Application Note AN-K-072

34433 HYDRANAL™ NEXTGEN Coulomat AG-FI

Test measurements using imidazole-free reagent for coulometric Karl Fischer titration

The reaction constant of the Karl Fischer reaction depends on the pH value. Therefore, Karl Fischer reagents contain buffer substances to ensure a stable pH and thus a stable reaction. Imidazole is widely used as a buffer in KF reagents.

In 2012, the European Union selected imidazole for substance evaluation in order to clarify whether it constitutes a risk to human health or to the environment. In 2015, imidazole was classified as a CMR substance and the statement H360D (may damage the unborn child) was added.

Meanwhile, other reagents free of imidazole are available for purchase. This Application Note summarizes test measurements with 34433 HYDRANALTM NEXTGEN Coulomat AG-FI.

SAMPLE AND SAMPLE PREPARATION

Three different water standards were used:

1. 34828 HYDRANALTM Water Standard 1.0
2. 34446 HYDRANALTM Water Standard 0.1 PC
3. 34748 HYDRANALTM Water Standard KF-Oven 220–230 °C

The liquid water standards were aspirated into a syringe and injected directly into the titration cell. The oven standard was poured into a sample vial and closed with a septum cap.

EXPERIMENTAL

A series of measurements (n = 6) were carried out with the two liquid standards (**1** and **2**; various sample sizes between 0.5–2.9 g) using a generator electrode without diaphragm. The measurements were repeated using a generator electrode with diaphragm. The cathode compartment was filled with 5 mL of 34840 HYDRANALTM Coulomat CG. Additionally, a 6-fold determination with the oven standard (**3**; various sample sizes between 50–70 mg) was carried out at an oven temperature of 230 °C.

RESULTS

The following three tables list the results of the measurements.

Table 1. Results of the measurements (n = 6) using a generator electrode without diaphragm.

Standard	Recovery	s(abs)	s(rel)
1 (1000 ppm)	100.1%	0.056%	0.06%
2 (100 ppm)	104.9%	0.654%	0.62%

Table 2. Results of the measurements (n = 6) using a generator electrode with diaphragm.

Standard	Recovery	s(abs)	s(rel)
1 (1000 ppm)	100.9%	0.298%	0.30%
2 (100 ppm)	104.1%	1.446%	1.41%

Table 3. Results of the measurements (n = 6) using a generator electrode with diaphragm.

Standard	Recovery	s(abs)	s(rel)
3 (5.55%)	99.29%	0.325%	0.33%

CONCLUSION

The results show that using the imidazole-free reagent, accurate and reproducible results are obtained.
The recoveries are within the expected range of 97–103% (1000 ppm and percent water; standards 1 and 3) and 90 to 110% (100 ppm water; standard 2).

Due to the lower water content of the 100 ppm standard 2, the relative and absolute standard deviations are higher compared to the 1000 ppm standard 1, but in an acceptable range.
There is no need to adapt the method parameters. The default parameters can be used.

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CONFIGURATION

OMNIS Titrator KF

The OMNIS Titrator KF offers you the complete package for volumetric Karl Fischer titration. Included in the package is the OMNIS Basic Titrator with magnetic stirrer for potentiometric end point titration, the function license for KFT with conditioning, the OMNIS Solvent Module and the complete accessories for volumetric Karl Fischer titration.

Benefit from the unique user-friendliness of an automatic start of titration after sample addition and maximum safety thanks to contact-free reagent handling with the 3S-Liquid Adapter and OMNIS Solvent Module.

OMNIS

A WHOLE NEW LEVEL OF PERFORMANCE

OMNIS Stand-Alone license

Enables stand-alone operation of the OMNIS software on a WindowsTM computer.

Features:

- The license already includes one OMNIS instrument license.
- Must be activated via the Metrohm licensing portal.