



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 HYDRANAL™ NEXTGEN Coulomat AG-FI.

SAMPLE AND SAMPLE PREPARATION

Three different water standards were used:

1. 34828 HYDRANAL™ Water Standard 1.0
2. 34446 HYDRANAL™ Water Standard 0.1 PC
3. 34748 HYDRANAL™ 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 HYDRANAL™ 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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