index from kinematic viscosity.*

ISO 3104:1994, *Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity.*

ISO 3405:1988, *Petroleum products — Determination of distillation characteristics.*

ISO 3675:1993, *Crude petroleum and liquid petroleum products — Laboratory determination of density or relative density — Hydrometer method.*

British Standard BS 1041, Part 4:1966, *Thermocouples.*

British Standard BS 4937, Part 4:1973, *Nickel-chromium/nickel-aluminium thermocouples. Type K.*

3 Principle

A cylindrical nickel-alloy test piece ("probe") having a thermocouple at its geometric centre is heated in a furnace to the specified temperature, and then transferred into a fixed volume of the quenching oil under test. The change in temperature at the centre of the probe is recorded as a function of time.

The cooling rate may be recorded simultaneously, or determined afterwards.

Measurements taken from these records are used to evaluate the quenching oil under test.

4 Reference quenching fluid

4.1 General

A reference quenching fluid is recommended for initial, and regular, cross-checking of the probe (see 5.2). The fluid shall be stored in a sealed container when not in use and shall be renewed after 200 quenches or two years, whichever is sooner.

4.2 Physical characteristics

The reference quenching fluid shall be an unblended straight run, high-viscosity-index paraffinic mineral oil completely free of any additive treatment. It shall have the physical characteristics listed in table 1.

4.3 Cooling characteristics

The reference quenching fluid shall exhibit cooling characteristics with average values within the limits listed in table 2 when subjected to the standard quench test.

5 Apparatus

5.1 Containing vessel

The sample shall be contained in a previously cleaned and dried tall-form vessel having a diameter of

115 mm \pm 5 mm and preferably manufactured from unbreakable material.

5.2 Thermal probe

5.2.1 General

The thermal probe consists of a nickel-alloy cylinder having a thermocouple located at its geometric centre and mounted upon a nickel-alloy support tube (see figure 1).

5.2.2 Probe dimensions

The probe shall have a diameter of 12,5 mm and a length of 60 mm. The thermocouple hot junction shall be located at its geometric centre [see figure 1 b)].

Table 1 — Physical characteristics of the reference quenching fluid

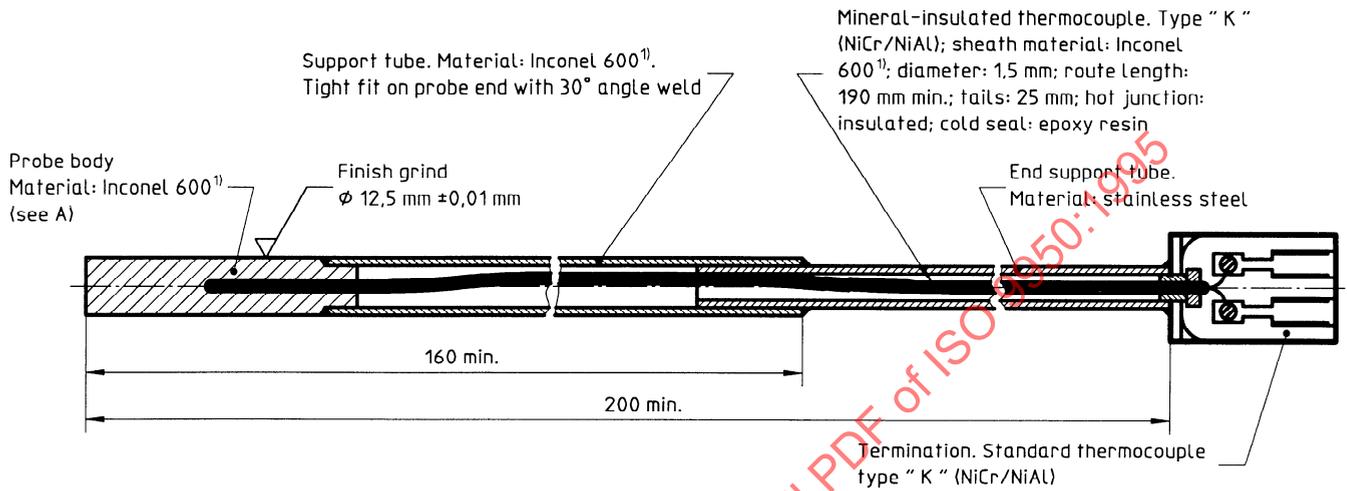
Physical characteristic	ISO test method	Minimum value	Maximum value
Kinematic viscosity at 40 °C, cSt ¹⁾	ISO 3104	19,0	23,0
Kinematic viscosity at 100 °C, cSt ¹⁾	ISO 3104	3,9	4,4
Kinematic viscosity index	ISO 2909	95	105
Density at 15 °C, kg/l	ISO 3675	0,855	0,870
Flash point, Pensky-Martens closed cup, °C	ISO 2719	190	210
5 % distillation, °C	ISO 3405	330	360
50 % distillation, °C	ISO 3405	400	420
Ash, %	—	2	8

1) 1 cSt = 10⁻⁶ m²/s.

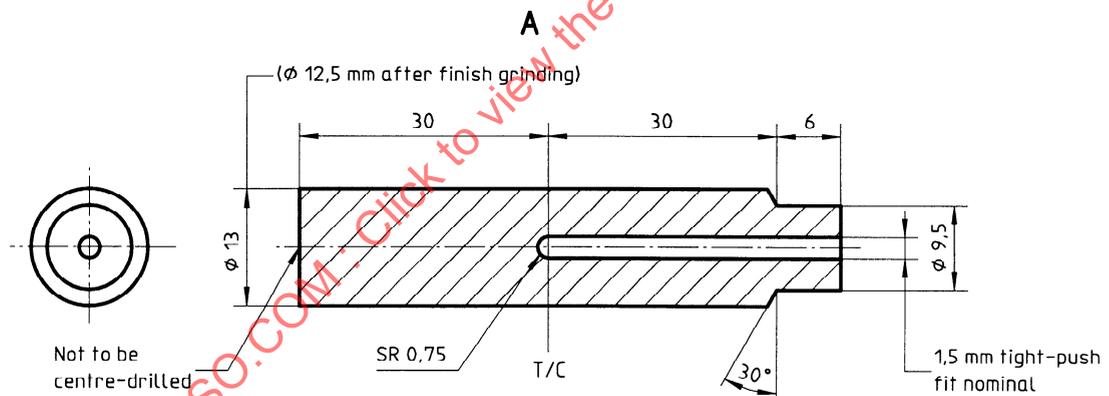
Table 2 — Cooling characteristics of the reference quenching fluid

Cooling characteristic	Minimum value	Maximum value
Maximum cooling rate, °C/s	47,0	53,0
Temperature at maximum cooling rate, °C	490	530
Cooling rate at 300 °C, °C/s	6,0	8,0
Time, in seconds, from immersion temperature to		
a) 600 °C	12,0	14,0
b) 400 °C	19,0	21,0
c) 200 °C	50,0	55,0

Dimensions in millimetres
 All tolerances $\pm 0,25$ except where otherwise stated
 All diameters to be concentric



a) General assembly



b) Probe details

1) Or equal alloy.

Figure 1 — Thermal probe

5.2.3 Probe materials

The probe shall be manufactured from Inconel 600¹⁾ grade nickel-chromium-iron alloy or other alloy with equal physical and thermal properties.

5.2.4 Thermocouple

The probe temperature shall be measured by a metal-sheathed mineral-insulated chromel/alumel thermocouple of the insulated junction type. The thermocouple shall have an outside diameter of

1) Nominal composition (mass fraction, expressed as percent): 72 min. Ni + Co; 14-17 Cr; 6-10 Fe; 0,15 max. C; 1 max. Mn; 0,015 max. S; 0,5 max. Si; 0,5 max. Cu.

Inconel is the tradename of a product supplied by the Inco group of companies. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

1,5 mm and be provided with an Inconel 600 alloy outer sheath.

5.2.5 Thermocouple support tube

The thermocouple support tube shall be manufactured from 12,5 mm o.d. Inconel 600 or equal alloy tube. Together, the probe plus support tube shall have a minimum length of 200 mm and a typical length of 355 mm; extended support tubes may be used for mechanical transfer reasons if necessary. The support tube may be reduced to 10,0 mm o.d. stainless steel at 160 mm from the end of the probe, if desired [see figure 1 a)].

5.2.6 Assembly

The thermal probe shall be assembled in accordance with figure 1 a). The thermocouple shall be a tight-push fit into the probe body, and shall be inserted prior to the assembly of the other components to ensure that the thermocouple tip is correctly located at the base of the machined hole.

The outer thermocouple support tube shall be TIG-welded to the probe body. TIG welding shall also be used to join the support tube elements in the stepped design.

In order to optimize probe life, it is recommended that the thermocouple be terminated in a plug-and-socket connection of the type shown. It is important that the thermocouple shall be of sufficient length to accommodate thermal expansion.

5.2.7 Surface condition of probe

5.2.7.1 Conditioning of new probes

The probe shall be conditioned prior to its initial use with any quenching oil by carrying out a minimum of six dummy quenches, or a greater number if required to achieve a constant result, in a general-purpose straight hydrocarbon oil from 850 °C, cleaning between quenches being carried out as specified in 5.2.7.2.

5.2.7.2 Cleaning

Upon completion of each quenching test, the probe shall be removed from the oil and allowed to cool below 50 °C. The probe surface shall be cleaned using an appropriate chlorinated solvent, followed by wiping with a dry lint-free cloth.

5.2.7.3 Reconditioning

Reconditioning shall be conducted when the maximum rate of cooling deviates by more than $\pm 5\%$ from the constant value obtained after initial conditioning (see 5.2.7.1).

The probe may be reconditioned by cleaning off loose scale with 600-grit emery paper followed by a sufficient number of dummy quenches (at least six) in oil from 850 °C until a continuous oxide film is formed on the probe and repeatable results, within the range obtained after the initial conditioning, are achieved.

5.3 Heating equipment

5.3.1 Furnace

The heating furnace shall be of the resistance-heated tube type, and shall be either horizontally or vertically mounted. The furnace shall be capable of maintaining a constant temperature over a heating zone of not less than 120 mm; the probe shall be positioned at the centre of the heating zone such that the probe temperature does not vary more than $\pm 2,5\text{ °C}$ over the 60 mm length.

5.3.2 Temperature controller

The temperature controller employed shall be capable of maintaining the heating zone of the furnace at a temperature of $850\text{ °C} \pm 5\text{ °C}$ during the soaking period.

5.3.3 Sample heater

The quenching oil sample shall be heated within the containing vessel (5.1), preferably by an internal electric resistance heater.

5.4 Transfer mechanism

Transfer of the probe (5.2) from the furnace (5.3.1) to the containing vessel (5.1) shall be made in not more than 2 s, preferably with an automated mechanism. The probe shall be positioned at the geometric centre of the quenching oil sample volume, for example with the aid of a mechanical support to prevent vibration and agitation. An electrically-activated event marker shall be incorporated into the system to indicate, on the time/temperature trace, the instant of probe contact with the oil.

5.5 Measurement system

5.5.1 General

The measurement system shall provide a permanent record of the cooling characteristics of each quenching oil sample tested, producing a record of variation in test probe temperature with respect to time, and cooling rate with respect to temperature (see 8.1).

The records may be produced using either of two methods: a) a computer technique, or b) a standard recording technique with electronic differentiation.

5.5.1.1 Computer technique

The probe thermocouple output is sampled, digitized and stored in the memory of the computer. The frequency of sampling shall be not less than 20 s^{-1} .

The plot of temperature as a function of time is produced either on-line during the test period or off-line after the test, using a plotter.

The cooling rate is calculated by numerical differentiation of the probe thermocouple output with respect to time. This rate is plotted as a function of probe temperature.

5.5.1.2 Standard recording technique

The plot of temperature as a function of time is produced by recording the probe thermocouple output using a Y-t recorder.

The trace of cooling rate as a function of temperature requires an electronic differentiator, details of which are given in figure 2, to produce a rate signal proportional to the variation in probe thermocouple output with time. Using an X-Y plotter, this rate signal is recorded relative to probe temperature.

5.5.2 Freedom from bias

The overall freedom from bias of the measurement system shall be not worse than $\pm 2,5 \%$ of the recorded value *excluding* thermocouple calibration effects.

The following equipment specification represents the *minimum* requirements.

5.5.2.1 Differentiator

The electronic differentiator (figure 2) shall meet the following specifications:

- it shall accept an input of at least 20 mV/s ;
- at an input of 20 mV/s , the output shall be not less than 100 mV with a temperature coefficient of the time constant of not more than $\pm 100 \text{ ppm}$ per degree Celsius;
- it shall incorporate a high-frequency cutoff with a time constant of not more than $0,1 \text{ s}$;
- the drift of the output voltage shall not exceed 1% of full scale over a period of 15 min .

5.5.2.2 Recording equipment

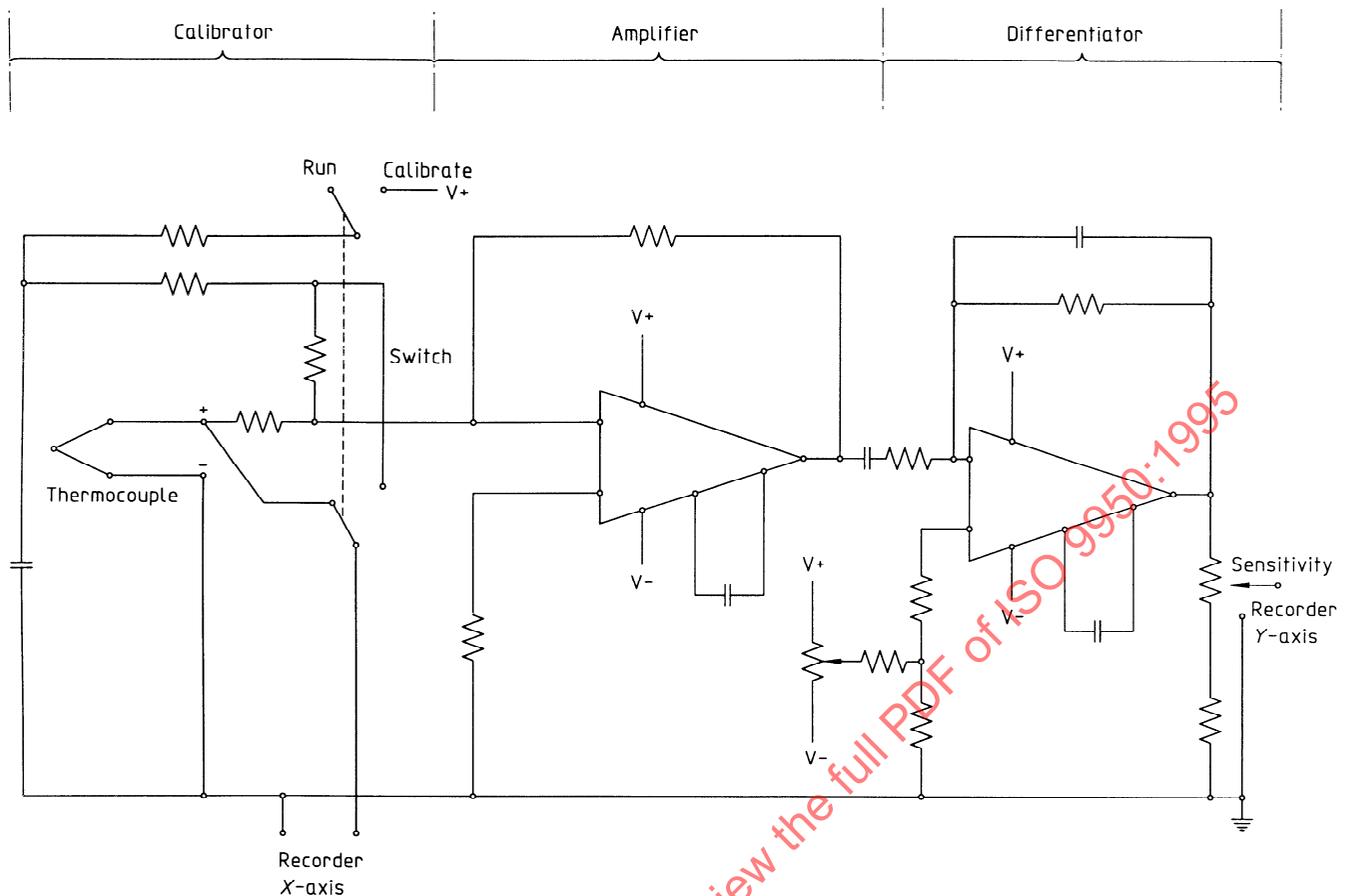
5.5.2.2.1 General

For an accurate analysis of recorded data, it is considered desirable for all test results to be drawn on graphs of standard scale calibration. Consequently the preferred system, specifying this calibration, is described in 5.5.2.2.2 and 5.5.2.2.3. However, alternative Y-t recorder systems are suggested in 5.5.2.2.4 which permit flexibility in the choice of equipment.

5.5.2.2.2 Cooling rate as a function of temperature

An X-Y recorder to record cooling rate as a function of temperature shall meet the following minimum specifications.

- Sensitivity:
temperature: $0,2 \text{ mV/mm}$;
rate: 5 mV/s or 10 mV/s or 20 mV/s shall occupy 250 mm .
- Linearity: equal to or better than $0,1 \%$.
- Repeatability: equal to or better than $0,5 \%$.
- Writing speed: minimum of 200 mm/s .



NOTE — Temperature coefficient of all resistors and capacitors: ± 100 ppm per degree Celsius.

Figure 2 — Typical circuit including differentiator

5.5.2.2.3 Temperature as a function of time

A $Y-t$ recorder to record temperature as a function of time shall meet the following minimum specifications.

- Sensitivity: equal to or better than 0,2 mV/mm.
- Linearity: equal to or better than 0,1 %.
- Accuracy: equal to or better than 0,5 %.
- Writing speed: minimum of 200 mm/s.
- Chart speed accuracy: 1 %.
- The equipment shall incorporate an event marker facility to record the *instant* of probe contact with the quenching oil.

5.5.2.2.4 Alternative systems for $Y-t$ recorders

One two-pen or two one-pen $Y-t$ recorders may be used as alternatives. The recorders shall have characteristics equivalent to those specified in 5.5.2.2.3.

5.6 Potentiometer, for checking the span of the temperature and rate axes of recorder and differentiator (see 5.5).

5.7 Stop-watch, for checking the chart speed of the recorder and differentiator (see 5.5).

6 Sampling

A quenching oil sample volume of 2 litres shall be used. Care shall be taken to ensure that the sample is representative and that the sampling vessel is clean and dry in order to avoid contamination.

7 Test procedure

7.1 Number of tests

Tests shall be repeated twice in the same quenching oil sample to obtain triplicate results.

7.2 Test temperatures

7.2.1 Probe temperature

The standard probe temperature shall be 850 °C. The probe shall be heated in air to a temperature of 850 °C \pm 5 °C, and soaked at this temperature for a period of 5 min prior to the commencement of the test.

The probe temperature shall be determined for each test by reference to the temperature axis of the Y-t chart recorder (see 5.5.2.2.3), making due allowance for the cold junction correction and any error shown in the calibration of the probe thermocouple (see 7.3.1). The cold junction and probe thermocouple calibration corrections shall be recorded on the chart prior to each test.

7.2.2 Sample temperature

The quenching oil sample shall be tested within the temperature range defined as the operating limits for the specific product.

Quenching oil temperatures may be chosen to suit individual requirements, but to facilitate comparisons it is recommended that tests be carried out with the oil at 40 °C, unless otherwise specified.

The quenching oil temperature at the start of the test shall be within \pm 2 °C of the intended test temperature.

The quenching oil sample shall be adequately agitated during the heating phase and when checking its temperature at the start of the test, in order to minimize thermal gradients. The test shall be carried out in still oil.

7.3 Calibration

7.3.1 Probe thermocouple calibration

7.3.1.1 General

Check the calibration of the probe thermocouple (5.2.4) by attaching a previously-calibrated thermocouple to the outer surface of the probe, and heating the probe to the test temperature. Once the probe temperature has been allowed time to equalize, compare the outputs of both thermocouples by means of a potentiometer. Discard the probe if the probe thermocouple output error exceeds \pm 2 % of the quoted e.m.f. for the measured test temperature in the range 850 °C \pm 5 °C. The correction of smaller errors is described in 7.3.1.3.

7.3.1.2 Frequency of recalibration

Carry out the calibration procedure in 7.3.1.1 on a new probe prior to use, on used probes prior to the commencement of a series of tests not exceeding 25 in number or, for probes in regular use, after every 25 tests.

7.3.1.3 Compensation for errors

Note the errors determined during the calibration and allow for them in the determination of the probe temperature prior to the commencement of each test, and in the determination of data for recording of results.

7.3.2 Recorder and differentiator calibration

7.3.2.1 Temperature axis

Check the zero setting prior to the commencement of each test. Check the chart span by means of the potentiometer (5.6) prior to the commencement of each uninterrupted series of tests, such that an input of 34 mV gives a deflection of 170 mm.

7.3.2.2 Rate axis

The differentiator shall either:

- a) incorporate an integral calibration facility,
- or
- b) be capable of calibration by external test equipment.

Check the calibration of the differentiator prior to the commencement of each uninterrupted series of tests. An input rate of 5 mV/s, 10 mV/s or 20 mV/s shall result in a preferred chart span of 250 mm.

Check the zero setting prior to the commencement of each test.

7.3.2.3 Time axis

Check the chart speed by means of a stop-watch (5.7) at the commencement of each series of tests.

7.4 Test

Introduce the sample of the quenching oil to be tested (6) into the containing vessel (see 5.1) and, if necessary, adjust to the required test temperature (see 7.2.2). Agitate the quenching oil sample during the heating period to minimize thermal gradients.

Insert the previously conditioned, cleaned and calibrated probe (5.2) into the furnace (5.3.1) and heat to the required temperature (7.2.1).

Switch off the furnace power before quenching [to prevent possible interference of the control system (see 5.3.2)], lower the recorder pens and transfer the probe from the furnace to the quenching oil sample, ensuring that the event marker (see 5.4) records the probe/oil contact.

When the probe temperature has fallen to 200 °C, or such lower temperature as may be required for specific tests, switch off the recorders and differentiator (see 5.5). Remove the probe from the oil and carry out the specified cleaning procedures (see 5.2.7).

8 Expression of results

8.1 Cooling curves

Plot the temperature as a function of time and the temperature as a function of cooling rate, for example in linear diagrams as shown in figure 3.

8.2 Cooling time

From the plot of temperature as a function of time (see 8.1), read the following data, correcting for cold junction and thermocouple calibration factors as indicated in 7.3.1.2.

Time required for probe temperature to drop from immersion temperature to:

- a) 600 °C (to the nearest 0,5 s);
- b) 400 °C (to the nearest 0,5 s);
- c) 200 °C (to the nearest 1 s).

8.3 Cooling rates

From the plot of temperature as a function of cooling rate (see 8.1), read the following data, correcting where appropriate for cold junction and thermocouple calibration factors as indicated in 7.3.1.2.

- a) Maximum cooling rate (to the nearest 0,5 °C/s);
- b) temperature at which the maximum cooling rate occurs (to the nearest 0,5 °C);
- c) cooling rate at 300 °C (to the nearest 0,5 °C/s).

9 Test report

The test report shall contain the following information:

- a) complete identification of the quenching oil sample;
- b) a reference to this International Standard, i.e. ISO 9950:1995;
- c) the results and the method of expression used (see clause 8);
- d) any unusual features noted during the determination;
- e) any deviation, by agreement or otherwise, from the procedure specified.