

## **HYDRANAL™ Technical Information Sheet T005**

### **External Extraction of Water**

Some samples do not dissolve in the media used for Karl Fischer titration and their water cannot be extracted within the usual time of titration. In many cases the water analysis can be done using the indirect method with a KF oven. Some samples decompose at temperature necessary to evaporate their water completely. Often a KF oven is not available or it is not worth to purchase because of a rare demand.

The external extraction of water can be the solution for such problems. Methanol is very useful for water extraction. The solvent used has to be very dry, the water content should be max. 0.01%. [34741 Hydranal-Methanol dry](#) is perfect for this application.

Sample size and chosen amount of methanol have to meet the water content of the sample. Calculations should be based on a rule saying that:

**1 mL of methanol is able to extract 1 mg of water.**

The time required for extraction depends on the consistence of the sample. We experienced, that an extraction time of 2 h is sufficient for many samples but exceptions have shown that also 24 h up to 48 h are required in some special cases.

#### **Example method:**

A suitable sample size (depending on expected water content and bulk density of the sample) is weighed (by accurate back-weighing, example 15.9021 g) into a 100 mL Erlenmeyer flask equipped with a stirring bar and mixed with 50 mL Hydranal-Methanol dry. Ideally, the methanol is handled with a 50 mL pipette so that it is protected against humidity. The weight of the methanol has to be noted as well (example 38.9100 g).

The mixture is stirred for around 2 hours (or longer) then the sample is allowed to precipitate if required.

An aliquot of approx. 5 mL of the supernatant solution are taken out with a syringe and injected into the KF titration vessel (handled by back-weighing, example 4.416 g).

The water content of the aliquot can be measured according to all volumetric or coulometric standard procedures.

The titration shows the total amount of water in the aliquot (sample + methanol) in mg (example 8.771 mg)

50 mL of the methanol in use is handled in a similar way in a same flask. From this the blank value (methanol plus handling) is determined (example 0.0120%).

**Example calculation:**

|          |                  |
|----------|------------------|
| Sample   | 15.9021 g        |
| Methanol | <u>38.9100 g</u> |
| Total    | 54.8121 g        |

Aliquot of extract 4.416 g corresponds to 1.2812 g sample + 3.1348 g methanol.  
3.1348 g methanol (x blank, 0.0120%) contains 0.3761 mg water.

|                                  |                   |
|----------------------------------|-------------------|
| Totally titrated amount of water | 8.7710 mg         |
| Minus water mass blank           | <u>-0.3762 mg</u> |
| Water mass in sample             | 8.3949 mg         |

$$\% \text{ water} = \frac{8.3949 \times 100}{1281.2} = 0.6552\%$$

An Excel calculation sheet is available on request.

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