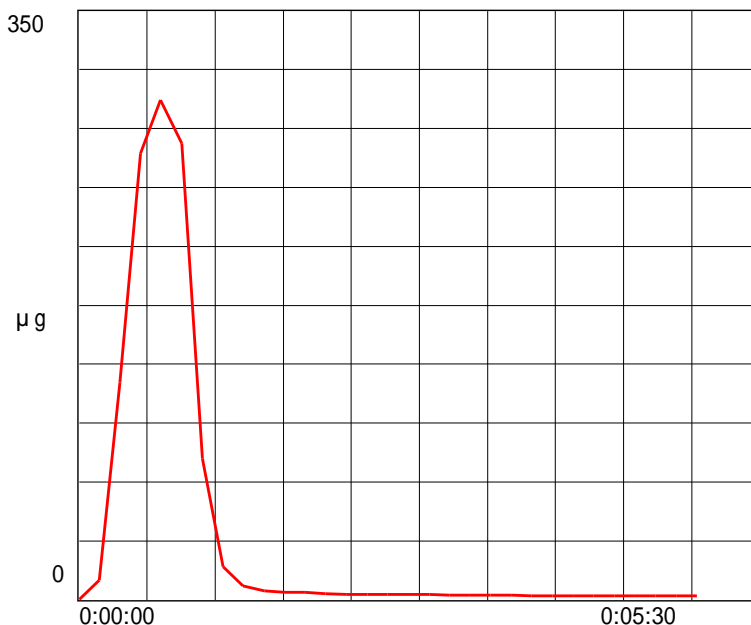


<< Recalculation result >>

| | | | |
|----------------------------|-----------------------|------------------------|-----------------------|
| Sample No. | : 02-01 | Date | : 2016/07/29 12:41:27 |
| Model | : MKC-710(MCU-710) | Time | : 00:05:00 |
| Serial No. | : 20260399 | | |
| Operator | : | | |
| | | | |
| Method name | : Evaporation(Sample) | | |
| Anolyte Reagent Name | : Anolyte | Anolyte Life Value | : 43 mg |
| Catholyte Reagent Name | : Catholyte | Catholyte Life Value | : 43 mg |
| Not Drift Stop | : -.- | Control gain | : 5.0 |
| t(stir) | : 0 s | t(wait) | : 15 s |
| t(max) | : 300 s | Cell type | : 2-Comp. |
| Option | : ADP- | Oven temp. | : 170 Deg.C |
| Pre-Treat | : 3 | Back purge | : 180 s |
| Cell purge | : 0 s | Sample purge | : ----- s |
| Calc.No. | : 2 | | |
| Sample name | : | | |
| Sample ID | : | Size(Size) * | : 0.1116 g |
| Size(Wt1) * | : 0.1116 g | Size(Wt2) * | : 0.0000 g |
| Blank (Wt1) | : 26.5000 µg | Dilution Coef. (FA) | : 1.00000 |
| Dissolve Samp. (B) | : ----- g | Conc.of Solvent (V1) | : ----- ppm |
| Dissolve Solvent (A) | : ----- g | | |
| Samp. Volume (Dens) | : ----- mL | Samp.Dens. (V2) | : ----- g/mL |
| Samp. Gas Volume (Temp.) | : ----- L | Samp. Gas Temp. (K) | : --- Deg.C |
| Recalculation comment | : | | |
| | | | |
| [Result] | | | |
| Concentration | : 0.9859 % | | |
| Moisture | : 1100.3 µg | Drift | : 0.05 µg/s |
| Detection temp. | : --- Deg.C | | |

[Titration Curve]



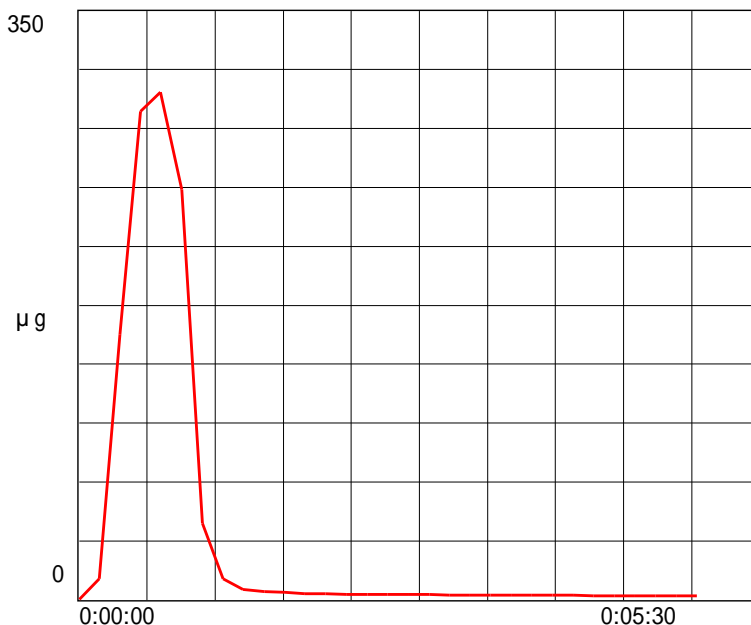
[Data List]

| No. | Time | Unit moisture(ug) | Total moisture(ug) | Temp.(C) |
|-----|----------|-------------------|--------------------|----------|
| 1 | 00:00:10 | 11.2 | 11.2 | --- |
| 2 | 00:00:20 | 128.8 | 140.0 | --- |
| 3 | 00:00:30 | 265.4 | 405.4 | --- |
| 4 | 00:00:40 | 296.7 | 702.1 | --- |
| 5 | 00:00:50 | 271.1 | 973.2 | --- |
| 6 | 00:01:00 | 83.4 | 1056.6 | --- |
| 7 | 00:01:10 | 19.3 | 1075.9 | --- |
| 8 | 00:01:20 | 7.7 | 1083.6 | --- |
| 9 | 00:01:30 | 4.8 | 1088.4 | --- |
| 10 | 00:01:40 | 4.0 | 1092.4 | --- |
| 11 | 00:01:50 | 3.6 | 1096.0 | --- |
| 12 | 00:02:00 | 3.4 | 1099.4 | --- |
| 13 | 00:02:10 | 3.0 | 1102.4 | --- |
| 14 | 00:02:20 | 3.1 | 1105.5 | --- |
| 15 | 00:02:30 | 2.8 | 1108.3 | --- |
| 16 | 00:02:40 | 2.8 | 1111.1 | --- |
| 17 | 00:02:50 | 2.7 | 1113.8 | --- |
| 18 | 00:03:00 | 2.5 | 1116.3 | --- |
| 19 | 00:03:10 | 2.4 | 1118.7 | --- |
| 20 | 00:03:20 | 2.4 | 1121.1 | --- |
| 21 | 00:03:30 | 2.4 | 1123.5 | --- |
| 22 | 00:03:40 | 2.1 | 1125.6 | --- |
| 23 | 00:03:50 | 2.2 | 1127.8 | --- |
| 24 | 00:04:00 | 2.2 | 1130.0 | --- |
| 25 | 00:04:10 | 2.0 | 1132.0 | --- |
| 26 | 00:04:20 | 2.1 | 1134.1 | --- |
| 27 | 00:04:30 | 1.9 | 1136.0 | --- |
| 28 | 00:04:40 | 1.9 | 1137.9 | --- |
| 29 | 00:04:50 | 2.0 | 1139.9 | --- |
| 30 | 00:05:00 | 1.9 | 1141.8 | --- |

<< Recalculation result >>

| | | | |
|----------------------------|-----------------------|------------------------|-----------------------|
| Sample No. | : 02-02 | Date | : 2016/07/29 12:53:31 |
| Model | : MKC-710(MCU-710) | Time | : 00:05:00 |
| Serial No. | : 20260399 | | |
| Operator | : | | |
| | | | |
| Method name | : Evaporation(Sample) | | |
| Anolyte Reagent Name | : Anolyte | Anolyte Life Value | : 44 mg |
| Catholyte Reagent Name | : Catholyte | Catholyte Life Value | : 44 mg |
| Not Drift Stop | : -.- | Control gain | : 5.0 |
| t(stir) | : 0 s | t(wait) | : 15 s |
| t(max) | : 300 s | Cell type | : 2-Comp. |
| Option | : ADP- | Oven temp. | : 170 Deg.C |
| Pre-Treat | : 3 | Back purge | : 180 s |
| Cell purge | : 0 s | Sample purge | : ----- s |
| Calc.No. | : 2 | | |
| Sample name | : | | |
| Sample ID | : | Size(Size) * | : 0.1092 g |
| Size(Wt1) * | : 0.1092 g | Size(Wt2) * | : 0.0000 g |
| Blank (Wt1) | : 26.5000 μ g | Dilution Coef. (FA) | : 1.00000 |
| Dissolve Samp. (B) | : ----- g | Conc.of Solvent (V1) | : ----- ppm |
| Dissolve Solvent (A) | : ----- g | | |
| Samp. Volume (Dens) | : ----- mL | Samp.Dens. (V2) | : ----- g/mL |
| Samp. Gas Volume (Temp.) | : ----- L | Samp. Gas Temp. (K) | : --- Deg.C |
| Recalculation comment | : | | |
| | | | |
| [Result] | | | |
| Concentration | : 0.9923 % | | |
| Moisture | : 1083.6 μ g | Drift | : 0.05 μ g/s |
| Detection temp. | : --- Deg.C | | |

[Titration Curve]



[Data List]

| No. | Time | Unit moisture(ug) | Total moisture(ug) | Temp.(C) |
|-----|----------|-------------------|--------------------|----------|
| 1 | 00:00:10 | 11.9 | 11.9 | --- |
| 2 | 00:00:20 | 157.1 | 169.0 | --- |
| 3 | 00:00:30 | 290.1 | 459.1 | --- |
| 4 | 00:00:40 | 301.5 | 760.6 | --- |
| 5 | 00:00:50 | 243.4 | 1004.0 | --- |
| 6 | 00:01:00 | 45.3 | 1049.3 | --- |
| 7 | 00:01:10 | 11.8 | 1061.1 | --- |
| 8 | 00:01:20 | 5.6 | 1066.7 | --- |
| 9 | 00:01:30 | 4.3 | 1071.0 | --- |
| 10 | 00:01:40 | 3.6 | 1074.6 | --- |
| 11 | 00:01:50 | 3.4 | 1078.0 | --- |
| 12 | 00:02:00 | 3.3 | 1081.3 | --- |
| 13 | 00:02:10 | 3.0 | 1084.3 | --- |
| 14 | 00:02:20 | 2.9 | 1087.2 | --- |
| 15 | 00:02:30 | 2.8 | 1090.0 | --- |
| 16 | 00:02:40 | 2.8 | 1092.8 | --- |
| 17 | 00:02:50 | 2.7 | 1095.5 | --- |
| 18 | 00:03:00 | 2.6 | 1098.1 | --- |
| 19 | 00:03:10 | 2.5 | 1100.6 | --- |
| 20 | 00:03:20 | 2.5 | 1103.1 | --- |
| 21 | 00:03:30 | 2.4 | 1105.5 | --- |
| 22 | 00:03:40 | 2.3 | 1107.8 | --- |
| 23 | 00:03:50 | 2.3 | 1110.1 | --- |
| 24 | 00:04:00 | 2.3 | 1112.4 | --- |
| 25 | 00:04:10 | 2.2 | 1114.6 | --- |
| 26 | 00:04:20 | 2.1 | 1116.7 | --- |
| 27 | 00:04:30 | 2.2 | 1118.9 | --- |
| 28 | 00:04:40 | 2.1 | 1121.0 | --- |
| 29 | 00:04:50 | 2.1 | 1123.1 | --- |
| 30 | 00:05:00 | 2.0 | 1125.1 | --- |

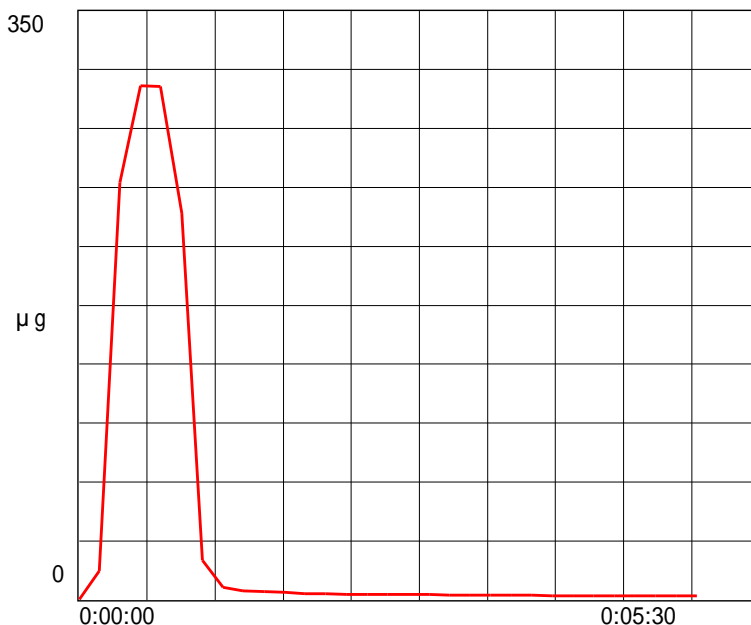
<< Titration result >>

Sample No. : 02-03 Date : 2016/07/29 13:06:38
 Model : MKC-710(MCU-710) Time : 00:05:00
 Serial No. : 20260399
 Operator :

Method name : Evaporation(Sample)
 Analyte Reagent Name : Analyte Analyte Life Value : 46 mg
 Catholyte Reagent Name : Catholyte Catholyte Life Value : 46 mg
 Not Drift Stop : --- Control gain : 5.0
 t(stir) : 0 s t(wait) : 15 s
 t(max) : 300 s Cell type : 2-Comp.
 Option : ADP- Oven temp. : 170 Deg.C
 Pre-Treat : 3 Back purge : 180 s
 Cell purge : 0 s Sample purge : ----- s
 Calc.No. : 2
 Sample name :
 Sample ID :
 Size(Wt1) * : 0.1171 g Size(Size) * : 0.1171 g
 Blank (Wt1) : 26.5000 µg Size(Wt2) * : 0.0000 g
 Dissolve Samp. (B) : ----- g Dilution Coef. (FA) : 1.00000
 Dissolve Solvent (A) : ----- g Conc.of Solvent (V1) : ----- ppm
 Samp. Volume (Dens) : ----- mL Samp.Dens. (V2) : ----- g/mL
 Samp. Gas Volume (Temp.) : ----- L Samp. Gas Temp. (K) : ----- Deg.C
 Recalculation comment : 1

[Result]
 Concentration : 0.9873 %
 Moisture : 1156.1 µg Drift : 0.05 µg/s
 Detection temp. : --- Deg.C

[Titration Curve]



[Data List]

| No. | Time | Unit moisture(ug) | Total moisture(ug) | Temp.(C) |
|-----|----------|-------------------|--------------------|----------|
| 1 | 00:00:10 | 17.0 | 17.0 | --- |
| 2 | 00:00:20 | 247.8 | 264.8 | --- |
| 3 | 00:00:30 | 305.2 | 570.0 | --- |
| 4 | 00:00:40 | 305.0 | 875.0 | --- |
| 5 | 00:00:50 | 229.7 | 1104.7 | --- |
| 6 | 00:01:00 | 23.3 | 1128.0 | --- |
| 7 | 00:01:10 | 6.9 | 1134.9 | --- |
| 8 | 00:01:20 | 4.9 | 1139.8 | --- |
| 9 | 00:01:30 | 4.1 | 1143.9 | --- |
| 10 | 00:01:40 | 3.7 | 1147.6 | --- |
| 11 | 00:01:50 | 3.4 | 1151.0 | --- |
| 12 | 00:02:00 | 3.2 | 1154.2 | --- |
| 13 | 00:02:10 | 3.1 | 1157.3 | --- |
| 14 | 00:02:20 | 3.0 | 1160.3 | --- |
| 15 | 00:02:30 | 2.9 | 1163.2 | --- |
| 16 | 00:02:40 | 2.8 | 1166.0 | --- |
| 17 | 00:02:50 | 2.7 | 1168.7 | --- |
| 18 | 00:03:00 | 2.6 | 1171.3 | --- |
| 19 | 00:03:10 | 2.5 | 1173.8 | --- |
| 20 | 00:03:20 | 2.5 | 1176.3 | --- |
| 21 | 00:03:30 | 2.3 | 1178.6 | --- |
| 22 | 00:03:40 | 2.4 | 1181.0 | --- |
| 23 | 00:03:50 | 2.2 | 1183.2 | --- |
| 24 | 00:04:00 | 2.2 | 1185.4 | --- |
| 25 | 00:04:10 | 2.1 | 1187.5 | --- |
| 26 | 00:04:20 | 2.1 | 1189.6 | --- |
| 27 | 00:04:30 | 2.1 | 1191.7 | --- |
| 28 | 00:04:40 | 1.9 | 1193.6 | --- |
| 29 | 00:04:50 | 2.0 | 1195.6 | --- |
| 30 | 00:05:00 | 2.0 | 1197.6 | --- |



Certificate of Analysis

Apura[®] Certified Reference Material

| | |
|---|--|
| Producer: | Merck KGaA, Frankfurter Str. 250, 64293 Darmstadt, Germany. |
| Accreditation: | Merck KGaA, Darmstadt, Germany is accredited as calibration laboratory according to DIN EN ISO/IEC 17025. |
| Description of CRM: | Water Standard Oven certified reference material for coulometric Karl Fischer Titration in combination with the Karl Fischer Oven technique |
| Ord. No.: | 1.88054.0005 |
| Lot No.: | FN1299854 |
| Composition: | potassium sulfate / sodium wolframate dihydrate |
| Certified value and uncertainty: | 0.99% ± 0.02 % (9.9 mg/g ± 0.2 mg/g) water content with expanded uncertainty U_{CRM} |
| Traceability: | The certified value of this reference material is directly traceable to SI-Unit (kg). |
| Method of Analysis: | The water content is determined by coulometric Karl Fischer oven method at 170°C and an extraction time of 600s on 10 samples according to ISO 760. The measurement value is additionally checked by thermogravimetry as independent procedure. |
| Storage: | +15°C to +25°C tightly closed in the original container |
| Application and correct use: | This certified reference material is intended for use as a standard for checking the accuracy of coulometric and volumetric water determinations using a Karl Fischer oven and thermobalances according to ISO 9001 chapter 7.1.5 "Monitoring and measuring resources". For the daily verification we recommend to accept a deviation of ± 0.03% (± 0.3 mg/g) from the certified value. |
| Date of release: | 2016/03/01 |
| Minimum shelf life: | 2019/02/28 |

A. Yildirim

Dipl.-Ing. Ayfer Yildirim
(Laboratory manager)

Expanded uncertainty U_{CRM} :

The expanded uncertainty U_{CRM} is calculated as $U_{CRM}=k \cdot u_{CRM}$, where $k=2$ is the coverage factor for a 95% coverage probability and u_{CRM} is the combined standard uncertainty in accordance to ISO Guide 34.

$$u_{CRM} = \sqrt{u^2_{\text{Characterisation}} + u^2_{\text{Homogeneity}} + u^2_{\text{Stability}}}$$

The combined standard uncertainty u_{CRM} is obtained from the standard uncertainties of the characterisation, the homogeneity and the stability.

$u_{\text{Characterisation}}$ is the uncertainty in accordance with DIN EN ISO/IEC 17025 which includes the contributions of the primary reference material and the measuring system.

$u_{\text{Characterisation}}$ in the certified value is calculated in accordance to EA-4/02 and GUM.

$u_{\text{Characterisation}}$ is 0.01% (0.1 mg/g) (calculated as $u_{\text{Characterisation}} = k \cdot u_{\text{Characterisation}}$ with $k=2$)

$u_{\text{Homogeneity}}$ is the between-bottle variation in accordance with ISO Guide 34. The assessment of homogeneity is performed by analysis of a representative number of systematically chosen sample units.

$u_{\text{Stability}}$ is the uncertainty obtained from short-term and long-term stability in accordance with ISO Guide 34. The stability studies are the basis for the quantification of the minimum shelf life of this water standard for the unopened ampoule.

Instructions for correct use

- The oven standard can be used in a temperature range of 150°C and 400°C.
- A minimum heating time (extraction time) of 300 sec is recommended.
- The weigh-in quantity for the KF oven technique is usually in the range of 80 mg to 300 mg.
- Setting parameters for the oven like minimum temperature and heating time as well as optimum sample size of the standard substance could vary depending on the used instrument.