Honeywell Specialty Chemicals Seelze GmbH, Wunstorferstrasse 40, 30926, Seelze, Germany
Tel.: +49 (0)5137-999-353 Fax: +49 (0)5137-999-698 hydranal@honeywell.com

HYDRANAL™ Technical Information Sheet T003 Rev. 5

Qualification of a Karl Fischer Oven System with Coulometric Cell

Qualification of a KF oven system in conjunction with a coulometer is always a combination of two parameters. On the one hand it needs to be confirmed that all water which is transferred to the titration cell is detected quantitatively. On the other hand, it needs to be guaranteed that all water which is evaporated from the sample is reaching the titration cell completely. We therefore recommend a two-step control concept to ensure the reliability of both the separate titration device and the whole system.

Step 1: Reliability of detected water content

Recovery rate of water added directly to the titration cell

A water standard with a known amount of water is injected directly into the titration cell. The desired absolute amount of water can be chosen by using the respective standard. By using different portion size, a linearity can be tested, or the portion can be adjusted to common amount of water in µg corresponding to the absolute water of routinely analyzed samples.

34828 HYDRANAL-Water Standard 1.0 (water content approx. 1000 µg/g)
34426 HYDRANAL-CRM Water Standard 1.0 (water content approx. 1000 µg/g)
34446 HYDRANAL-Water Standard 0.1 PC (water content approx. 100 µg/g)
34847 HYDRANAL-Water Standard 0.1 (water content approx. 100 µg/g)

Recommendation for recovery (from literature, e.g. Ph. Eur.):
1000 µg ± 25 µg (± 2.5%) or 100 µg ± 10 µg (± 10%)

Step 2: Tightness of the oven system

Recovery rate of water added indirectly via evaporation in the oven

The water amount used for this step does not have to correspond to the water amount in the routinely analyzed samples. We recommend using rather higher amounts of water in order to detect possible leakage in the system. For example, if a tubing system shows a 10% leakage, by using 100 µg of water, only 10 µg would be missing, what may not be recognized due to general uncertainty or detection limits. By using 5000 µg absolute water, the missing 10% would be 500 µg, which is much easier to recognize.
Two standards are available for use at different temperature. Both materials contain water in a range of around 5%. A sample size of about 100 mg corresponds to the above recommended 5000 µg water.

34693 HYDRANAL-Water Standard KF-Oven 150-160°C (water content approx. 5.0%, based on lactose hydrate)
34748 HYDRANAL-Water Standard KF-Oven 220-230°C (water content approx. 5.55%, based on potassium citrate hydrate)

Our recommendation for recovery: 5000 µg ± 200 µg (± 4%)

Common practice is to use 34693 standard if your routinely analyzed samples are heated at temperature below 200°C and 34748 standard if your routinely analyzed samples are heated at temperature above 200°C. However, both standards can be used for testing tightness of the KF oven system no matter which temperature is applied for heating the sample.

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NOTE:
Both solid standards need to be used at the temperature from the range stated in the product name. Don't apply lower or higher temperature as it will cause incorrect results. Applying lower temperature will cause that not all water will be released from the standard and result will be lower than the value stated on Report of Analysis. Applying higher temperature will cause decomposition of the standard and result will be too high.

The chosen temperature for the check with the oven standards is not applicable for heating the sample. Also, it cannot be assumed that sample releases its water fast and completely at 100°C (boiling point of water). Independently from the tightness check, an individual suitable heating temperature needs to be determined in advance for every sample via temperature ramp (gradient) or test runs at different temperature values.

General recommendations for KF oven use:

- The KF oven is often used in conjunction with the coulometric determination in combination with a cell without diaphragm. The anodic compartment is filled with about 150 mL 34739 HYDRANAL-Coulomat AG-Oven. A cell with diaphragm additionally requires 34840 HYDRANAL-Coulomat CG in the cathode compartment.
- The device is switched on, it titrates automatically dry. Upon reaching a low and stable drift (<10 µg H_2O/min), the carrier gas is switched on.
- A gas flow of 70-80 mL/min is recommended. 34241 HYDRANAL-Molecular sieve 0.3 nm is well-suited as a drying agent for the carrier gas. If two drying towers are available, the first can be alternatively filled with 34788 HYDRANAL-Humidity absorber.
- Before the first sample measurement, a lead time of approx. 30-60 min is recommended to get again a stable drift with the carrier gas. A cell in good conditions allows a drift level of 3-10 µg H_2O/min.
- To determine the blank value in triplicate, three empty injection vials are heated at the chosen temperature with a fixed minimum titration time of 300 s.
- Blank and sample vials are recommended to be prepared at the same time under same room conditions.
- 50-100 mg of the chosen oven standard are weighed accurately by differential weighing and heated at the recommended temperature (see also instruction for use added to the individual standards). A relative stop drift of 2-5 µg/min is suggested as stop criterion (for Mettler Toledo ovens as stop criterion a delay time of 3-5 s is recommended). For the determination of standards or sample material no maximum time limits should be set in the method. Time limits can lead to an artificial stop of the determination resulting in reduced recoveries.
- The drift value should be stable and low between each measurement. It should be comparable to the original drift value that was observed while preparation with carrier gas. An additional conditioning mode of 5-15 minutes between each sample can help to stabilize the whole system. Make sure that the automatic drift correction is turned on.
- To compensate evaporated solvent it is recommended to refill the cell to its original filling level after each work day by injection of 34741 HYDRANAL-Methanol dry.
- The catholyte is recommended to be replaced on a weekly basis. The anolyte provides a much higher water capacity and can be typically used for more than one week. Maintained reliability of the cell should be frequently monitored.
- Additionally, follow the general working procedure of the instructions for use provided by your device supplier.

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