



Application Note AN-K-074

Water content in crude oil determined with Karl Fischer titration

Fully automated determination using the oven method according to ASTM D4928

Crude oil contains water. Before transport, water is removed to reduce costs. Furthermore, the presence of water in crude can cause corrosion. Therefore, it is essential to determine the water content in crude oil. Previously, ASTM D4928 described a direct coulometric Karl Fischer titration to determine water in crude oil. This quickly contaminated the titration cell, requiring regular cleaning and frequent reagent exchange. ASTM D4928 was then revised to include

coulometric Karl Fischer titration in combination with the oven method. In this method, the sample is heated in an oven. The water evaporates and is carried into the titration cell by an inert carrier gas. The water content is determined in the titration cell. The oven method avoids titration cell contamination and significantly reduces reagent consumption. It can be fully automated, minimizing handling errors and workloads, with outstanding reproducibility.

SAMPLE AND SAMPLE PREPARATION

The method is demonstrated for three different crude oil types. The samples are homogenized before being

filled into sample vials.

EXPERIMENTAL

This analysis is carried out on an automated system consisting of an 874 Oven Sample Processor and an 851 Titrando equipped with a coulometric titration cell (Figure 1).



Figure 1. The 874 Oven Sample Processor, 851 Titrando and coulometric titration cell, all controlled by tiamo software.

RESULTS

The analysis demonstrates acceptable results and well-defined titration curves. The results for the three different crude oil samples are shown in Table 1. An example titration curve is displayed in Figure 2.



Figure 2. Example titration curve of the water content determination in crude oil.

Table 1. Results for the water content determination in crude oil according to ASTM D4928.

Water content (n = 4)	Mean in $\mu\text{g H}_2\text{O/g sample}$	SD(rel) in %
Sample 1	853	2.09
Sample 2	4865	0.44
Sample 3	41111	0.43

The oven method is the perfect option to determine water content in crude oils precisely and reliably. Using the 874 Oven Sample Processor allows fully automated determination, freeing up valuable time

and thus increasing laboratory productivity. Furthermore, by fully automating the analysis, the reproducibility can be increased and sample analysis failures due to improper handling can be reduced.

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CONFIGURATION



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.



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