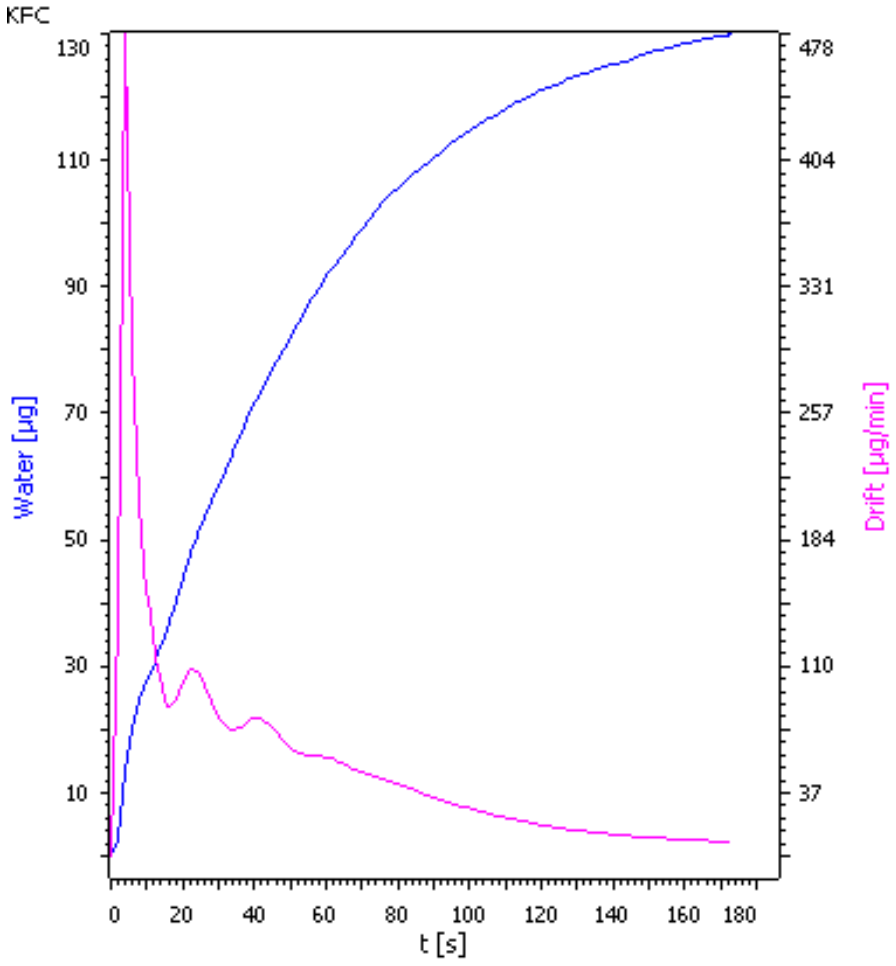
899库仑水分测定仪联用885全自动卡式炉样品处理器测定变压器油中水分含量

KF Application Note K–47

Determination of the water content in transformer oil with 885 Compact Oven Sample Changer and 899 Coulometer



This Application Note describes the determination of the water content in transformer oil using the oven technique.

Metrohm

Method description

Sample

Transformer oil (fresh)

Sample preparation

Approximately 4 g of sample were weighed into the sample vial, tightly closed with the cap, and placed on the rack of the 885 Compact Oven Sample Changer.

Electrodes

Double Pt-wire electrode	6.0341.100
Generator electrode with diaphragm	6.0344.100

Solutions

HYDRANAL®-Coulomat AG Oven	Fluka 34739
HYDRANAL®-Coulomat CG	Fluka 34840

Instrumentation

885 Compact Oven Sample Changer	2.885.0010
899 Coulometer	2.899.0010
Needle holder 58 mm	6.2049.040
Remote cable	6.2141.390

Analysis

All measurements were carried out at the same temperature using the same parameters.

In a first step, the titration vessel was conditioned. Then a determination with an empty sample vial was carried out to prepare the system and rinse all tubing. Following the system preparation, three blank values (empty sample vials) were determined and the mean value of the blank was saved as common variable. Since half of the sample vial was filled with sample, half of the blank value was subtracted from the EP of the sample determination (for more information on blank values, see AN-K-48). Subsequently, the water content of the samples was determined. Between two sample measurements, the titration vessel was conditioned again.

Parameters 899 Coulometer

Conditioning	on
Start drift	10 µg/min
Drift correction	auto
Automatic start	off
Stabilizing time	10 s
Cond. stop time	off
Measured value display	off
Pause	0 s
Request sample ID	off
Request sample size	off
Request sample unit	off
Hold at request	off
Endpoint at	50 mV
Titration rate	optimal
Stop criterion	rel. drift
Relative stop drift	10 µg/min
Extraction time	120 s
Generator electrode	with diaphragm
Generator current	auto mA
Stirrer	on
Stirring rate	15
I(pol)	10 µA
Electrode test	off
Time interval MP	2 s
Temperature	25 °C
Stop time	off

Parameters 885 Compact Oven Sample Changer

Temperature	105 °C
Flow rate	100 mL/min
Gas supply	valve
Gas type	nitrogen
End of series	conditioning

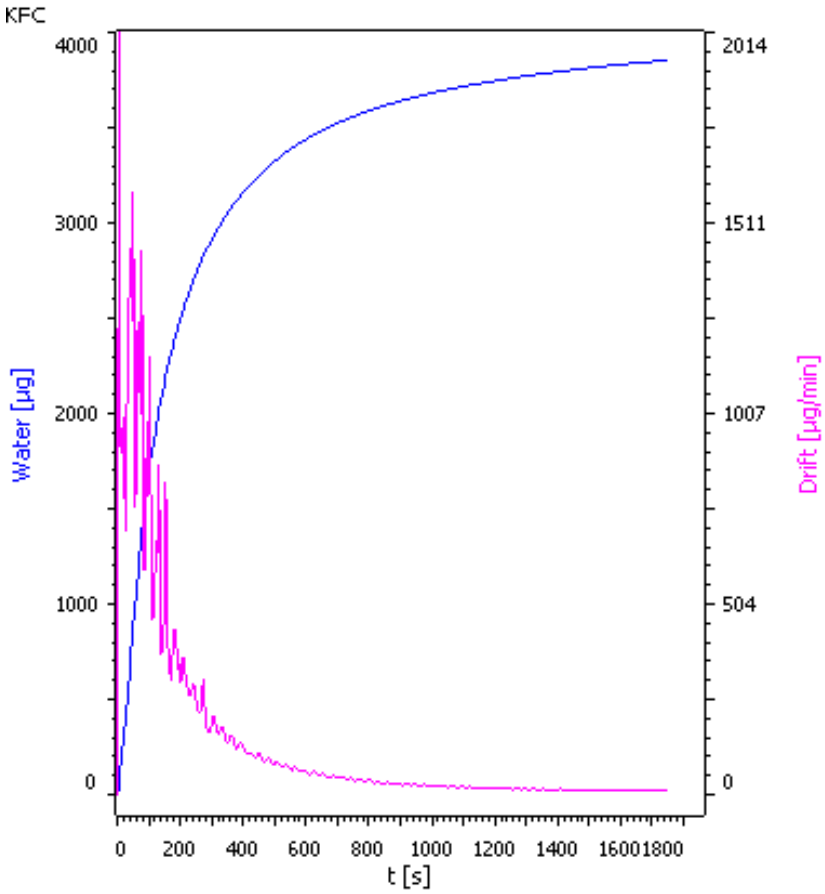
Results

Mean (n = 10) [µg/g]	RSD [%]
20.5	1.56

899库仑水分测定仪联用885全自动卡式炉样品处理器测定明胶中水分含量

KF Application Note K–50

Determination of the water content in gelatine with 885 Compact Oven Sample Changer and 899 Coulometer



This Application Note describes the determination of the water content in gelatine using the oven technique.

Metrohm

Method description

Sample

Gelatine (leaves)

Sample preparation

The sample was cut into small pieces and approximately 40 mg were weighed into the sample vial, tightly closed with the cap and placed on the rack of the 885 Compact Oven Sample Changer.

Electrodes

Double Pt-wire electrode	6.0341.100
Generator electrode without diaphragm	6.0345.100

Solutions

HYDRANAL®-Coulomat AG Oven	Fluka 34739

Instrumentation

885 Compact Oven Sample Changer	2.885.0010
899 Coulometer	2.899.0010
Remote cable	6.2141.390

Analysis

All measurements were carried out at the same temperature using the same parameters.

After starting the sample series, the Sample Changer moves to the conditioning vial, the needle pierces the septum, the gas flow is started and the titration vessel is conditioned. Then a determination with an empty sample vial is carried out to prepare the system and rinse all tubing. Following the system preparation, three blank values (empty sample vials) are determined and the mean value of the blank is saved as common variable. This value is subtracted from the EP of the sample determination. Subsequently, the water content of the samples is determined. Between two sample measurements, the titration vessel is conditioned again.

Parameters 899 Coulometer

Conditioning	on
Start drift	10 µg/min
Drift correction	auto
Automatic start	off
Stabilizing time	10 s
Cond. stop time	off
Measured value display	off
Pause	0 s
Request sample ID	off
Request sample size	off
Request sample unit	off
Hold at request	off
Endpoint at	50 mV
Titration rate	optimal
Stop criterion	rel. drift
Relative stop drift	10 µg/min
Extraction time	120 s
Generator electrode	without diaphragm
Generator current	400 mA
Stirrer	on
Stirring rate	15
I(pol)	10 µA
Electrode test	off
Time interval MP	2 s
Temperature	25 °C
Stop time	off

Parameters 885 Compact Oven Sample Changer

Temperature	150 °C
Flow rate	50 mL/min
Gas supply	valve
Gas type	nitrogen
End of series	conditioning

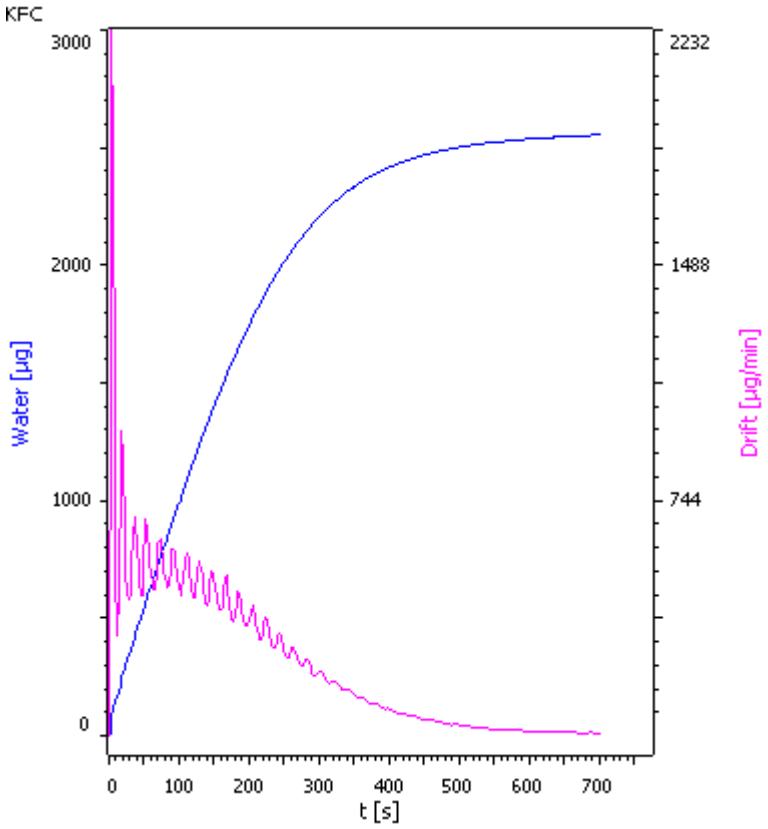
Results

Mean (n = 10) [mg/g]	RSD [%]
113.0	0.67

899库仑水分测定仪联用885全自动卡式炉样品处理器测定塑料小球中水分含量

KF Application Note K–49

Determination of the water content in plastic pellets with 885 Compact Oven Sample Changer and 899 Coulometer



This Application Note describes the determination of the water content in plastic pellets using the oven technique.

Metrohm

Method description

Sample

Plastic pellets (polycarbonate)

Sample preparation

Approximately 3 g of sample were weighed into the sample vial, tightly closed with the cap and placed on the rack of the 885 Compact Oven Sample Changer.

Electrodes

Double Pt-wire electrode	6.0341.100
Generator electrode without diaphragm	6.0345.100

Solutions

HYDRANAL®-Coulomat AG Oven	Fluka 34739

Instrumentation

885 Compact Oven Sample Changer	2.885.0010
899 Coulometer	2.899.0010
Remote cable	6.2141.390

Analysis

All measurements were carried out at the same temperature using the same parameters.

After starting the sample series, the Sample Changer moves to the conditioning vial, the needle pierces the septum, the gas flow is started, and the titration vessel is conditioned. Then a determination with an empty sample vial is carried out to prepare the system and rinse all tubing. Following the system preparation, three blank values (empty sample vials) are determined and the mean value of the blank is saved as common variable. This value is subtracted from the EP of the sample determination. Subsequently, the water content of the samples is determined. Between two sample measurements, the titration vessel is conditioned again.

Parameters 899 Coulometer

Conditioning	on
Start drift	10 µg/min
Drift correction	auto
Automatic start	off
Stabilizing time	10 s
Cond. stop time	off
Measured value display	off
Pause	0 s
Request sample ID	off
Request sample size	off
Request sample unit	off
Hold at request	off
Endpoint at	50 mV
Titration rate	optimal
Stop criterion	rel. drift
Relative stop drift	10 µg/min
Extraction time	120 s
Generator electrode	without diaphragm
Generator current	400 mA
Stirrer	on
Stirring rate	15
I(pol)	10 µA
Electrode test	off
Time interval MP	2 s
Temperature	25 °C
Stop time	off

Parameters 885 Compact Oven Sample Changer

Temperature	250 °C
Flow rate	50 mL/min
Gas supply	valve
Gas type	nitrogen
End of series	conditioning

Results

Mean (n = 10) [µg/g]	RSD [%]
766.7	0.55

采用卡式炉样品处理器相对空白值对水分测定结果的影响

KF Application Note K–48

Sample preparation with the oven technique – relative blank

Large sample sizes can lead to subtraction of too high blank values. This Application Note describes the calculation of a relative blank and thus helps to improve the accuracy of the method.

Metrohm

Relative blank values

Generally, samples with water contents in the low ppm range require large sample sizes. If the sample should be analyzed using the oven technique, large sample sizes can lead to wrong or even negative results. One possible reason for the wrong results can be the subtraction of too high blank values.

Why are the blank values too high?

Blank values are determined using empty sample vials. If for the sample determination, half of the volume (or even more) of the vial is filled with sample, the blank value determined with the empty vial is too high and to obtain correct results, only half of the blank should be subtracted.

For water determinations in large sample sizes, we therefore recommend calculating with relative blank values.

$$\text{blank}_{\text{rel.}} = \text{blank}_{\text{empty vial}} \times \frac{V_{\text{sample}}}{V_{\text{vial}}}$$

$\text{blank}_{\text{rel.}}$	relative blank value
$\text{blank}_{\text{empty vial}}$	blank value of empty sample vial
V_{sample}	volume of sample
V_{vial}	total volume of sample vial

Examples

Volume of vial filled with sample [%]	Total blank value subtracted from EP [%]
50	50
60	40
70	30
80	20

Results

The following results of water determinations in oil illustrate the influence and the importance of the relative blank. Measurements were performed using direct injection and oven technique.

Direct injection

Mean (n = 10) [µg/g]	RSD [%]
19.2	0.45

Oven technique (885 Compact Oven Sample Changer/899 Coulometer)

Mean (n = 10) [µg/g]	RSD [%]
14.0	2.34

Result with subtraction of $\text{blank}_{\text{empty vial}}$

Mean (n = 10) [µg/g]	RSD [%]
20.7	1.45

Result with subtraction of $\text{blank}_{\text{rel.}}$

Oven technique (874 Oven Sample Processor/851 Titrando)

Mean (n = 10) [µg/g]	RSD [%]
14.7	1.91

Result with subtraction of $\text{blank}_{\text{empty vial}}$

Mean (n = 10) [µg/g]	RSD [%]
20.7	1.45

Result with subtraction of $\text{blank}_{\text{rel.}}$

Conclusion

Water determinations obtained by the oven technique show a too low water content when the blank value of the empty vial is subtracted. In contrast, the subtraction of the relative blank value leads to results that agree with those using direct injection.