# Application Bulletin

Of interest to: General analytical laboratories E L 1, 2, 4, 6, 7, 8, 9, 16

## Validation of Metrohm conductometers using Standard Operating Procedures


### *Summary*

Among other things, **GLP** (**G**ood **L**aboratory **P**ractice) requires that the accuracy and precision of analytical instruments are checked at regular intervals using **Standard Operating Procedures (SOPs)**.

The procedures described below are meant as a guideline for setting up a Standard Operating Procedure to check your conductometer and the conductivity measuring cell. The limits specified should be regarded as examples. Depending on the requirements placed on the accuracy of the measuring system these limits may have to be redefined in the Standard Operating Procedure.


### *Application range*

These test specifications can be used with the following Metrohm instruments:

587 Conductometer
644 Conductometer and
712 Conductometer.

Of course, older conductometers can also be checked in a similar way.

### *Required instruments and reagents*

- 2.767.0010 Calibrated Reference
- KCl conductivity standard, e.g. *c*(KCl) = 0.1000 mol/L, Metrohm no. 6.2301.060 (the electrical conductivity of this standard solution is 11.67 mS/cm at 20 °C and 12.88 mS/cm at 25 °C)
- Possibly other conductivity standards with lower electrical conductivities; these can be obtained, e.g., from Fluka, Merck, NIST (USA), etc.
- Ethanol, p.a.
- Demineralized or distilled water

## *Procedure*

### *1. Checking the conductometer with the 2.767.0010 Calibrated Reference*

Electrode cables can also be a source of trouble. It is therefore recommended to include the original sensor cables in the testing process (only possible with plug-in cables).

If, on the other hand, only the conductometer is to be checked, then you need the following tested standard cables, which form part of the accessories of the 2.767.0010 Calibrated Reference:

- 6.2150.020 Electrode cable for Metrohm plug-in head G, with 2 x plug B (corresponds to 6.2104.080 electrode cable)
- 2 x 6.2150.000 Cable with plug B on both ends (corresponds to 6.2106.020 cable)

If possible, the cell constant on the conductometer to be checked is set to 1.000 $cm^{-1}$, the measuring and reference temperature to 20 °C and the temperature coefficient to 1.00 or 0.00 %/°C.

Parameters using the 712 Conductometer as an example:

>cond/parameters

| | |
|---|---|
| cell constant | 1.000 /cm |
| meas.temp. | 20.0 °C |
| ref.temp. | 20.0 °C |
| TC selection: | const. |
| TC const. | 0.00 %/°C |
| frequency: | auto |
| meas.type: | standard |

The following equation applies:

$$\kappa = G * c$$

where

$\kappa$ = electrical conductivity [µS $cm^{-1}$]
G = conductance [µS]
c = cell constant [$cm^{-1}$]

The measurements are always carried out with the lid closed (over the solar cell of the 2.767.0010). For the first two tests the banana plugs of the electrode cable are plugged into the input sockets of the conductometer and the electrode plug into socket 5 or into socket 6 of the Calibrated Reference. Afterwards, the two input sockets of the conductometer are connected with the sockets 1 and 2 or 2 and 3 of the 2.767.0010 using two cables with plug B on both ends. The values given below are examples only.

**587 Conductometer**

| | Value read off | Theoretical value | Difference | Error tolerance 587* |
|---|---|---|---|---|
| Socket 5 | 69.5 | 70.01 | –0.5 | ±1% (±0.7 µS/cm) |
| Socket 6 | 2.18 | 2.17 | 0.01 | ±2% (±0.04 µS/cm) |
| Sockets 1 + 2 | – | 10008 | – | – |
| Sockets 2 + 3 | 990 | 999 | –9 | ±1% (±10 µS/cm) |

***Assessment:*** The checked instrument is OK.

**644 Conductometer**

| | Value read off | Theoretical value | Difference | Error tolerance 644* |
|---|---|---|---|---|
| Socket 5 | 69.9 | 70.01 | –0.1 | ±2% (±1.4 µS/cm) |
| Socket 6 | 2.18 | 2.17 | 0.01 | ±2% (±0.04 µS/cm) |
| Sockets 1 + 2 | 10000 | 10008 | –8 | ±2% (±200 µS/cm) |
| Sockets 2 + 3 | 1010 | 999 | 11 | ±2% (±20 µS/cm) |

***Assessment:*** The checked instrument is OK.

**712 Conductometer**

| | Value read off | Theoretical value | Difference | Error tolerance 712* |
|---|---|---|---|---|
| Socket 5 | 70.07 | 70.01 | 0.06 | ±0.5% (±0.35 µS/cm) |
| Socket 6 | 2.168 | 2.17 | –0.002 | ±0.5% (±0.011 µS/cm) |
| Sockets 1 + 2 | 10010 | 10008 | 2 | ±0.5% (±50 µS/cm) |
| Sockets 2 + 3 | 999.2 | 999 | 0.2 | ±0.5% (±5.0 µS/cm) |

***Assessment:*** The checked instrument is OK.

In combination with a Pt 100 or Pt 1000 resistance thermometer the 712 Conductometer can also be used for temperature measurements. To check the temperature reading the two red input sockets of the 712 are connected with the sockets 1 and 2 (Pt 100) or 2 and 3 (Pt 1000) of the 2.767.0010 Calibrated Reference using two cables with plug B on both ends.

| | Value read off / °C | Theoretical value / °C | ΔT / °C | Error tolerance 712* / °C |
|---|---|---|---|---|
| Sockets 1 + 2 | –0.2 | –0.20 | 0 | ±0.1 |
| Sockets 2 + 3 | 0.1 | 0.17 | –0.07 | ±0.1 |

***Assessment:*** The checked instrument is OK.

*) According to the technical specifications given in the Instructions for use of the 587, 644 or 712 Conductometers.

If the measured values are clearly outside the given tolerance ranges then a new adjustment of the conductometer is necessary. Please contact the Metrohm service department.

## *2. Checking the conductivity measuring cell with a conductivity standard*

Conductivity measuring cells that have been stored dry must be rinsed with ethanol and then placed in distilled water for at least one hour. Measuring cells that have already been used are rinsed well with distilled water and then any adhering water drops should be removed as far as possible (shake measuring cell and touch the outside lightly with a soft paper tissue).

The measuring vessel is first rinsed well with distilled water and then with the conductivity standard. It is then filled with the standard solution and thermostatted at 20 °C.

The measuring cell is first immersed several times in the conductivity standard and then positioned so that the upper side openings are completely immersed. Any air bubbles inside the measuring cell can be removed by swirling and gentle tapping.

Switch on the conductometer and, if possible, enter the corresponding values for the cell constant (this is printed on the conductivity measuring cell) as well as for the measuring and reference temperature.

Parameters using the 712 Conductometer as an example:

>cond/parameters

| | |
|---|---|
| cell constant | value printed on the cell (e.g. 0.78 /cm) |
| meas.temp. | 20.0 °C |
| ref.temp. | 20.0 °C |
| TC selection: | const. |
| TC const. | 0.00 %/°C |
| frequency: | auto |
| meas.type: | standard |

Wait until the temperature is constant and then read off the measured value. If the KCl conductivity standard from Metrohm (6.2301.060) is used the electrical conductivity must be 11.67 ± 0.15 mS/cm.

With the 644 Conductometer the conductance value read off must be multiplied by the cell constant as the latter cannot be set on this instrument. In this way the electrical conductivity of the standard solution is obtained.

If the measured conductivity lies outside the expected range then we recommend that the above measurement is repeated with a different concentration of conductivity standard [e.g. *c*(KCl) = 0.01 mol/L]. At 20 °C an electrical conductivity of 1.28 mS/cm should now be obtained. If the measured value still lies outside the tolerance range then the new cell constant can be calculated from the measured conductance (set the cell constant for this measurement to 1.000 $cm^{-1}$) as follows:

new cell constant = theoretical conductivity / measured conductance

Example for *c*(KCl) = 0.1 mol/L at 20 °C:

new cell constant = 11.67 mS $cm^{-1}$ / 14.55 mS = **0.80 $cm^{-1}$**

If the results are still unsatisfactory when the new cell constant is used then the conductivity measuring cell should be checked and replaced if necessary.

Possible causes of a cell constant alteration:

- If the conductivity measuring cell is stored in distilled water then algae, fungi or bacteria may grow on the Pt surfaces of the electrode.
- Measurements in suspensions and emulsions can lead to deposits on the Pt surfaces of the electrode.
- Storage in drinking water can lead to lime deposits.
- The platinum black may be partially removed (mechanical effects).

All the possibilities mentioned above lead to a reduction in the active surface of the measuring cell and therefore inevitably to an alteration of the cell constant.

## *Literature*

Further information on this topic:

- Application Bulletin No. 102 «Conductometry»
- Special leaflets for conductivity measuring cells (in the sensor boxes)