

BS ISO 18089:2015



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Corrosion of metals and alloys — Determination of the critical crevice temperature (CCT) for stainless steels under potentiostatic control

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National foreword

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**Corrosion of metals and alloys —
Determination of the critical crevice
temperature (CCT) for stainless steels
under potentiostatic control**

Corrosion des métaux et alliages — Détermination de la température critique de corrosion caverneuse (TCCC) des aciers inoxydables sous contrôle potentiostatique





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Foreword

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The committee responsible for this document is ISO/TC 156, *Corrosion of metals and alloys*.

Introduction

Stainless steel is susceptible to pitting corrosion, crevice corrosion, and stress-corrosion cracking, etc., although it is used as generally corrosion-resistant material. The basic methodology for testing localized corrosion was first standardized in ASTM G 150. This method describes the susceptible to pitting corrosion and it is also standardized in ISO 17864. In this International Standard, the susceptible to crevice corrosion is examined. This is performed by recording the electrochemical critical crevice corrosion temperature for a material using a specific crevice former. Crevice corrosion phenomenon is generally of a random nature and therefore these measurements require at least a couple of values.

Corrosion of metals and alloys — Determination of the critical crevice temperature (CCT) for stainless steels under potentiostatic control

1 Scope

This International Standard describes the procedure for determining the critical crevice temperature (CCT) for stainless steels under potentiostatic control.

The principal advantage of the test is the rapidity with which the CCT can be measured in a single test procedure. The CCT, as determined in this International Standard, can be used as a relative index of performance, for example, to compare the relative performance of different grades of stainless steel.

The test described in this International Standard is not intended to determine the temperature at which crevice corrosion will occur in service.

This method is not intended for materials with critical pitting temperature (CPT) values below 20 °C measured in accordance with ISO 17864, when measured in the same test solution and at the same potential

2 Normative references

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

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 8044, *Corrosion of metals and alloys — Basic terms and definitions*

ISO 17864, *Corrosion of metals and alloys — Determination of the critical pitting temperature under potentiostatic control*

ISO 18070, *Corrosion of metals and alloys — Crevice corrosion formers with disc springs for flat specimens or tubes of stainless steels in corrosive solutions*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 8044, ISO 17864 and the following apply.

3.1

critical crevice temperature

CCT

lowest temperature on the surface of the specimen at which stable propagating crevice corrosion occurs under specified test conditions

Note 1 to entry: The critical crevice corrosion temperature is defined as the temperature of the specimen at which the current density exceeds a specified value. A recommended value is 10 $\mu\text{A cm}^{-2}$, referring to area exposes in the measurement to make sure that it is above the passive current, for 60 s. A 60 s delay is used in order to ensure that the observed current increase originates from stable propagating crevice corrosion, and not a short-lived current peak.^[4]

3.2 temperature ramp rate

rate at which the temperature of the surface of the specimen is increased during the test

4 Principle

4.1 The test involves increasing the temperature of the surface of the specimen at a specified rate, while exposing the specimen to a solution and maintaining the potential of the specimen at a specified value. The temperature of the surface of the specimen is increased by heating the solution.

4.2 The temperature ramp rate, environment and applied potential may be varied, depending on the material.

5 Equipment

5.1 Experimental set-up is a vessel with immersion heater or a thermostatic bath.

5.1.1 The test specimen, a reference electrode connected appropriately for measuring the electrode potential, see [5.10](#), an auxiliary electrode, a port for insertion of a temperature-measuring device and a facility for stirring the solution in a repeatable manner.

NOTE This can be achieved using a mechanical stirring device, using a peristaltic pump or by bubbling inert gas through the solution at a controlled rate.

5.1.2 Any part of the test cell or specimen holder that comes into contact with the solution shall be constructed from an inert material. The connection of the electrode shall be design so no corrosion occurs in the connection point.

NOTE 1 Examples of how the connections can be made are given in [Annex A](#).

NOTE 2 Polycarbonates, glass, polytetrafluoroethylene (PTFE), polypropylene (PP), polyethylene (HD-PE) are suitable materials but styrolic plastics are not allowed.

5.1.3 The ratio of the volume of solution in the test cell to the specimen area shall be at least 100 ml/cm² specimen area.

5.2 Potentiostat shall be capable of controlling the electrode potential to within ± 1 mV of a preset value.

5.3 Electrode potential-measuring instrument with a high input impedance of the order of 10^{11} Ω to 10^{14} Ω , to minimize current drawn from the system during measurement. The sensitivity and accuracy of the instrument shall be sufficient to detect a change of 1,0 mV.

5.4 Current-measuring instruments capable of measuring a current to within 2 % of the actual value. The current in the circuit is evaluated from the potential drop measured across a known resistor.

NOTE In many potentiostats, this measurement is made internally, but measurements can also be made externally by locating a resistor in the current line from the auxiliary electrode to the auxiliary connection on the potentiostat.

5.5 Temperature controller capable of increasing the temperature of the surface of the specimen from 2 °C to 100 °C at a controlled rate. This is achieved by heating or cooling the solution. Above 10 °C, the average rate of temperature change of the specimen shall be controlled to within ± 30 % of the desired value, where the average is calculated over a temperature range of 10 °C.

NOTE At temperatures above 85 °C, avoid evaporation by suitable equipment.

5.6 Temperature measurement instrumentation capable of measuring the temperature of the test solution with an accuracy of ± 1 °C.

5.7 Crevice formers or specimen holders of different types can be used in this International Standard.

NOTE [Annex B](#) describes examples of different crevice formers and relevant aspects concerning these. Other different types of crevice formers can be used if relevant parties agree.

5.8 Test solution, commonly containing of chlorides.

5.9 Auxiliary electrode of high-purity platinum or other materials inert to the test solution. The auxiliary electrode may be constructed in the form of thin foil, a sheet, a rod, or in the form of a gauze. It can also be supported on a glass frame. The area of the auxiliary electrode shall be at least the area of the specimen.

5.10 Reference electrode shall be maintained at ambient temperature external to the test cell and connected to the test cell via a Luggin capillary probe.

NOTE The silver/silver chloride electrode is preferred but there are other alternatives. The potentials of these Ag/AgCl electrodes at 25 °C relative to the standard hydrogen electrode at 25 °C are given in [Annex D](#).

6 Procedure

6.1 Preparation of reference electrodes

6.1.1 The difference in potential between the reference electrode and two validation electrodes shall be measured. These electrodes shall be traceable to the standard hydrogen electrode and used and maintained solely for the purpose of validation. If the potential difference is greater than 3 mV, the test electrode shall be rejected.

6.1.2 The validation electrodes shall be stored in optimum conditions and regularly compared. Replacement shall be undertaken if the potential difference between these varies by more than 1 mV.

6.2 Preparation of specimen

6.2.1 The minimum specimen area of 1 cm² shall be used but the dimensions shall be compatible with the crevice former or the crevice holder used for the test. Examples of specimens are given in [Annex A](#).

6.2.2 The surface finish shall be as reproducible as possible. Any test surface may be tested but it is recommendable that all surfaces exposed to the test solution, including cut edges and drilled holes, shall be abraded down to a surface finish of at least P 80grit paper. Care shall be taken to avoid overheating the surface.

NOTE Ultrasonic cleaning can be used after the preparation.

6.2.3 The specimens shall be left in air, at a temperature higher than the dew point temperature for at least 24 h before testing, to be able to form a stable oxide.

The time elapsed between grinding and immersion can have an influence on the subsequent crevice corrosion behaviour. The elapsed time selected will depend on the purpose of the test, but should be standardized for a particular set of tests. Little variation in surface film thickness occurs after 24 h, and hence a minimum elapsed time of 24 h is often useful.

6.2.4 The specimen shall be cleaned immediately prior to immersion in the solution by degreasing, rinsing in high-purity water with a conductivity less than 1 $\mu\text{S cm}^{-1}$ (according to ISO 3696, grade 2),

followed by ethanol or a similar solvent, and air drying. After degreasing, care shall be taken not to contaminate the test surface of the specimen.

6.3 Preparation of solution

6.3.1 In principle, the test solution is prepared using reagent grade chemicals and high purity water. Other type of solution may be used if relevant parties agree.

6.3.2 Guidelines on the selection of the solution for stainless steels are given in ISO 17864.

6.4 Setting up the test

6.4.1 All parts shall be mounted together, for example, according to [Annex B](#). The exposed surface area of the specimen shall be measured and the area under the crevice former shall be estimated.

6.4.2 For materials with a CCT value expected to be less than 40 °C the temperature ramp shall start at (2 ± 1) °C.

NOTE 1 The solution can be cooled by placing a beaker of the solution in an ice bath.

NOTE 2 Some CCT values can be found in References [\[1\]](#), [\[2\]](#), [\[3\]](#) and [\[4\]](#).

6.4.3 For materials with a CCT value >40 °C, the temperature ramp shall start <30 °C.

6.4.4 The specimen, counter electrode and salt bridge shall be placed in the test vessel. The test vessel shall then be filled with the solution. It is important to ensure that the salt bridge is filled with the test solution and is free of air bubbles, particularly in the restricted space at the tip.

NOTE A wick, or equivalent device, can be placed in the salt bridge to ensure electric contact, even when small gas bubbles are formed during the test.

6.4.5 The solution shall be stirred continuously throughout the test.

NOTE This can be achieved using a mechanical stirring device, using a peristaltic pump or by bubbling inert gas through the solution at a controlled rate.

6.4.6 The tip of the reference electrode shall be positioned so that it is at a distance from the specimen of about, but not closer than, twice the diameter of the tip.

6.4.7 The electrodes shall be connected to the potentiostat and data-recording device, and the connections for temperature measurement and control shall be made. The potential shall not be applied to the specimen until the temperature of the solution has been stable to within ± 1 °C of the desired initial test temperature for a minimum of 10 min.

6.4.8 The open-circuit potential of the test specimen shall be recorded and the desired anodic potential shall then be applied to the specimen.

NOTE 1 An anodic potential of +944 mV v.s. SHE is a common used value for high alloyed stainless steels.

NOTE 2 The potential can be set to a different value depending on the purpose, but the same potential for a particular set of test shall be applied and values obtained with different applied potentials cannot be compared. Guidelines for selecting the potential and temperature are given in ISO 17864.

6.4.9 When the potential has been applied for 60 s or longer, the temperature of the specimens shall be increased at a controlled rate.

NOTE 1 The rate of the temperature increase will affect the result and if different materials shall be compared the identical conditions (crevice former, torque, surface preparation, temperature increase and the applied potential) shall be used.

NOTE 2 1 °C/min is a common used temperature increase for high alloyed stainless steels.

6.4.10 The current and solution temperature shall be monitored throughout the test. The minimum sampling rate shall be 10 readings per minute.

6.5 Ending test

6.5.1 The test shall be terminated when the CCT has been determined and when it has been made sure that the current originates from stable propagating crevice corrosion, and not a short-lived current peak.

6.5.2 The specimen shall be removed from the solution and rinsed in water, cleaned with ethanol, rinsed with high-purity water with a conductivity less than 1 µS/cm, (ISO 3696, grade 2), cleaned with ethanol or a similar solvent and dried in air.

6.5.3 The specimen shall be inspected using an optical microscope at 20× magnification to determine whether crevice corrosion have occurred. If corrosion attacks are found on other places than under the crevice former the value shall be excluded.

7 Assessment of results

7.1 The CCT shall not be compared with values obtained using a different procedure i.e. with another surface finish, crevice former, torque, temperature ramp rate or at a different critical current density.

NOTE For materials that exhibit a very high passive-current density a higher critical current density can be chosen.

7.2 Crevice corrosion phenomenon is generally of a random nature, thus, the number of experiments required for a given condition are preferable more than seven considering data scattering, and at least two experiments for an experimental condition are required.

8 Test report

The test report shall include the following information:

- a) a reference to this International Standard, i.e. ISO 18089:2015;
- b) the test solution and its volume;
- c) a full description of the test material from which the specimens were taken, including composition and structural condition, type of product and section thickness;
- d) the orientation and size of test specimens;
- e) the surface finish of specimens, including the storage time between carrying out the final surface finish and testing;
- f) the test area of crevice assembly;
- g) the type of crevice assembly and the torque;
- h) the electrode potential;
- i) the temperature ramp rate;

j) a description of the specimen surface after testing;

NOTE 1 If no crevice corrosion were observed, this shall be noted explicitly.

k) the formula for conversion of test-solution temperature to specimen temperature if a specimen holder is used;

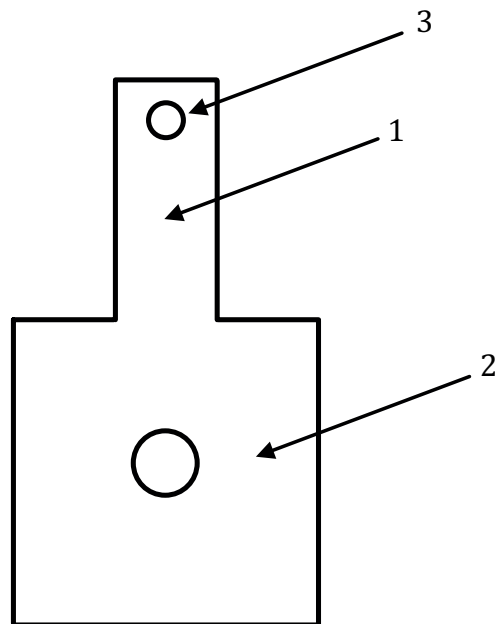
l) the electrochemical critical crevice corrosion temperature (CCT).

NOTE 2 CCTs below 10 °C shall only be reported as <10 °C.

Annex A (informative)

Examples of how the connections of the electrode can be made

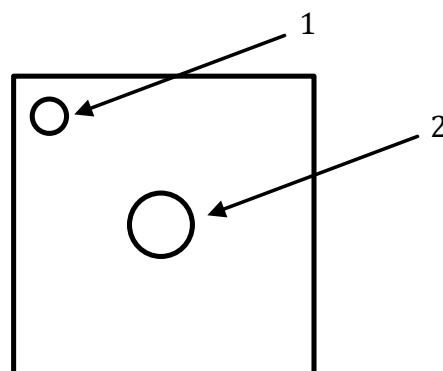
Different experimental setups and connections can be used. In this annex, some examples of specimens and how to connect them electrochemically are given.



Key

- 1 line of solution
- 2 area where the crevice former shall be placed
- 3 electric connection

Figure A.1 — A schematic figure of a specimen



Key

- 1 electric connection
- 2 area where the crevice former shall be placed

Figure A.2 — A schematic figure of a specimen

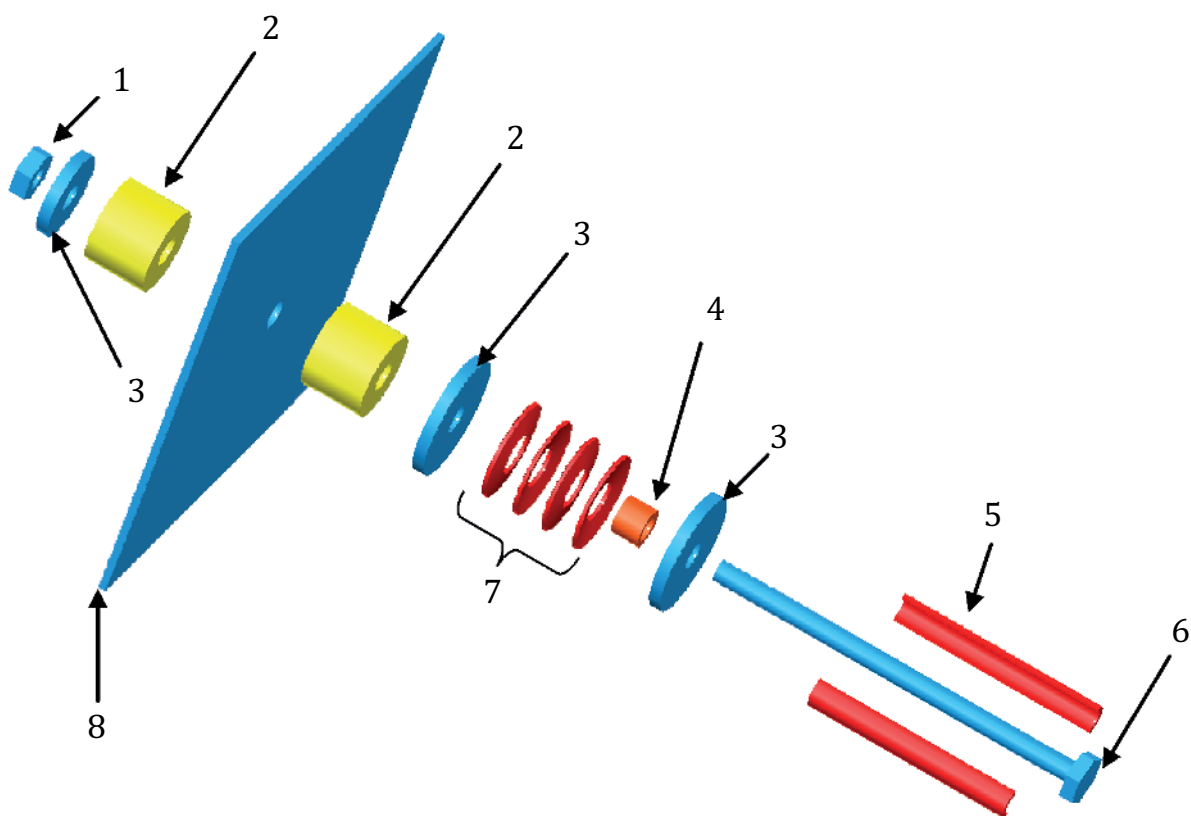
Annex B (normative)

Schematic representation of different crevice formers used for CCT-measurements

B.1 General

Different crevice formers can be used to create a crevice for CCT measurements. The alternatives are specified in this annex.

B.2 Crevice former based on ISO 18070



Key

- | | | | |
|---|-------------------|---|---|
| 1 | nut | 5 | insulation house (split only for imaging purpose) |
| 2 | crevice former | 6 | bolt |
| 3 | washer | 7 | disc springs |
| 4 | centralizing ring | 8 | test specimen |

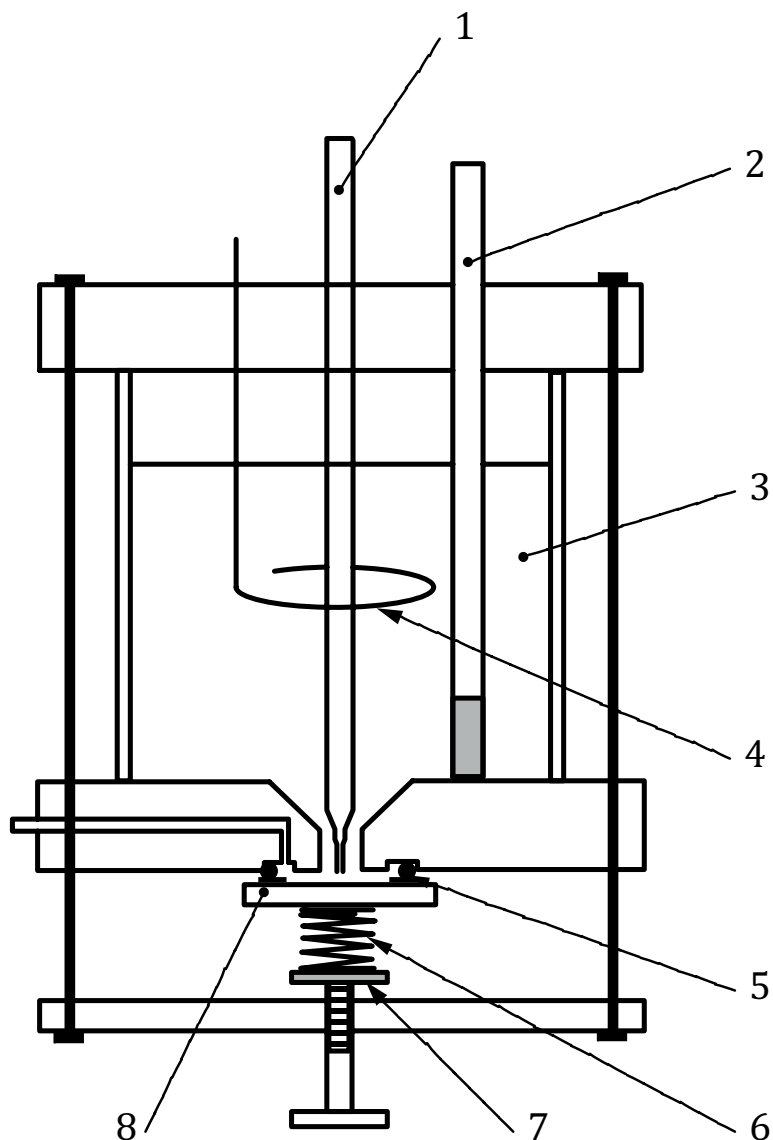
Figure B.1 — A schematic picture of the crevice former^[5]

A centre hole in 7 mm in diameter shall be drilled in the middle. One hole at one end of the tube shall be made for electrical connection. All electrical connections should be made using platinum or titanium wires and connecting the specimens to a contact point outside the test vessels. Other materials are not to be used for electrical connections to in order to avoid any possibilities of galvanic actions. Coating of contact points is not allowed.

The crevice former shall be assembled according to ISO 18070. All the materials and dimensions are specified there.

B.3 Specimen holder for flat specimens based on ISO 17864

The advantage of a specimen holder is that minimal sample preparation is needed. The specimen holder can be flexible as far as the principle of the figure is reserved. Other alternatives are presented in ISO 17864 but the cell shall not be flushed by purified water since crevice corrosion is measured.



Key

- | | | | |
|---|-----------------------------|---|---|
| 1 | luggin capillary | 5 | rubber washer (crevice former) and O-ring |
| 2 | inert gas with gas disposer | 6 | disc springs |
| 3 | test solution | 7 | washer |
| 4 | counter electrode | 8 | specimen |

Figure B.2 — A schematic figure of the specimen holder

The specimen holder shall be designed to allow application of a constant torque of 0,6 Nm to a screw against the reverse of the specimen. The face of the specimen presses against a circular washer of EPDM (ethylene propylene diene monomer), hardness 70, rubber is exposed to the test solution, see [Figure B.2](#).

If a specimen holder is used the temperature of the specimen relative to the temperature of the solution shall be calibrated. Guidelines for calibrating the temperature of the surface of the specimen relative to the temperature of the solution are given in [Annex C](#).

Annex C (normative)

Calibration of specimen temperature vs. solution temperature if a specimen holder is used

C.1 If a specimen holder is used, the temperature of the specimen relative to the temperature of the solution shall be calibrated. The magnitude of the temperature gradient will depend on the geometry and dimensions of the specimen. The calibration shall be performed using the procedure for the CCT test, given in [Clause 6](#) except that no control or measurement of the electrode potential of the specimen is required.

NOTE When using this calibration of specimen temperature vs. solution temperature the seasonal changing environmental conditions need to be considered.

C.2 The specimen size and geometry, solution volume, stirring rate and temperature ramp rate shall be the same for the calibration test as for the CCT test.

C.3 The specimen temperature shall be measured by installing a suitable temperature measuring device inside the specimen, as close as possible (<1 mm) to the surface of the specimen in contact with the solution. The temperature measuring device shall be located at the centre of the specimen.

C.4 Calibration of the specimen temperature relative to the solution temperature shall be performed by taking measurements of the solution temperature and the corresponding specimen temperature, as the temperature of the bulk solution is increased at the temperature ramp rate. Measurements shall be taken at intervals of not more than 10 °C in the temperature range of interest.

C.5 The specimen temperature calibration formula shall be calculated based on a linear order regression analysis.

C.6 The accuracy of the measurement of the temperature of the specimen based on the calibration data shall be $\pm 1,0$ °C.

Annex D
(informative)

Potential difference of selected reference electrodes at 25 °C with respect to the standard hydrogen electrode (SHE)

Reference electrode	Potential difference with respect to SHE, V
Silver/silver chloride electrode in saturated KCl	0,196
Silver/silver chloride electrode in 1 M KCl	0,222
Silver/silver chloride electrode in 0,1 M KCl	0,288

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