

Application Note AN-K-070

Water in petroleum products

Fully automated determination according to ASTM D6304

Moisture in petroleum products causes several issues: corrosion and wear in pipelines and storage tanks, an increase in debris load resulting in diminished lubrication, blocked filters, or even harmful bacterial growth. As a result, increased water content can lead to infrastructure damage, higher maintenance costs, or even unwanted downtimes. Due to these costly factors, water content is a critical factor regulated in many trade specifications, and also defines the price of these products. An accurate and reliable determination is therefore necessary, since even small deviations can have a large impact on the selling price.

As petroleum products contain low moisture content, coulometric Karl Fischer titration is the method of choice. Using a Karl Fischer oven to vaporize the water present in the sample prior to titration not only greatly reduces matrix interferences, it can also be fully automated. This allows a reliable and cost efficient analysis of the water content according to ASTM D6304 (Procedure B) in products such as diesel, hydraulic oil, lubricant, additive, turbine oil, and base oil.

SAMPLE AND SAMPLE PREPARATION

To demonstrate the versatile use of the moisture determination in petroleum products with the Karl Fischer oven, the results from various samples such as diesel, hydraulic oil, lubricant, additive, turbine oil, and base oil are shown in this application note.

All samples are completely homogenized before sampling. After homogenization, the sample is weighed directly into the sample vial. Sample size is dependent on the expected amount of water. The sample vials are sealed airtight and placed on the sample rack.

EXPERIMENTAL

Prior to starting the sample determinations, the titration cell is conditioned and the oven is heated to the desired temperature. The ideal oven temperature releases all water quickly but does not lead to sample decomposition, which would falsify the result.

Once the system is prepared and stable, the sample is placed in the oven. A carrier gas passes through the sample, transferring the vaporized water into the titration cell where the water content is determined.

The titration and the gas extraction of the sample is stopped as soon as the defined endpoint is reached and the drift (amount of water per time period) falls below a predefined value.



Figure 1. Fully automated system consisting of an 874 Oven Sample Processor with 851 Titrando for the coulometric Karl Fischer after vaporization of any moisture present in the sample.

RESULTS

For all samples, acceptable standard deviations are achieved using the oven accessory, regardless of whether the sample had a low or high water content or contains interfering additives. The results for the various samples are given in Table 1.

Table 1. Results of the water determination in various petroleum products after vaporization of the moisture within the sample according to ASTM D6304.

Sample (n = 4)	Mean / (mg/L)	SD(abs) / (mg/L)	SD(rel) / (%)
Diesel	27.8	1.7	6.08
Hydraulic oil	44.6	0.7	1.57
Lubricant	22.9	1.1	4.63
Additive	2830.7	6.2	0.22
Turbine oil	18.9	2.5	13.39
Base oil	17.0	0.6	3.41

CONCLUSION

With the 874 Oven Sample Processor and 851 Titrando, a fully automated determination of water in petroleum products is possible according to ASTM D6304 Procedure B. Furthermore, to prevent inaccurate results caused by sample decomposition at higher temperatures, it is possible to determine the optimal oven temperature for each sample with the 874 Oven Sample Processor. Thus, using the water evaporation technique the moisture content can be determined reliably without matrix interferences at the optimal temperature.

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CONFIGURATION



851 Titrando with generator electrode with diaphragm

Coulometer including generator electrode with diaphragm and 801 Magnetic Stirrer.

Coulometry is the ideal method for water content determination in liquids, solids, and gases when it comes to water content determination in the trace range (10 µg to 10 mg absolute water). In addition, coulometry is an absolute method and thus no titer determination is necessary.

Coulometric titrations are carried out easily and quickly with the 851 Titrando.

Recommended measuring range: 10 µg - 200 mg absolute water

For use with OMNIS Software, tiamo software, or Touch Control unit. Compliance with GMP/GLP and FDA regulations such as 21 CFR Part 11, if required



874 Oven Sample Processor

The 874 Oven Sample Processor is used for automatic thermal sample preparation in Karl Fischer titration. The oven method is particularly suitable for samples that do not release their water until higher temperatures have been reached, for sparingly soluble samples, or those that react with the KF reagent.

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