

Validation of the 774 Oven Sample Processor in combination with a KF Coulometer by using standard operating procedures

Of interest to:

General analytical laboratories; pharmaceuticals; petrochemistry; plastics, photo industry; food analysis
F 1, 4, 5, 6, 7

Summary

GLP (Good Laboratory Practice) requires, among other things, the regular checking of the accuracy and precision of analytical instruments by using standard operating procedures (= SOPs).

The user is recommended to validate Metrohm instruments as a whole integral measuring system, i.e. to carry out a series of determinations with certified standards and to critically assess the results by statistical means.

Checking the electronic components of measuring instruments can and ought to be carried out in the context of regular service work by technical specialists from the manufacturing company. All Metrohm instruments are provided with start-up test routines that check that the relevant components are functioning properly when the instrument is switched on. If this produces no error message then it can be assumed that the instrument is functioning perfectly. Metrohm also supplies its instruments with built-in diagnosis programs that, if faults or malfunctions should occur, permit the user to check the functioning of particular assemblies and localize the fault. These diagnosis programs can also be included in a validation method.

As a guideline for drawing up a standard operating procedure for checking the complete analytical system Metrohm recommends the procedure described below. The limiting values mentioned should be regarded as being proposals. Depending on your company-specific requirements for the accuracy of the measuring system these limits may need to be redefined in the standard operating procedures.

Range of application

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

774 Oven Sample Processor in combination with
756 or 831 KF Coulometer

In a similar way validation can also be carried out with the 832 KF Thermoprep.

Test periods

Carrying out the check at yearly intervals seems to be advisable. If an instrument is in continuous use then a shorter checking interval may be required, e.g. every six months or even every three months.

An exceptional validation is required if one or more components of the analytical system are replaced.

Internal test routines

Metrohm instruments have internal start-up test und test routines. During the start-up test the display elements are checked and the contents of the program memory are checked using a check-sum test. The functioning of the data memory area is checked by using a write/read test. In addition, the RS-232 interface is subjected to a detailed test.

If your instruments are serviced at regular intervals you can usually dispense with the specific validation of the instrument electronics.

Service and maintenance

An essential prerequisite for GLP-conform operation of all the instruments used in the laboratory is their thorough care and cleaning and, in particular, the proper handling of the instruments. The Instructions for Use supplied with the instrument should be available to all laboratory workers. We also recommend that all the relevant instruments are subjected to a regular annual service. Many Metrohm agencies can offer service contracts for your instruments.

Instruments and accessories

«Stand-alone»-system:

- 2.774.0010 Oven Sample Processor
- 2.756.0X10 KF Coulometer or
2.831.0X10 KF Coulometer
- 6.2419.000 Sample vials for 774
- 6.1448.050 Septum seals
- 6.1808.040 Adapter for needle
- 6.1808.090 Adapter for needle
- 2.728.0010 Magnetic stirrer
- 6.2141.020 Remote cable 774 – 756/831
- 6.2125.110 Cable 774 – 756/831
- 6.5618.000 Test tool for 774 oven temperature
- 2.767.0010 Calibrated Reference
- Certified, calibrated temperature measuring device (not available from Metrohm)
- Certified, calibrated flowmeter (not available from Metrohm)
- Analytical balance (min. resolution 0.1 mg)

For a system controlled by *tiamo* the following cables are additionally required:

- 6.2125.060 Cable 774 – PC
- 6.2134.040 Cable 756/831 – PC

The Metrodata *tiamo* full software (6.6056.102) is supplied free-of-charge together with the 774 Oven Sample Processor.

Reagents

- HYDRANAL Coulomat AG Oven, Riedel-de Haën # 34739
- HYDRANAL Standard sodium tartrate dihydrate, Riedel-de Haën # 34803
- HYDRANAL Water Standard KF Oven (potassium citrate monohydrate), Riedel-de Haën # 34748
- Molecular sieve, pore size 0.3 nm, Metrohm # 6.2811.000
- Carrier gas (air or N₂)

Individual instrument checks

The analytical system consisting of 774 Oven Sample Processor and 756 or 831 KF Coulometer (with or without *tiamo*) is installed, connected and configured according to the Instructions for Use.

1. Oven Sample Processor 774

Checking the **oven temperature** of the 774 requires the use of the 6.5618.000 Test tool, which consists of a measuring insert for the heating block of the Oven Sample Processor and a temperature sensor. The temperature sensor is inserted so far into the measur-

ing insert that it reaches the base inside the brass body and is then fixed in position using the Allen wrench supplied. The complete test tool is then connected to a certified, calibrated thermometer (not available from Metrohm) and then inserted like a sample vessel into the oven block of the 774 and fixed in position (see Metrohm Leaflet No. 8.110.1661). The keyboard of the 774 Oven Sample Processor is used to set the temperature to be checked to 160 °C (if sodium tartrate dihydrate is later to be used for the validation of the whole system) or 220 °C (if potassium citrate monohydrate is to be used). During the temperature measurement the gas flow must be switched off, as the carrier gas flow through the needle could produce cooling effects.

Key sequence on the 774 keyboard:

Switch off pump/valve:

```
<4>/<FLOW>  
<ENTER>  
<SELECT> (to «Off»)  
<ENTER>
```

Set oven temperature:

```
<5>/<HEATER>  
«160» or «220»  
<ENTER>  
<ENTER>
```

Wait until the 774 Oven Sample Processor has reached the selected temperature. After a further 20 min, which is required to establish the temperature equilibrium between the oven and the brass body of the test tool, the temperature is read off from the thermometer. Compare the actual temperature with the set oven temperature. The **difference** should be **<±4 °C**. For oven temperatures below 150 °C this limit is greatly undercut; at higher temperatures the variation could be up to ±4 °C because of thermal radiation losses from the test tool; these result from its design. At oven temperatures above 200 °C a larger deviation may be measured (**difference <± 6°C**).

If *tiamo* is used then, after the measurement, the oven temperature of the 774 is reset manually to «INIT»:

```
<5>/<HEATER>  
<SELECT> (to «INIT»)  
<ENTER>  
<ENTER>
```

In a system without *tiamo* the selected temperature is retained for the subsequent measuring method using the particular standard chosen.

In order to check the **carrier gas flow of the 774 Oven Sample Processor** a certified, calibrated flowmeter (not available from Metrohm) is included in the carrier gas flow at the upper end of the tower of the Oven Sample Processor. The outlet line of the external flowmeter remains open. The gas outlet and inlet openings («to drying flask» and «from drying flask») at the left-hand side of the 774 tower are bridged with a piece of tubing, i.e. the drying flask is excluded/bridged. The type of carrier gas must be set both at the internal and external flowmeters (air or N₂). As a solid will be used as the standard for the subsequent validation of the complete system, the gas flow is set to 40 mL/min. The test flowmeter is first purged with the corresponding carrier gas for 1 min. The measured values are compared after a further 2 min. Please note that the internal flowmeter of the 774 Oven Sample Processor has been calibrated at a temperature of 0 °C and a pressure of 1.013 bar. If the calibration of the external flowmeter has not been carried out under standard conditions then its measured value must be converted accordingly (see Metrohm Leaflet No. 8.110.177). Otherwise incorrect differences would be obtained and the variation between the two instruments could possibly be larger than the **range of tolerance of ±20% of the nominal value** (for the flowmeter of the 774). The values set here are retained for the subsequent validation of the whole system.

2. 756/831 KF Coulometer

The 756 or 831 KF Coulometer is checked by using the 767 Calibrated Reference. The procedure is carried out exactly as given in the protocol for checking the measuring amplifier (see Literature).

Validation of the whole analytical system

In order to validate the whole system consisting of the 774 Oven Sample Processor and 756 or 831 KF Coulometer the measured results from a series of 6 to 10 analyses of a special KF oven standard are used. We recommend the use of certified solid substances, such as sodium tartrate dihydrate (as per USP 24 <921> Water Determination) or potassium citrate monohydrate. These reagents can be obtained from leading chemical manufacturers.

Certified solid standard substances are available with the following water content:

Sodium tartrate dihydrate	(15.66 ± 0.05)%
Potassium citrate monohydrate	(5.55 ± 0.05) %

Special attention must be given to the heating period for solid substances. It must be selected so that the transfer of the whole amount of water is assured while the duration of the analysis still remains within reasonable limits (<30 min).

Requirements

- KF instruments should not be set up in rooms subject to high temperature variations. They must not be placed beside heating sources or cooling thermostats. If there are water baths or sinks in the immediate vicinity then an adequate protection against splashes must be provided.
- The test setup must be protected against direct sunlight and drafts must be avoided. The system must be in thermal equilibrium.
- The balance should previously have been validated.
- The carrier gas used is usually air or N₂.
- The sample vials and their septa must have been conditioned to the ambient surroundings of the laboratory for at least one day before use.
- When carrying out the KF water determination gloves must be worn, as skin moisture could otherwise influence the precision of the results.
- The determination series should be carried out without any interruption.

Procedure

1. Setting the titration parameters

Standard parameters can normally be used. The relevant parameters are summarized in Tab. 1.

Tab. 1: Control and titration parameters.

	Parameter	Setting
Control parameter Coulometry:	EP at U	50 mV
	Control range	70 mV
	Max.Rate	max. µg/min
	Min.Rate	15 µg/min
	StopCrit	rel.Drift
	rel.Drift	5 µg/min
Titration parameter Coulometry:	Start Drift	10 µg/min
	I(pol)	10 µA
Preselection Coulometry:	Generator I*	400 mA
Oven settings:	Initial temp.	depends on sample, normally 50 °C
Gas flow:	Flow rate**	40...60 mL/min
	Gas type	air or N ₂
	Factor gas flow	1.0 (for air or N ₂)

* For a Generator electrode with diaphragm the parameter Generator I is set to «auto».

** The flow rate of the carrier gas for solid substances is set to 40 mL/min (for liquid substances normally to 60 mL/min).

2. Carrying out the analyses

1. When the individual tests have been carried out you must ensure that the system is returned to a

proper functioning condition, i.e. all electrode cables and tubing must again be connected correctly.

2. In the validation the double hollow needle of the 774 Oven Sample Processor is used with a shortened piercing needle for solid samples (see Fig. 1) This requires the use of special adapters (6.1808.040 and 6.1808.090). These penetrate only a few millimeters deeper into the sample vial than the exhaust needle. This prevents both the sample from being turbulently mixed and the contamination of the following samples and the conditioning vessel by substances adhering to the needle tip.

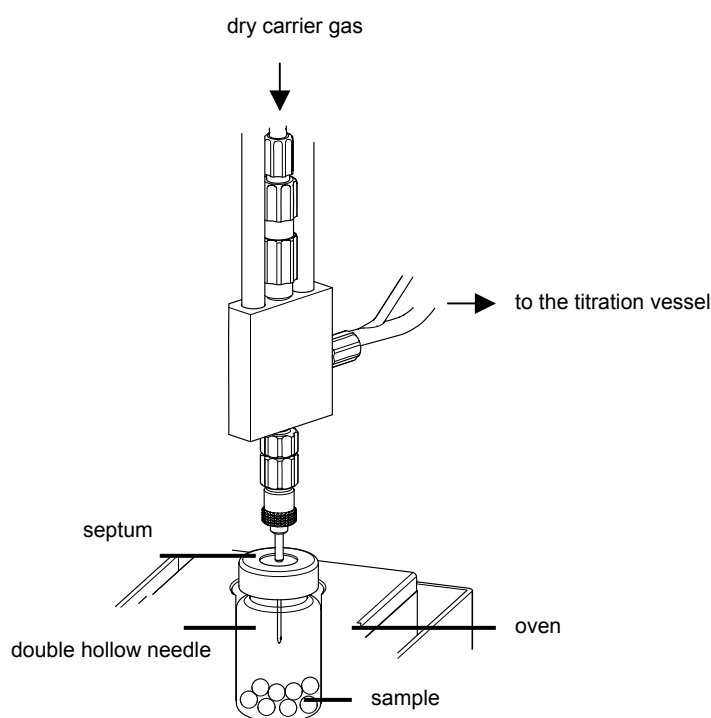


Fig. 1: Sample vial with double hollow needle (headspace technique).

3. The titration vessel is filled with 150 mL of the reagent HYDRANAL Coulomat AG Oven (Riedel-de Haën # 34739) and closed off with a drying tube (containing fresh molecular sieve). The electrodes and the carrier gas supply are led into the vessel. Care must be taken that everything is absolutely tight with respect to penetration by atmospheric humidity.

4. Each analysis consists of the following steps:

- Conditioning the titration vessel
- Water extraction from the sample
- Water transport to the titration vessel
- Karl Fischer titration
- Calculation of the result

During conditioning the titration vessel is titrated to dryness under stirring, with the 774 Oven Sample Processor being in the so-called conditioning status. This conditioning step must be carried out before each determination. Then the water is driven out from the sample with a flow of dry carrier gas combined with the increased oven temperature and transported to the titration vessel where the determination itself takes place by coulometric Karl Fischer titration.

5. 6 to 10 determinations are carried out using the same instrument settings and different weights of the standard. The weights of the standard should be varied in random order; the upper limit is given by the reagent capacity of 1000 mg water per 100 mL. For solid standard substances we recommend the use of weights in the range 40 ... 80 mg.

To prepare a determination series the corresponding amounts of standard are weighed out into the sample vials and these are sealed with septa. It is essential that gloves are worn for this.

The sample rack is filled in the following sequence:

- 1 x system preparation
- 3 x blank values
- 6...10 x standards.

In addition, an empty conditioning vessel is required in special position No. 36.

In order to check that the whole system is functioning properly a so-called system preparation vial (empty) is placed on the sample rack before each series of determinations. This is followed by three blank value vials. The mean of the blank value determinations is stored as a Common Variable and taken into account when calculating the result (subtracted). The system preparation and the blank value determinations must be carried out under exactly the same conditions as the analyses of the samples.

6. The sample data together with the associated methods are entered or called up in the sample table of the **tiamo** software or in the silo of the 756/831 KF Coulometer. If a **tiamo**-controlled system is used these are the following methods:

- 1 x 774 – Coulometer - systemprep
- 3 x 774 – Coulometer - blank value
- 6...10 x 774 – Coulometer - water content

These are standard **tiamo** methods.

In a «stand-alone» system the corresponding methods must be set at the 756/831 KF Coulometer (Blank, KFC-B) and 774 Oven Sample Processor. The silo and statistics function are activated by pressing the keys <9>/<SILO> and <7>/<STATISTICS> on the KF Coulometer.

7. If **tiamo** is used then the software takes complete control. The determination series is started via button <START>.

Without **tiamo** the 774 Oven Sample Processor takes control; the series is then started by pressing the <START> key on the 774. When this is done the KF Coulometer must be in the STOP condition.

Software control is always to be preferred, also because of the simple data storage in a database that is GLP-conform.

The determination series is started and processed without any interruptions.

3. Practical recommendations

Water is present everywhere. Ensure that your working technique is absolutely 'water-free'. You must ensure that the titration vessel is completely tight; it must be checked at regular intervals. The KF cell should be equipped with a drying tube (6.1403.030) filled with molecular sieve. It should be opened as seldom as possible.

A clean and exact working procedure is assumed as a matter of course and is an essential prerequisite for the validation of Karl Fischer titrators.

Evaluation of the result

For the validation of measuring instruments the precision (degree of scatter) and the accuracy of the measured results are relevant. These quantities are evaluated in the following way:

The values obtained from the 6-10 determinations are used to calculate the mean value \bar{x} and the absolute standard deviation s_{abs} . These calculations can be carried out directly in the instrument with the built-in statistics function, if available; by using suitable software (e.g. **tiamo**, spread-sheet analysis) on a PC or by using a pocket calculator.

Complex calculations carried out by various calculation aids can produce slightly differing results because of the different degrees of internal accuracy involved; this means that the values calculated internally by the instrument should always be preferred.

Mean value

$$\bar{x} = \frac{x_1 + x_2 + \dots + x_n}{n} = \frac{1}{n} \sum_{i=1}^n x_i = \frac{\text{sum of individual value}}{\text{number of individual value}}$$

Standard deviation

$$s_{abs} = \sqrt{\frac{1}{n-1} \sum_{i=1}^n (x_i - \bar{x})^2} = \sqrt{\frac{\sum_{i=1}^n x_i^2 - \frac{\left(\sum_{i=1}^n x_i\right)^2}{n}}{n-1}}$$

1. Precision or repeatability

The precision of the measurement is expressed by the relative standard deviation.

Rel. standard deviation

$$s_{rel} = \frac{s_{abs} \times 100}{\bar{x}} = \frac{\text{abs. std. deviation} \times 100}{\text{mean value}}$$

Requirement: the relative standard deviation must be $\leq 1.0\%$.

2. Accuracy

The correctness is solely based on the manufacturer's guaranteed and certified water content of the standard. Please note that you should only use the value found and guaranteed by the manufacturer (and not the general value given on the label).

Requirement: the recovery rate should be between 98-102%.

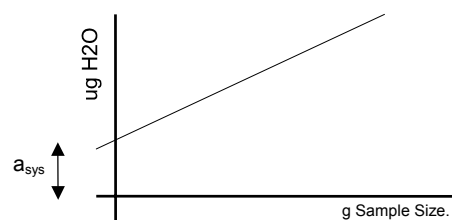
$$\text{Recovery rate} = \frac{\text{mean value} * 100}{\text{theoretical value}}$$

3. Systematic errors

a. Linear regression $\mu\text{g H}_2\text{O}/\text{sample weight}$

In order to discover systematic errors, e.g. method-specific errors, technical working problems, a linear regression can be calculated from $\mu\text{g H}_2\text{O}$ against the sample weight in g. A powerful pocket calculator or a PC with a statistics package or spread-sheet program should be used for this. The sample weight is plotted as the x-coordinate (independent variable) and the $\mu\text{g H}_2\text{O}$ found as the y-coordinate (dependent variable). The linear regression constructs a straight line through the measuring points so that the sum of the squares of the individual variations is at a minimum. The regression curve is described by the equation: $y = bx + a$, with "a" standing for the y-axis intercept and "b" for the slope of the curve (see below drawing).

Systematic errors are indicated by a significant variation of the zero point coordinate of the y-axis (intercept), i.e. the regression curve calculated from the pairs of values $\mu\text{g H}_2\text{O}/\text{sample weight}$ does not cut through the y-axis at exactly the origin of the coordinate system.



a_{sys} as a measure for the systematic error is calculated from the mean value of the x-values, the mean value of the y-values and the coefficient of regression b (slope).

Calculation formula:

$$b = \frac{\sum_{i=1}^n (x_i - \bar{x})(y_i - \bar{y})}{\sum_{i=1}^n (x_i - \bar{x})^2} = \frac{\sum_{i=1}^n x_i y_i - \frac{\sum_{i=1}^n x_i * \sum_{i=1}^n y_i}{n}}{\sum_{i=1}^n x_i^2 - \frac{(\sum_{i=1}^n x_i)^2}{n}}$$

$$a_{\text{sys}} = y\text{-axis intercept} = \bar{y} - b * \bar{x}$$

Any blank value obtained must be subtracted from a_{sys} .

Evaluation:

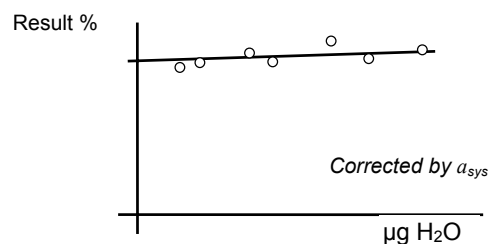
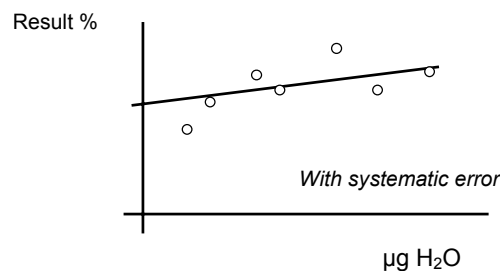
Should $a_{\text{sys}} > \pm 200 \mu\text{g H}_2\text{O}$ then it must be assumed that there is a systematic error. A check of all possible system-specific sources of interference can then no longer be avoided. If it is not possible to optimize the working technique then the individual values obtained must be corrected by the value of a_{sys} (read off $-a_{\text{sys}}$ in $\mu\text{g H}_2\text{O}$), so that the systematic error of the method is not included in the evaluation of the titrator. As a result the relevant characteristic data for the reproducibility and correctness of the titration results must be recalculated using the corrected values. This correction of the systematic error must, if applicable, also be used for sample titrations.

b. Linear regression calculated % H₂O/found $\mu\text{g H}_2\text{O}$

A further possible method for discovering systematic errors is the presentation of the regression curve (scatter diagram) of the pairs of values % H₂O/found $\mu\text{g H}_2\text{O}$. As this diagram also allows a good visual impression of the scattering of the results, we recommend that you produce such a diagram.

Significant positive or negative slopes of the regression curve indicate an apparent dependency of the % H₂O on the amount of $\mu\text{g H}_2\text{O}$. This can also be an indication of a method-specific systematic error.

The slope $b_{T/Vol}$ (coefficient of regression b, calculation formula see above) from the curve equation $y = bx + a$ should under ideal circumstances be 0.000, i.e. the curve should be a horizontal line through $y = \text{theoretical value}$.



Evaluation:

If b is larger than $\pm 0.01\%/ \mu\text{g}$ then it must also be assumed that there is a systematic error. A correction of the found value ($\mu\text{g H}_2\text{O}$) by a_{sys} ($\mu\text{g H}_2\text{O} - a_{\text{sys}}$ in μg) and a subsequent recalculation of the value will show a dramatic improvement in the presentation of the regression curve (% H₂O against $\mu\text{g H}_2\text{O}$).

Summary:

If systematic errors are present then an attempt must be made to optimize the working technique and to adapt the Standard Operating Procedure (SOP) accordingly. If no optimization is possible or if a given method is used without alteration then the calculation of the relevant characteristic data should be carried out with corrected consumption values ($\mu\text{g H}_2\text{O}$ found - a_{sys} in μg).

Troubleshooting recommendations

If the relative standard deviation is too high (poor precision):

- Optimize the titration and control parameters (see Metrohm Application Bulletins under Literature references).
- Check (tubing) connections.
- Check whether the titration cell is contaminated, the septum and seals are leaking, the molecular sieve has been consumed or whether conditioning has been poor (too high or scattered start drift).
- Check electrode and titration parameters. Clean the platinum electrode mechanically with a moist cleaning agent (aluminum oxide or toothpaste), rinse and dry it, treat it with conc. HNO₃ or replace it. Align the Pt pins so that they are parallel.
- Clean generator electrode with conc. HNO₃.
- Use fresh certified standard substances.
- Check whether the reagent has been contaminated or is exhausted. Use fresh reagent (possibly from a different manufacturing batch).

- Check the oven heating temperature.
- Check whether the sample vials closures are tight; it must not be possible to rotate the closures.
- Balance/weighing in: balance not accurate enough, drafts, temperature influences, contamination, temperature difference between balance and sample, careless weighing, sample weight too low.

Procedure if values do not meet specifications

Comments must be made about all anomalous values in the validation protocol and the further procedure must be mentioned.

If variations that are too high are found then the various points mentioned under "Troubleshooting recommendations" must be conscientiously checked and the errors eliminated. It is essential that the validation process is repeated. If satisfactory results still cannot be obtained when the test series is repeated then the validation process should be carried out again by a different person on a different day.

All Metrohm instruments have special diagnosis programs that allow instrument malfunctions to be identified and localized. If there is a justified suspicion that faults are present in the instrument (repeatedly occurring errors, large scatter) then the diagnosis program must be carried out conscientiously and step by step. You can read about the exact procedure in the Instructions for Use of the particular instrument.

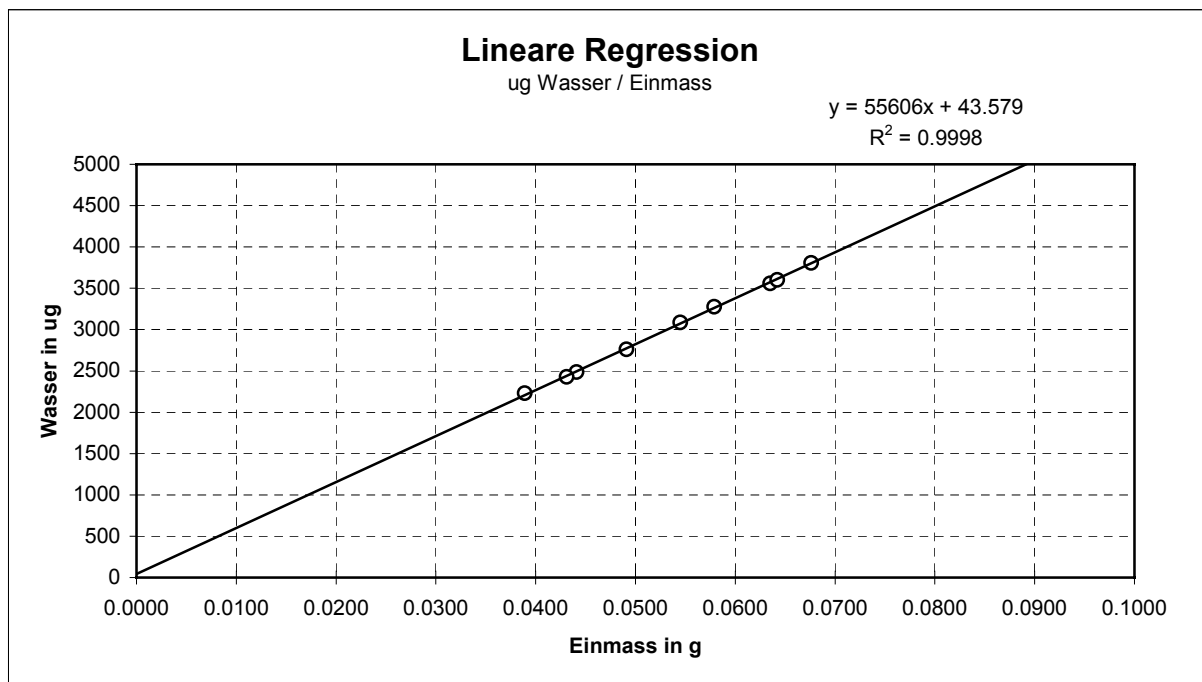
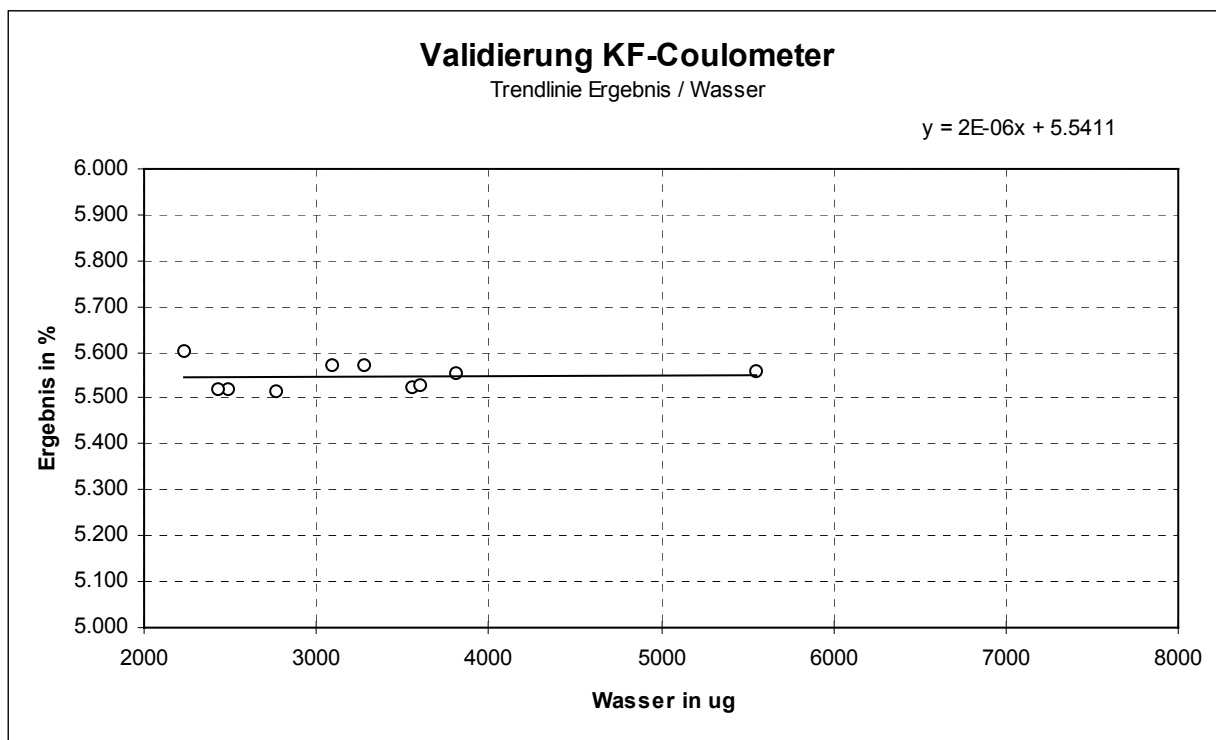
If a malfunction can be localized during the diagnosis then this should be reported to your local Metrohm Service Department and the instrument should no longer be used until the malfunction has been remedied.

Literature

- Metrohm Monograph No. 8.023.5003: Water determination by Karl Fischer Titration, Metrohm 2003
- Hydranal®-Praktikum, Wasserreagenzien nach Eugen Scholz für die Karl-Fischer-Titration. Riedel-de Haën, Ausgabe 1996.
- ISO 9000 und die Karl Fischer-Titration, Riedel de Haën 1996.
- Metrohm Instructions for Use: 774 Oven Sample Processor.
- Metrohm Instructions for Use: 756/831 KF Coulometer.
- Metrohm Instructions for Use: 767 Calibrated Reference.
- 756, 831 Protocol of measuring amplifier check mvp756d030505.doc
- Metrohm leaflet 8.110.1663: Measuring set for temperature checking of the 774 Oven Sample Processor Metrohm
- Metrohm leaflet 8.110.17731: Gas flow validation of the 774 Oven Sample Processor.

On the following pages you will find examples of validation records as well as diagrams of the described linear regressions. The last page can be used as master for copies of the validation records.

Validierungsprotokoll				Firma: Metrohm AG, Herisau	
KF-Coulometer				Abteilung: Appl.Lab.- Marketing Titration	
Reagenz:	HYDRANAL-Coulomat AG Oven		Gerät:	756 KF-Coulometer & 774 Oven Sample Pr.	
	Nr. 34739	Riedel-de Haën	Modus:	KFC	
Charge/Lot:	2720		Elektrode:	Gen. ohne Diaphragma	6.0345.100
Standard:	HYDRANAL-Water Standard KF-Oven				
Wassergehalt:	5.59 %				
S _{abs} :	+/- 0.05 %		Temperatur in °C:	220	
Charge / Lot:	62760		Blindwert [ug]:	51.5	
<u>Titrierparameter:</u>					
Endpunkt	50 mV	Max. Rate	max ug/min		
Regelbereich	70 mV	Min. Rate	15 ug/min		
Startdrift	10 ug/min	N2	40 mL/min		
Stopdrift	rel. Drift 5 ug/min				
I _{pol}	10 uA				
Generator I	400 mA				
Einmass:	Wasser:	Ergebnis [%]:	No.	Kommentar :	
0.0491	2760.30	5.5169	1		
0.0441	2485.00	5.5181	2		
0.0635	3560.20	5.5255	3		
0.0579	3278.20	5.5729	4		
0.0989	5548.30	5.5579	5		
0.0389	2231.10	5.6031	6		
0.0545	3087.10	5.5699	7	Mittelwert [%]: 5.5468	S _{abs} : 0.0292
0.0676	3805.20	5.5528	8	a _{sys} : 43.579 ug	S _{rel} : 0.53 %
0.0642	3602.00	5.5304	9	a _{syskor} : -7.921 ug	b _{sw/ug} : 0.000002
0.0431	2430.00	5.5202	10	Wiederfindung [%]: 99.23	
<u>Entscheid:</u> Validierung i. O.					
Datum: 13.06.2001		Unterschrift: <i>R. Schlink</i>		Visum: <i>red</i>	



Validation protocol				Company:	
				Department:	
KF-Coulometer				Instrument:	
				Mode:	
Reagent:				Electrode:	
Charge/Lot:				Standard:	
Watercontent:				Temperature in °C:	
S _{abs} :				Blankvalue [ug]:	
Charge/Lot:					
<u>titration parameter</u>					
Sample size [g]	Water [ug]	Content [%]:	No.	Comment:	
			1		
			2		
			3		
			4		
			5		
			6		
			7	Mean [%]:	S _{abs} :
			8	a _{sys} :	S _{rel} :
			9	a _{syskorr} :	b _{sw/ug} :
			10	Recovery [%]:	
<u>Adjudication</u>					
Date:		Signature:		Visa:	

