# Application Bulletin 278/1 e

# Validation of the Metrohm 743 Rancimat using Standard Operating Procedures

Of interest for:
General analysis, organic chemistry, pharmaceuticals, petrochemistry, food industry, biology, cosmetics, paints
B 1, 3, 4, 5, 7, 8, 12, 14


### *Summary*

**GLP** (**G**ood **L**aboratory **P**ractice) requires periodic checking of the reproducibility and accuracy of analytical instruments by using **S**tandard **O**perating **P**rocedures (**SOPs**).

This Application Bulletin is intended to provide you with a guideline for the validation of your Rancimat. A standard operating procedure is proposed which allows all the Rancimat functions which are relevant for the measurement to be checked. The limits given are to be taken as being examples of what can be achieved under normal laboratory conditions. Depending on the demands placed upon the accuracy of the measuring system, it may be necessary to redefine these limits in the standard operating procedure. If very careful work is carried out under appropriate conditions then it may be possible to achieve lower limits.

The operating software for the 743 Rancimat contains prepared **GLP tests** for the temperature, conductivity and gas flow measurements. The operator can determine whether and which tests are to be carried out, in addition the interval between the tests and the required accuracy can also be defined. If the GLP function is activated then a comment will be made on each result report as to whether the GLP test requirements have been fulfilled. In order to be able to carry out the most important tests Metrohm offers the **6.5616.000 GLP Test Set** as an option.

Further information about the topics of QA, GLP and validation can also be found in the brochure **"Quality management with Metrohm"** which is available from your local Metrohm agency.


### *Field of application*

These instructions are applicable to the following Metrohm instrument:

- 743 Rancimat

### *Checking period*

It is a good idea to repeat the check on the Rancimat at yearly intervals. Apart from this, an extra validation should be carried out after one or more Rancimat components have been exchanged.

### *Internal instrument checking routines*

Metrohm instruments have their own internal start-up checking routines. During the switch-on test the display elements and the contents of the program memory are checked by carrying out check sum tests. The functioning of the data memory sector is tested by a write/read test. With the 743 Rancimat a check is made as to whether a PC is connected and that the control software has been started. If no error message is produced then it can be assumed that the instrument is working perfectly.

If Rancimats are checked within the context of regular service work being carried out then a special validation of the instrument electronics is no longer necessary.

### *Maintenance / Service*

An essential requirement for ensuring the GLP-conform operation of each individual instrument used in the laboratory is that instrument care and cleansing are carried out with great care and, in particular, that the instruments are handled properly. The 'Instructions for Use' provided for the particular instrument must be available to all laboratory technicians.

Checking the electronic and mechanical functional groups of Metrohm instruments can and should be carried out by specialists from the manufacturing company within the framework of regular service work. Many Metrohm agencies offer favorably-priced service contracts for your instruments. For this reason if faults or errors should occur the 743 Rancimat is equipped with a built-in diagnostics program which allows the service technician to check the functioning of particular assemblies in order to localize the error.

We also recommend that all the instruments used in the validation process are subjected to a regular annual service.

### *Methods*

The 6.5616.000 GLP Test Set is necessary in order to validate the Rancimat. It contains a certified Pt-100 temperature sensor, an insert for the heating block and a test resistor (10 kΩ). The Rancimat has a measuring input for making measurements with the temperature sensor.

In order to validate the Rancimat Metrohm recommends that the following steps are carried out:

1. Preparation: activate the GLP function.
2. Validate the temperature of the Rancimat heating blocks with the help of the GLP Set.

3.1. Validate the conductivity inputs with the help of the 10 kΩ test resistor.

3.2. Validate the conductivity cells with the help of a standard conductivity solution.

*As the absolute correctness of the measured conductivity plays no part in the determination of the induction time, we recommend that the cell constants of the conductivity cells according to 3.2. are not calibrated and validated routinely. This is only important if the stability time is to be determined in addition to the induction time.*

*If the conductivity cells are validated regularly according to 3.2. then it is not necessary to check the measuring inputs with the test resistor according to item 3.1 as well.*

4. Validate the temperature correction with the help of a measurement in inert oil.
5. Measure the reproducibility in oil.

Within normal limits the influence of the gas flow on the induction time can be neglected so that it is not necessary to validate the gas flow.

### *Required accessories*

- 6.5616.000 GLP Test Set for 743 Rancimat
- Silicone oil, e.g. Fluka 85409
- Edible oil, e.g. sunflower oil
- 6.2301.060 Standard conductivity solution c(KCl) = 0.1 mol/L

# Procedure

### *1. Activating the GLP-function*

1. In the window ***743 Rancimat Control*** select the instrument 1...4 for which GLP monitoring is to be activated.
2. Click on ***743 Rancimat Control / Tools / GLP-Test / Properties***.
3. In the GLP properties window click on the register card ***Temperature***. Activate the field ***Enable GLP monitoring***. Under ***GLP alert interval*** enter the number of days until the next GLP test is due and enter the tolerance value for the GLP test under ***Tolerance***. Metrohm recommends a maintenance interval of 365 days and a tolerance of ± 0.5 °C.
4. In the GLP properties window click on the register card ***Conductivity***. Activate the field ***Enable GLP monitoring***. Under ***GLP alert interval*** enter the number of days until the next GLP test is due and enter the tolerance value for the GLP test under ***Tolerance***. Metrohm recommends a maintenance interval of 365 days and a tolerance of ± 5 µS/cm.
5. Click on ***<OK>*** to close the ***GLP-Properties*** window.

### *2. Validation of the temperature*

This test requires the use of the 6.1111.010 Calibrated Temperature Sensor and the 6.1253.000 GLP Insert; these belong to the 6.5616.000 GLP Test Set. The temperature sensor must be connected to the "Pt100" connection on the rear panel of the 743 Rancimat.

Please note that the temperature of the GLP Test Set used must be stable before the GLP test is started. If the GLP Test Set is inserted into a cold heating block then this condition is fulfilled after the normal warming-up period of the heating block and the GLP Test Set. If, however, the GLP Test Set is inserted into a heating block which is already warm then it is necessary to wait at least 30 min before the start of the GLP test.

For the validation of the temperature it is sufficient to check the instrument at a single temperature.

#### *Preparation*

1. Insert the temperature sensor into an opening of the PTFE cylinder from above.
2. Place the aluminum cylinder in a vertical position and insert the temperature sensor from above into the aluminum cylinder until it reaches the stop.
3. Attach the temperature sensor to the aluminum cylinder by slightly tightening the hexagon screw with the help of the Allen key.
4. Connect the temperature sensor to the "Pt100" connection on the rear panel of the 743 Rancimat.

***Procedure***

1. In the window ***743 Rancimat Control*** select the instrument 1...4 for which the GLP temperature test is to be carried out.
2. In the window ***743 Rancimat Control*** select the determination method whose temperature is to be used in the GLP test and alter the temperature if necessary.
3. Click on ***743 Rancimat Control / Tools / GLP-Test / Temperature*** and then click on ***<Next>*** in the GLP Test window.
4. Enter the calibration data of the temperature sensor used (entered data are retained) and click on ***<Next>***. A certified temperature sensor has three calibration points. Each point consists of a temperature-resistance pair.
5. Under ***TEST-PARAMETER*** for ***Block A*** and ***Block B*** select the channels in which the temperature test is to be carried out. At least one channel must be selected per block. Enter the required ***Test duration per channel*** and click on ***<Next>***.

   Metrohm recommends checking one channel per block and entering a delay period of 10 min.
6. The selected channels are checked in succession. The entered test duration applies to each channel.
7. Insert the temperature sensor with PTFE and aluminum cylinder in the first selected channel and click on ***<Next>*** to start the measurement.
8. If the temperature defined in the method has not yet been reached then heating is started automatically. In the ***Measure status*** window the message ***Waiting until temperature is reached...*** appears.
9. When the temperature defined in the method has been reached the message ***Measuring temperature. Please wait...*** appears.
10. When the measurement has been carried out you are requested to insert the external temperature sensor and GLP insert into the next selected channel and to start the measurement. Care must be taken that changing the temperature sensor from one channel to the next is carried out as quickly as possible in order to prevent cooling down.
11. Carry out the GLP temperature measurement for all channels in succession.
12. When the last temperature measurement has been carried out the test results for the measured channels are shown.
13. Check the results and click on ***<Finish>*** in order to store the results of the GLP test.

***Assessing the results:***

The variation between the measured temperature and the set temperature displayed should not exceed ± 0.5 °C, otherwise the measurements should be repeated and the test duration per channel should be extended if necessary.

## *3.1. Validation of the conductivity inputs with the test resistor*

This test is carried out with the 6.2109.030 Test Resistor which belongs to the 6.5616.000 GLP Test Set available from Metrohm as an option. The test resistor must be connected to the electrode connections of the 743 Rancimat.

***Procedure***

1. In the window ***743 Rancimat Control*** select the instrument 1...4 for which the GLP conductivity test is to be carried out.
2. Click on ***743 Rancimat Control / Tools / GLP-Test / Conductivity*** and then click on ***<Next>*** in the GLP Test window.
3. Switch on the test resistor option and enter the resistance of 6.2109.030 Test Resistor: 10000 Ω. Then click on ***<Next>***.
4. Attach the 6.2109.030 Test Resistor to the electrode connections of each selected channel in succession. Click on ***<Next >*** each time.
5. When the last conductivity measurement has been carried out the test results for the measured channels are shown.
6. Check the results and click on ***<Finish>*** in order to store the results of the GLP test.

***Assessing the results:***

The variation between the measured conductivity and the set conductivity displayed should not exceed ± 5 µS/cm.

## *3.2. Validation of the conductivity measurement with a standard conductivity solution*

This test is carried out with a standard solution of known conductivity. Before the GLP conductivity test with a standard solution is carried out for the first time the cell constant of the conductivity cell must be determined. The determination of the cell constant is described in Section 4.4.1 of the 'Instructions for Use' of the 743 Rancimat..

If a calibrated conductivity cell is used then care must be taken that it is always used in the same channel.

As the absolute correctness of the measured conductivity plays no part in the determination of the induction time it is not necessary to routinely validate the cell constant of the conductivity cell. This is only important if the stability time is to be determined in addition to the induction time.

***Conductivity of the standard solution***

c(KCl) = 1 mmol/L can be used as the standard solution; this is prepared from the 6.2301.060

Conductivity Standard (KCl 0.1 mol/L) available as an option. The conductivity standard is diluted 1:100 with distilled water.

This solution has a conductivity of:

| Temperature | Conductivity |
|---|---|
| 18 °C | 127 µS/cm |
| 19 °C | 130 µS/cm |
| 20 °C | 133 µS/cm |
| 21 °C | 136 µS/cm |
| 22 °C | 138 µS/cm |
| 23 °C | 141 µS/cm |
| 24 °C | 144 µS/cm |
| 25 °C | 147 µS/cm |

*Instead of the standard solution c(KCl) = 0.1 mol/L, ready-to-use dilute standard solutions with conductivities in the range 100 - 140 µS/cm can also be used. These solutions are available commercially and are supplied with a quality certificate.*

***Preparation***

1. Place a measuring vessel with cover and filled with 60 mL standard solution in each measuring position.

***Procedure***

1. In the window ***743 Rancimat Control*** select the instrument 1...4 for which the GLP conductivity test is to be carried out.
2. Click on ***743 Rancimat Control / Tools / GLP-Test / Conductivity*** and then click on ***<Next>*** in the GLP Test window.
3. Switch on the Standard solution option and enter the value for the conductivity of the standard solution in the appropriate field. Click on ***<Next>***.
4. Click on ***<Next>*** in the GLP Test window. The conductivity is now measured in all channels.
5. When the last conductivity measurement has been carried out the test results for the measured channels are shown.
6. Check the results and click on ***<Finish>*** in order to store the results of the GLP test.

***Assessing the results:***

The variation between the measured conductivity and the set conductivity displayed should not exceed ± 10 µS/cm, otherwise the measurement must be repeated with a new standard conductivity solution. If the variation is still too large then the conductivity cell must be recalibrated and the GLP test repeated.

## *4. Validation of the temperature correction*

The temperature correction Delta T describes the variation of the actual temperature of the sample from the temperature of the heating block and is one of the parameters of the method. The variation between the temperature of the heating block and that of the sample is mainly caused by the flow of air which cools the sample. This means that it must be determined separately for each temperature, each gas flow and each heating block. It can be determined automatically with the external 6.1111.010 Temperature Sensor (included in the 6.5616.000 GLP Test Set available from Metrohm as an option).

If work is carried out properly then the variation remains within very narrow limits so that instead of individually determined values the values given in the table below can be used as an approximation. These values apply to a gas flow of 20 L/h:

| Set temperature | Delta T |
|---|---|
| 80 °C | +1.1 °C |
| 90 °C | +1.3 °C |
| 100 °C | +1.4 °C |
| 110 °C | +1.5 °C |
| 120 °C | +1.6 °C |
| 130 °C | +1.7 °C |
| 140 °C | +1.8 °C |
| 150 °C | +1.9 °C |
| 160 °C | +2.0 °C |

If the temperature correction is to be determined experimentally then Metrohm recommends that a separate method is drawn up for each heating block and each temperature and that the temperature correction is determined separately for each of these methods. This should be carried out before the validation of the temperature correction.

If measurements are to be made at different temperatures then it is sufficient to check the validation at a single temperature.

***Preparation***

1. Fill a reaction vessel with 5 g silicone oil.
2. Insert the air tube and temperature sensor in the reaction vessel cover and fix in place with distance pieces.
3. Place reaction vessel cover with temperature sensor on the reaction vessel.
4. Push temperature sensor right down to the bottom (the sensor must contact the vessel base).
5. Place reaction vessel in Position 2 or 3 and connect to the gas inlet tube.

***Procedure***

1. In the window ***743 Rancimat Control*** select the required method for the heating block for which you want to validate the temperature correction Delta T.
2. Open the window containing the method parameters. Note the previous value for Delta T

in the validation protocol and set the value for Delta T to 0. Then click on ***<Save>***.

3. Start the gas flow and the heating manually.
4. Click on ***743 Rancimat Control*** / ***Tools*** / ***Determine delta T*** / ***Block A*** or ***743 Rancimat Control*** / ***Tools*** / ***Determine delta T*** / ***Block B*** to start the determination of the temperature correction.
5. The first window to open contains data concerning the external temperature sensor; the calibration data of the temperature sensor used can be entered here (the last data to be entered are displayed here).
6. Click on ***OK***. The window ***Determination of Delta T*** appears.
7. If the temperature defined in the method has not yet been reached then the message ***Waiting until temperature is reached...*** appears in the window.
8. When the temperature defined in the method has been reached the message ***Measuring temperature. Please wait...*** appears in the window.
9. As soon as the two measured temperatures are stable the message ***Measured temperature is now stable!*** appears. The temperature correction Delta T which has been determined can now be accepted in the method with ***<Accept>***. The message ***Delta T is now set in the corresponding method*** now appears. The value determined for Delta T can also be taken over manually in other methods which use the same temperature.

***Assessment of the results:***

The variation between the measured temperature correction and the previously used temperature correction should not exceed ± 0.3 °C.

## *5. Measuring the reproducibility in oil*

In order to check the functioning of the complete measuring setup we recommend that a measurement is carried out on edible oil. As the absolute induction time is unknown and is also not stable over longer periods of time only the scatter of the measurements is to be checked. Metrohm recommends that a double determination is carried out.

Even if measurements are to be made at different temperatures it is sufficient to check the reproducibility at a single temperature.

***Preparation***

1. Fill each of two measuring vessels with 60 mL distilled water.
2. Place the measuring vessels together with the measuring vessel covers in the openings provided for them in the 743 Rancimat and connect the electrode plug of the cover to the corresponding socket of the 743 Rancimat.
3. Fill each of two reaction vessels with 3 g sunflower oil.
4. Attach an air tube to the reaction vessel covers.
5. Place the reaction vessel cover on the reaction vessel. Rotate the cover so that the air tube is as close as possible to the vessel wall.
6. Attach the white connecting tube to the reaction vessel cover connection.

***Procedure***

7. In the window ***743 Rancimat Control*** select the required method for which you want to determine the reproducibility.
8. Start the heating manually.
9. Enter the sample identifications ID1 and ID2 in the corresponding fields in the ***743 Rancimat Control*** window.
10. Wait until the required temperature has been reached in the heating block.
11. Insert the two reaction vessels in the 743 Rancimat and connect the gas inlet tube and the white connection tube to the measuring vessel.
12. Click on the ***Start*** button in the operating field of the ***743 Rancimat Control*** window.
13. When the measurement is finished the results can be seen in the ***743 Rancimat Results*** / ***Determination data*** window.
14. To print out the results click on ***743 Rancimat Results*** / ***File*** / ***Print*** / ***Determination acc. to method /Print***.

***Assessing the results:***

The absolute difference between the measured induction times of two samples should not exceed more than 10% of the mean value of the two induction

times. The induction time of the oil sample should be between 4 hours and 10 hours.

## 6. Printing GLP results

In order to print out the GLP certificate click on ***743 Rancimat Control / Tools / GLP-Test / Print GLP-Results***. The GLP certificate contains information about the GLP tests for temperature and conductivity.

The GLP certificate printout for the selected instrument contains the printing date, the serial number of the instrument and the results of the last GLP test carried out on this instrument. Each of the GLP tests for temperature, conductivity and gas flow contains the following information:

| | |
|---|---|
| Date | Date of the last GLP test carried out. |
| Tester | Person who carried out the GLP test. |
| Test result | Information about whether the test was passed or not. |

If the test results are to be printed out then GLP monitoring must be switched on.

In order to print out all the GLP results click on ***743 Rancimat Results / View / GLP*** and on ***File / Print / All Temperature GLP's*** and on ***OK*** and ***OK***. Click on ***File / Print / All Conductivity GLP's*** and on ***OK*** and ***OK***.

The detailed GLP results of all the GLP tests carried out will be printed out.

### Printing out the temperature correction value

In order to print out the value determined for the temperature correction the corresponding method must be opened in the ***743 Rancimat Control / File / Open Method*** window. Click on ***Print*** in order to print out a report of the corresponding method. This must be repeated for each validated method.

### Printing out the results of the reproducibility measurement

Mark the determinations in the ***743 Rancimat Results / Determination Data*** window and click on ***File / Print***. In the ***Printing of...*** window activate the field ***Determination acc. to method*** and click on ***Settings***.

In the ***Program settings*** window activate the ***Header, Results, Curve, Method parameters*** fields and ***Method description*** and click on <***OK***>.

Click on ***Print*** in the ***Printing of...*** window and on ***<OK>***.

## Procedure if the values obtained do not fulfil the requirements

All such values must be documented in the validation protocol and the further procedure must also be documented.

If the variations are too large then the various points given under "Recommendations for remedying faults" must be thoroughly checked and the causes of the faults must be removed. The validation must be repeated.

## Literature

- 743 Rancimat 'Instructions for Use'

### *Recommendations for remedying faults*

Information about remedying faults can also be found in the 'Instructions for Use' for the 743 Rancimat. The following lists are not exhaustive and are only examples of possible sources of error.

#### *Possible sources of error*

| *Error* | *Cause* | *Remedy* |
|---|---|---|
| **High variations between measurements in different channels** | Dirty reaction vessels | Blow out disposable vessels with nitrogen before use, remove dust from the outside of disposable vessels |
| | | Thoroughly clean reusable vessels |
| | | Check reusable vessels for damage and scratches and exchange if necessary |
| | Molecular sieve exhausted | Dry or replace molecular sieve |
| | Different amounts of samples | Ensure sample weights are correct |
| | Samples not inserted at the same time or not started immediately after insertion | Place samples in the heating block and start without delay |
| | Dirty reaction vessel openings in the heating block | Clean heating block openings |
| | Inhomogeneous samples | Homogenize samples |
| | Sample contamination during weighing out | Avoid contamination |
| **High variations between measurements in the two heating blocks** | Incorrect Delta T setting | Determine temperature correction for each heating block separately |
| | Molecular sieve exhausted | Dry or replace molecular sieve |
| | 6.1111.010 Temperature sensor not functioning properly | Replace or recalibrate temperature sensor |
| | Dirty reaction vessel openings in the heating block | Clean heating block openings |
| **High variations between measurements in different instruments** | Incorrect Delta T setting | Determine temperature correction again |
| | Molecular sieve exhausted | Dry or replace molecular sieve |
| | Incorrect weight of silicone oil for determining the temperature correction | Use the weight given in the instructions for determining Delta T |
| | Incorrect sample weight | Correct sample weight |
| | 6.1111.010 Temperature sensor not functioning properly | Replace or recalibrate temperature sensor |
| | Dirty reaction vessel openings in the heating block | Clean heating block openings |
| **Atypical curves with particular samples**  | Samples release other substances before induction time is reached | Enter evaluation delay or evaluation suppression |
| | Measuring vessel cover or tubing releases substances | Heat measuring vessel cover and tubing for 1 to 2 hours at 80°C in drying oven. |
| **Very noisy curves** | Conductivity cell not working properly | |
| | Conductivity cell contaminated | Clean conductivity cell |
| | Fatty particles in measuring vessel | Clean measuring vessel and conductivity cell, use foam barrier |

## *Appendix*

***Validation certificate***

# GLP Certificate

17.11.2000 09:06:47

**Serial number** 02136

## GLP Temperature

| | |
|---|---|
| Date | 15.06.2000 16:00:59 |
| Tester | Administrator |
| Test result | Test passed |

## GLP Conductivity

| | |
|---|---|
| Date | 15.06.2000 11:52:52 |
| Tester | Administrator |
| Test result | Test passed |

## GLP Gas flow

| | |
|---|---|
| Date | 15.06.2000 11:40:15 |
| Tester | Administrator |
| Test result | Test passed |

***Validation certificate for temperature validation of the Rancimat heating blocks using the 6.5616.000 GLP Set***

# GLP Temperature

17.11.2000 09:08:15

| | | | | | |
|---|---|---|---|---|---|
| **Date** | 15.06.2000 16:00:59 | | | **Tester** | Administrator |
| **Serial number** | 02136 | | | **Sensor designation** | 6.1111.010 Nr. 19 |
| **Temperature point 1** | 50 °C | **Resistance point 1** | 119.41 Ohm | | |
| **Temperature point 2** | 100 °C | **Resistance point 2** | 138.52 Ohm | **Test duration** | 15 min |
| **Temperature point 3** | 200 °C | **Resistance point 3** | 175.86 Ohm | **Tolerance** | ± 0.5 °C |

**Temperature Block A** 121.60 °C **Temperature Block B** 121.60 °C

| A 1 | A 2 | A 3 | A 4 | B 1 | B 2 | B 3 | B 4 |
|---|---|---|---|---|---|---|---|
| ☑ ☑ | ☑ ☑ | ☑ ☑ | ☑ ☑ | ☑ ☑ | ☑ ☑ | ☑ ☑ | ☑ ☑ |
| 121.28 °C | 121.36 °C | 121.28 °C | 121.23 °C | 121.44 °C | 121.52 °C | 121.52 °C | 121.44 °C |

**Legend** ☐ ☐ Test done / test passed

***Validation certificate for validation of the conductivity inputs using the 10kΩ test resistor***

## GLP Conductivity

17.11.2000 09:08:36

**Serial number** 02136 **Date** 15.06.2000 11:35:58 **Tester** Administrator

◉ **Test resistance** **Conductivity** 100.0 µS/cm

○ **Standard solution**

**Tolerance** ± 5.0 µS/cm

| A 1 | A 2 | A 3 | A 4 | B 1 | B 2 | B 3 | B 4 |
|---|---|---|---|---|---|---|---|
| ☑ | ☑ | ☑ | ☑ | ☑ | ☑ | ☑ | ☑ |
| 98.8 µS/cm | 98.9 µS/cm | 98.8 µS/cm | 98.8 µS/cm | 98.9 µS/cm | 98.9 µS/cm | 98.8 µS/cm | 98.8 µS/cm |

**Legend** ☐ Test passed

***Validation certificate for validation of the conductivity cell using a standard conductivity solution***

## GLP Conductivity

17.11.2000 09:08:36

**Serial number** 02136 **Date** 15.06.2000 11:52:52 **Tester** Administrator

○ **Test resistance**

◉ **Standard solution** **Conductivity of standard solution** 141.0 µS/cm

**Tolerance** ± 5.0 µS/cm

| A 1 | A 2 | A 3 | A 4 | B 1 | B 2 | B 3 | B 4 |
|---|---|---|---|---|---|---|---|
| ☑ | ☑ | ☑ | ☑ | ☑ | ☑ | ☑ | ☑ |
| 141.3 µS/cm | 141.2 µS/cm | 140.9 µS/cm | 141.0 µS/cm | 141.5 µS/cm | 141.3 µS/cm | 141.5 µS/cm | 141.1 µS/cm |

**Legend** ☐ Test passed

***Method printout for documentation of the determined temperature correction using a measurement in inert oil***

```
Method "120deg"  print. date 17.11.2000 11:07


Creator                                                 unknown
Creation date                                           17.11.2000 11:
07

Parameters
        Temperature                                     120 °C
        Delta T                                         1.60 °C
        Gas flow                                        20 L/h
        Use pump                                        Yes
        Start delay                                     0 min
        Start mode                                      per channel
        Stop criteria Time                              No
                Time                                    0.0 h
        Stop criteria Conductivity                      No
                Conductivity                            0 uS/cm
        Stop criteria Endpoint(s)                       Yes
        Shut off heater at end of determination         No
        Shut off gas flow at end of determination       No

Evaluation
        Evaluate induction time                         Yes
        Evaluate stability time                         No
                Delta kappa                             0
        Interpretation delay                            0.0 h
        Interpretation suppression Start                0.0 h
```

```
                Interpretation  suppression End                  0.0 h

        Curves
                Time-axis (x) scaling                           Automatic
                        End Value                               6.0 h
                Conductivity-axis (y) scaling                   Automatic
                        End Value                               200 uS/cm

        Formulas
                Formula 1 for induction time                    No
                        Formula R1 = InductionTime * 1.000/1.000 + 0.0
                Formula 2 for stability time                    No
                        Formula R2 = StabTime² * 0.000 + StabTime * 1.000/1.00
        0 +0.000
                Formula 3 for stability time                    No
                        Formula R3 = StabTime² * 0.000 + StabTime * 1.000/1.00
        0 +0.000

        Standards
                Standard formula active                         Yes
                        Formula according to                    AOCS Cd 12b-92
                        Factor                                  2.000
                        Target temperature                      110.0 °C

        Documentation
                Include Header                                  Yes
                Include Results                                 Yes
                        Include induction time                  Yes
                        Include stability time                  No
                        Include formula 1 result                Yes
                        Include formula 2 result                No
                        Include formula 3 result                No
                        Include standards result                Yes
                Include Curve(s)                                Yes
                Include Method Parameters                       Yes
                Include Description                             No
                Send to Printer                                 Yes
                Send to File                                    No
                        Directory

        Description
```

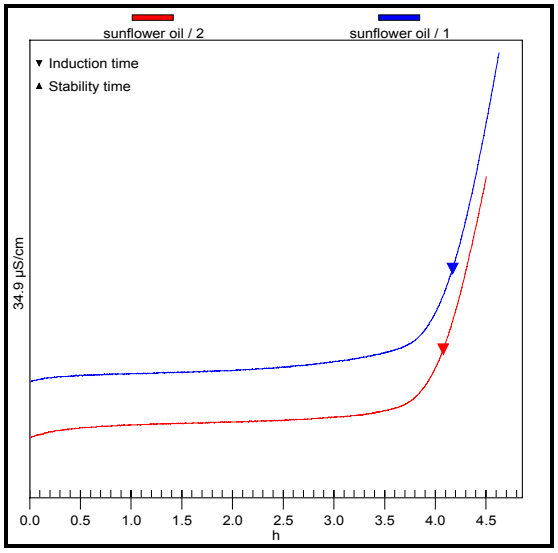
***Measuring the reproducibility in sunflower oil***

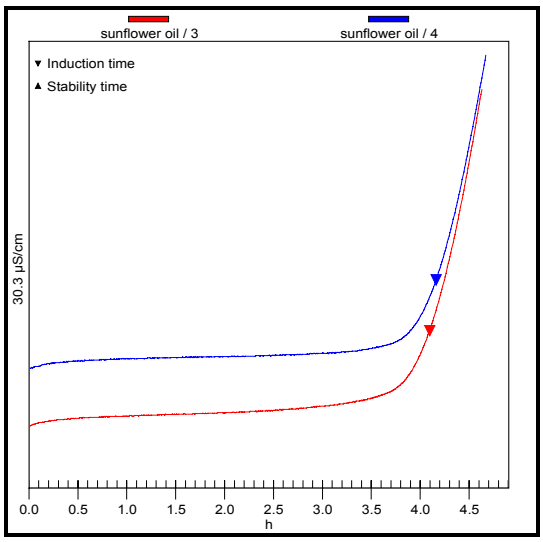
**Block A**

| ID 1 | Channe | Temperature | Induction time |
|---|---|---|---|
| sunflower oil | 2 | 110°C | 4.08 h |
| sunflower oil | 1 | 110°C | 4.17 h |

**Block B**

| ID 1 | Channe | Temperature | Induction time |
|---|---|---|---|
| sunflower oil | 2 | 110°C | 4.10 h |
| sunflower oil | 1 | 110°C | 4.16 h |



***Example of a validation record of the 743 Rancimat***

# *Validation Record 743 Rancimat*

| | | | |
|---|---|---|---|
| Company: | *Metrohm AG* | Division: | *Applications Lab* |
| Date: | *16 June 2000* | Time: | *15:30* |
| User: | *B. Zumbrägel* | | |
| Serial number: | *02136* | | |

## *1. Calibration points of the temperature sensor*

| Designation: | *no. 15* | Temperature [°C] | Resistance [Ohm] | |
|---|---|---|---|---|
| Point 1 | | *50* | *119.41* | |
| Point 2 | | *100* | *138.52* | |
| Point 3 | | *200* | *175.86* | |

## *2. Temperature validation*

| ***Block A*** | Temperature [°C] | Requested temperature [°C] | Tolerance | ***Block B*** | Temperature [°C] | Requested temperature [°C] | Tolerance |
|---|---|---|---|---|---|---|---|
| Channel 1 | *121.28* | *121.6* | ± 0.5 °C | Channel 1 | *121.44* | *121.6* | ± 0.5 °C |
| Channel 2 | *121.36* | *121.6* | ± 0.5 °C | Channel 2 | *121.52* | *121.6* | ± 0.5 °C |
| Channel 3 | *121.28* | *121.6* | ± 0.5 °C | Channel 3 | *121.52* | *121.6* | ± 0.5 °C |
| Channel 4 | *121.23* | *121.6* | ± 0.5 °C | Channel 4 | *121.44* | *121.6* | ± 0.5 °C |

☒ passed ☐ not passed

## *3.1. Validation of the conductivity inputs*

☒ test done ☐ test not done

| ***Block A*** | Conductivity [µS/cm] | Requested value [µS/cm] | Tolerance | ***Block B*** | Conductivity [µS/cm] | Requested value [µS/cm] | Tolerance |
|---|---|---|---|---|---|---|---|
| Channel 1 | *98.8* | 100 | ± 5 µS/cm | Channel 1 | *98.9* | 100 | ± 5 µS/cm |
| Channel 2 | *98.9* | 100 | ± 5 µS/cm | Channel 2 | *98.9* | 100 | ± 5 µS/cm |
| Channel 3 | *98.8* | 100 | ± 5 µS/cm | Channel 3 | *98.8* | 100 | ± 5 µS/cm |
| Channel 4 | *98.8* | 100 | ± 5 µS/cm | Channel 4 | *98.8* | 100 | ± 5 µS/cm |

☒ passed ☐ not passed

## 3.2. Validation of the conductivity measurement with conductivity standard

☒ test done ☐ test not done

| Temperature | | 23 °C | | | | | |
|---|---|---|---|---|---|---|---|
| **Block A** | Conductivity [µS/cm] | Requested value [µS/cm] | Tolerance | **Block B** | Conductivity [µS/cm] | Requested value [µS/cm] | Tolerance |
| Channel 1 | 141.3 | 141.0 | ± 10 µS/cm | Channel 1 | 141.5 | 141.0 | ± 10 µS/cm |
| Channel 2 | 141.2 | 141.0 | ± 10 µS/cm | Channel 2 | 141.3 | 141.0 | ± 10 µS/cm |
| Channel 3 | 140.9 | 141.0 | ± 10 µS/cm | Channel 3 | 141.5 | 141.0 | ± 10 µS/cm |
| Channel 4 | 141.0 | 141.0 | ± 10 µS/cm | Channel 4 | 141.1 | 141.0 | ± 10 µS/cm |

☒ passed ☐ not passed

## 4. Validation of the temperature correction

☒ test done ☐ test not done

| Temperature | | 120 °C | | | | | |
|---|---|---|---|---|---|---|---|
| **Block A** | Method | | | **Block B** | Method | | |
| | Delta T [°C] | Old delta T [°C] | Tolerance | | Delta T [°C] | Old delta T [°C] | Tolerance |
| | 1.60 | 1.60 | ± 0.3 °C | | 1.60 | 1.60 | ± 0.3 °C |

☒ passed ☐ not passed

## 5. Determination of the reproducibility in oil

☒ test done ☐ test not done

| Method | | R_110 | | Method | | R_110 | |
|---|---|---|---|---|---|---|---|
| Temperature | | 110 °C | | Temperature | | 110 °C | |
| Sample | | sunflower oil | | Sample | | sunflower oil | |
| **Block A** | Induction time [h] | Difference of induction times [h] | Tolerance (10% of mean) [h] | **Block B** | Induction time [h] | Difference of induction times [h] | Tolerance (10% of mean) [h] |
| Determin. 1 | 4.08 | 0.09 | 0.41 | Determin. 1 | 4.10 | 0.06 | 0.41 |
| Determin. 2 | 4.17 | | | Determin. 2 | 4.16 | | |
| Mean | 4.13 | | | Mean | 4.13 | | |

☒ passed ☐ not passed

| Signature: | | Visa: | |
|---|---|---|---|

# ***Validation Record 743 Rancimat***

| | | | |
|---|---|---|---|
| Company: | | Division: | |
| Date: | | Time: | |
| User: | | | |
| Serial number: | | | |

## *1. Calibration points of the temperature sensor*

| Designation: | | Temperature [°C] | Resistance [Ohm] | |
|---|---|---|---|---|
| Point 1 | | | | |
| Point 2 | | | | |
| Point 3 | | | | |

## *2. Temperature validation*

| ***Block A*** | Temperature [°C] | Requested temperature [°C] | Tolerance | ***Block B*** | Temperature [°C] | Requested temperature [°C] | Tolerance |
|---|---|---|---|---|---|---|---|
| Channel 1 | | | ± 0.5 °C | Channel 1 | | | ± 0.5 °C |
| Channel 2 | | | ± 0.5 °C | Channel 2 | | | ± 0.5 °C |
| Channel 3 | | | ± 0.5 °C | Channel 3 | | | ± 0.5 °C |
| Channel 4 | | | ± 0.5 °C | Channel 4 | | | ± 0.5 °C |

☐ passed ☐ not passed

## *3.1. Validation of the conductivity inputs*

☐ test done ☐ test not done

| ***Block A*** | Conductivity [µS/cm] | Requested value [µS/cm] | Tolerance | ***Block B*** | Conductivity [µS/cm] | Requested value [µS/cm] | Tolerance |
|---|---|---|---|---|---|---|---|
| Channel 1 | | 100 | ± 5 µS/cm | Channel 1 | | 100 | ± 5 µS/cm |
| Channel 2 | | 100 | ± 5 µS/cm | Channel 2 | | 100 | ± 5 µS/cm |
| Channel 3 | | 100 | ± 5 µS/cm | Channel 3 | | 100 | ± 5 µS/cm |
| Channel 4 | | 100 | ± 5 µS/cm | Channel 4 | | 100 | ± 5 µS/cm |

☐ passed ☐ not passed

### *3.2. Validation of the conductivity measurement with conductivity standard*

☐ test done ☐ test not done

| Temperature | | °C | | | | | |
|---|---|---|---|---|---|---|---|
| ***Block A*** | Conductivity [µS/cm] | Requested value [µS/cm] | Tolerance | ***Block B*** | Conductivity [µS/cm] | Requested value [µS/cm] | Tolerance |
| Channel 1 | | | ± 10 µS/cm | Channel 1 | | | ± 10 µS/cm |
| Channel 2 | | | ± 10 µS/cm | Channel 2 | | | ± 10 µS/cm |
| Channel 3 | | | ± 10 µS/cm | Channel 3 | | | ± 10 µS/cm |
| Channel 4 | | | ± 10 µS/cm | Channel 4 | | | ± 10 µS/cm |

☐ passed ☐ not passed

### *4. Validation of the temperature correction*

☐ test done ☐ temperature correction used from table

| Temperature | | °C | | | | | |
|---|---|---|---|---|---|---|---|
| ***Block A*** | Method | | | ***Block B*** | Method | | |
| | Delta T [°C] | Old delta T [°C] | Tolerance | | Delta T [°C] | Old delta T [°C] | Tolerance |
| | | | ± 0.3 °C | | | | ± 0.3 °C |

☐ passed ☐ not passed

### *5. Determination of the reproducibility in oil*

☐ test done ☐ test not done

| Method | | | | Method | | | |
|---|---|---|---|---|---|---|---|
| Temperature | | °C | | Temperature | | °C | |
| Sample | | | | Sample | | | |
| ***Block A*** | Induction time [h] | Difference of induction times [h] | Tolerance (10% of mean) [h] | ***Block B*** | Induction time [h] | Difference of induction times [h] | Tolerance (10% of mean) [h] |
| Determin. 1 | | | | Determin. 1 | | | |
| Determin. 2 | | | | Determin. 2 | | | |
| Mean | | | | Mean | | | |

☐ passed ☐ not passed

| Signature: | ✕ | Visa: | ✕ |
|---|---|---|---|