



## HYDRANAL<sup>®</sup>-Coulomat Oil

### **A reagent for the coulometric water determination in oils .....**

Oils are used in many different technical applications. In practice, oils have to pass extensive analytical tests to meet continuously increasing quality requirements. Some of the more stringent requirements call for tests on samples containing less than 10 ppm water. HYDRANAL-Coulomat Oil is very well suited for the analysis of oil samples.

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For the determination of water contents in the low ppm range, coulometry should be the method of choice. Besides its high precision, this method has distinguished itself due to the ease of introduction of liquid samples with a syringe. With the publication of the ASTM method, D 4928-96 „Standard test methods for water in crude oils by coulometric“ the coulometric determination of water in crude oils was established as an accepted procedure.

### Applications

A few applications for the determination of low water content in oils can be mentioned here. In transformer oils the water content is decisive for determining the service life of the oil and, within the framework of process controls, lubricating oils have to be tested on a regular basis. The water content of cosmetic products is as decisive for their shelf life as it is for cooking oils in the food industry. The water content also has a major influence on the raw material costs - especially when dealing with crude oils.

### Development of the reagent

For a typical Karl Fischer titration, the preferred media is an alcohol, in which, however, due to the differences in polarity, non-polar ingredients of oil are insoluble. The reagent is based on methanol, to which defined quantities of aromatic and halogenated hydrocarbons have been added as solubilizers to increase its solubilizing properties. Also in HYDRANAL-Coulomat Oil, samples of oil in suspension may be carried out. Nevertheless, the determination of water contents can be performed without any difficulties. The addition of xylene results in a higher solubility of especially tar, relevant amounts of which are contained in crude oils. Thereby the formation of tar layers on the indicator electrode can be inhibited and no disturbances are to be anticipated for the identification of the end points. This optimally aligned formulation meets all requirements of customary coulometers with regard to the conductivity of the reagents used. This new reagent is intended for use as an anolyte for coulometric cells with diaphragm in combination with the catholyte HYDRANAL-Coulomat CG.

### Validation of the reagent

Our HYDRANAL-Laboratory performed an extensive validation of this new reagent using coulometers of various manufacturers. In all of the validations, a 5 mL charge of HYDRANAL-Coulomat CG was used as the catholyte, and 100 mL of HYDRANAL-Coulomat Oil was filled in the anode compartment.

For various oils the water contents were determined in samples of 2 mL each (n = 10). Furthermore, the rate of recovery (n=3), as well as the reproducibility, was determined with HYDRANAL-Water Standard 1.00. This standard is tested against the reference material

Cat. No.	Product Name	Description	Pack Size
34868	HYDRANAL-Coulomat Oil	Anolyte for titration of oils	100 mL; 500 mL
34840	HYDRANAL-Coulomat CG	Catholyte, free of halogenated hydrocarbons, 50 mL contains 10 x 5 mL ampoules	50 mL
34847	HYDRANAL-Water Standard 0.1	Standard for coulometric KF titration, 1 g contains 1.00 mg = 0.10 % water, contains 10 glass ampoules of 4 mL. Tested against NIST SRM 2890	40 mL
34828	HYDRANAL-Water Standard 1.00	Standard for coulometric KF titration, 1 g contains 0.10 mg = 0.01 % water, contains 10 glass ampoules of 4 mL. Tested against NIST SRM 2890	40 mL

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NIST SRM 2890. A sample of the validation results appears in **Table 1** below.

Due to the high absorption capacity of HYDRANAL-Coulomat Oil the determination of water in many samples, even with high water contents, can be performed with exceptional reproducibility. Regardless of the oil samples to be tested, rates of recovery exceeding 99 % with a simultaneously very high reproducibility can be determined.

### Disturbances

HYDRANAL-Coulomat Oil has been formulated with the optimum blend of solubilizers with regard to the majority of oil samples. However, there may be that one unique sample where extra chloroform is desired. We have found that an additional 10 % chloroform, by volume, may be added without affecting the results. Oils blended with additives may experience side reactions that simulate water contents, which are too high. These samples have to be tested according to the indirect method with the KF oven. For this procedure HYDRANAL-Coulomat E or HYDRANAL-Coulomat AG Oven is especially suitable as anolyte.

When using coulometric cells without a diaphragm, the quantities of solubilizers contained in the reagent lead to side reactions. The thus determined results will exceed the real value by 5-10 %.

### Technical support

We'll be glad to provide you with support in the analysis of your sample based on our twenty years of experience with Karl Fischer titration. We can suggest a solution to your analytical problem and, if necessary, develop an individual analytical method for you. Our comprehensive application collection makes daily work easier for HYDRANAL users, and is always at your disposal anytime.

Just call us! We'll be pleased to answer any questions regarding sample analysis using the Karl Fischer method, and on HYDRANAL-reagents.

Sample type	Water Content		Rate of recovery	
	mg/kg	RSD/%	Rate/%	RSD/%
Fuel unleaded	73,3	2,2	99,3	0,8
Oil Baysilon M 50 EL	79,6	2,3	99,8	0,4
Crude oil BCF	225	5,5	99,3	2,4
Crude oil FRT	109	2,8	99,5	3,0
Silicon oil AK 20	75,0	3,4	99,6	1,4
Silicon oil TR 50	76,5	2,5	99,7	0,5
Salad oil	300	2,1	99,9	0,9
Transformer oil	140	3,1	99,0	2,5
Transformer oil Teresso 46	27,5	0,7	99,7	1,2

**Table 1** ..... Determination of the water contents (n =10) resp. the rates of recovery (n=3) of the different oil samples

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