

Solutions for Testing of Used Oil

made by **ECHⁱ**



ECH Elektrochemie Halle supports customers involved in the testing of used oils.

ECH instruments are configured to provide customers with efficient and accurate solutions to used oil parameter tests, namely water content, total acid number (TAN) and total base number (TBN).

All ECH instruments are configured with full conformity to relevant ASTM, IP, ISO and UOP methods.

Water content

Water content in used oils is a critical parameter, and a test in which ECH is hugely experienced with. Excess water content in oils can reduce the life expectancy significantly. The effects of both free and emulsified water are more harmful compared to dissolved water therefore moisture levels in oils should remain below the saturation point. For in service oils this means that around 100 - 300 ppm (0,01 - 0,03 %) levels should be regulated. The most accurate test for the determination of water content in used oils is according to **ASTM D 6304** (procedure A, B & C) depending on sampling preference. Coulometric Karl Fischer titration will allow users to analyse the water content down to 1 part per million.

- Procedure A - Direct injection by **Aquamax KF Plus**
- Procedure B - Headspace technique by **Aqua 40.00 Vario**
- Procedure C - Oven evaporation technique by **Aquamax KF PRO Oil**

Total Acid Number (TAN) and Total Base Number (TBN)

TAN according to ASTM D 664 by **Titramax VT TAN/TBN**

TBN according to ASTM D 2896 by **Titramax VT TAN/TBN**

Water Content of Used Oil

Procedure A - Direct injection

aquamax KF Plus

Description

Procedure A is the method of **direct injection**, ideal for oils without any known compound interferences (high base number additives).

A known volume or mass of sample is to be injected into a pre-conditioned titration cell where any present water will be automatically titrated with results calculated and shown in ppm, % or mg/kg.

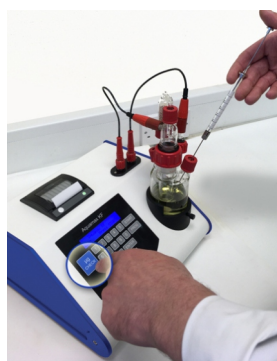
This technique has been the entry level method for many years and remains a tried and tested procedure.

The **Aquamax KF Plus** from ECH has been conforming to **ASTM D 6304** since its inception.

Designed with robust glassware, built-in battery, printer and available with a portable carry case, the Aquamax KF Plus allows for both in-laboratory and onsite analysis, allowing samples to be analysed directly at the source.



The Aquamax KF Plus fulfils the requirements of the standard ASTM D 6304: Standard Test Method for Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fischer Titration.



Water check button and syringe

Water check

The µg check button allows the operator to simply press go, inject 1 µL or maybe 10 µL of distilled water (as required by some ASTM methods) and verify if the instrument and reagent are working within their required specification.

The µg check overrides the programmed calculation and displays/prints out a report of the verification check. The coulometer then automatically reverts to the pre-programmed setting.

Features

- Simple operation
- 10 user programmable methods
- 1 ppm/100 %
- Results in ppm, mg/kg, % water, µg water
- Multi language display & printout
- Small footprint
- Integral high speed printer
- Integral battery
- Fully portable
- Low drift cell design
- Results Manager software
- Automatically compensated errors (patented technique)



Aquamax KF Portable with integral battery, carrying handle and transport case

Water Content of Used Oil

Procedure B - Headspace technique

aqua vario_{40.00}

Description

Each sample is weighed into a vial, the vial is sealed and placed in the oven module. Each vial is heated to the point the water vaporizes from the sample.

The vaporized water is then transported from the sample into the titration cell using a non-reactive carrier gas such as pre-dried ambient air as used in the ECH Closed Loop carrier gas technique.

Procedure B is currently the only available automated method of coulometric Karl Fischer titration, with ECH offering an autosampler with a large variety of sample plates.

Using a closed loop system with pre dried ambient air such as the **Aqua 40.00 Vario** from ECH can provide end-users with over 1000+ samples in one charge of coulometric KF reagents. No methanol can escape from the reagent into the laboratory air.



AQUA 40.00 Vario PLUS - automatic version with sampler

Flexible for different vial sizes



For different vial sizes:
Sample plate and oven unit
easily exchangeable

Features

- Configurable temperature programmes
- Automatic identification of interchangeable oven and sample plate of the autosampler
- Prioritized express samples can set individually by user
- Software complies with requirements of FDA to 21 CFR Part 11 (Software with user-specific access, routine methods for individual and definable user levels, profound documentation and archiving of all measured data)

Advantages of the AQUA 40.00 Vario

- Easy automation with autosampler
- Reduced reagent consumption
- No external gas required
- No evaporation of methanol from the reagent
- Additional gas drying is not required due to closed-loop circulation of extraction gas
- Stand-by titration for automatic conditioning and easy blank tests
- Short measuring times, even with complicated samples
- Suitable for more than 15 different vial sizes (2 R - 50 R)



AQUA 40.00 Vario
as manual version

Water Content of Used Oil

Procedure C - Oven evaporation technique

aquamax KF

PRO OIL

Description

The oven evaporation technique is ideal for low ppm water content samples as unlike procedure B, there is no blank value accrues.

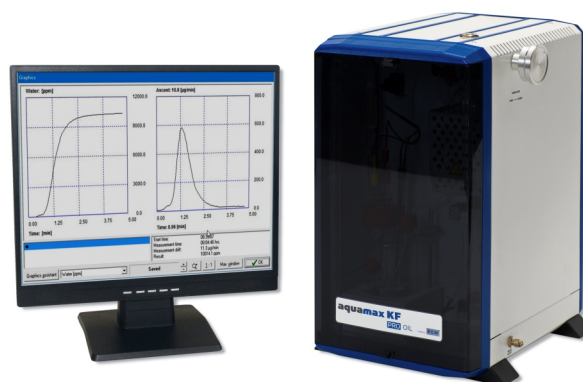
A known volume or mass of sample is to be injected via a gas tight syringe into the heating chamber. The sample should be heated to the point of water vaporization.

The water is transferred by a dry carrier gas (such as pre-dried ambient air with the ECH Closed Loop) directly into the titration cell where the coulometric reaction occurs.

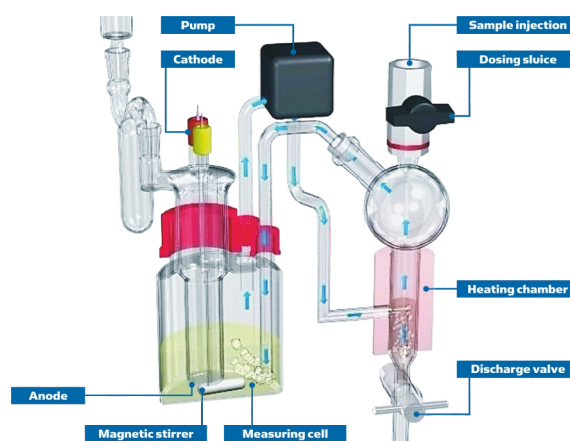
The sample itself remains in the evaporation chamber and is subsequently removed via automatic waste removal pumps. Sample heating must be monitored to avoid the risk of decomposition from the samples itself which could lead to unwanted compounds from the sample entering the titration cell and causing as a side reaction.

Using the correct temperatures and a closed loop carrier gas system yields benefits of analysing over 1000 samples in one charge of Karl Fischer solvent. Interferences of oil compounds are avoided by the indirect heating. There is no contamination of the coulometric titration cell.

The next generation of Aquamax KF systems, the **Aquamax KF PRO Oil** was specifically designed for titration according to procedure C.



The Aquamax KF PRO Oil fulfils the requirements of the standard ASTM D 6304: Standard Test Method for Determination of Water in Petroleum Products, Lubricating Oils and Additives by Coulometric Karl Fischer Titration.



Closed-loop carrier gas circulation

Advantages of the Aquamax KF PRO Oil

- Closed loop principle avoids methanol evaporation from the KF solvent
- Reagent capacity is used completely
- Additive and Sulphur side reactions minimized
- By large sample amounts low limit of detection (LOD)
- By using of temperature programs it is possible to separate free and chemically bonded water
- Temperature ramping program allows you to distinguish between various types of bonded water
- No blank value meaning true ppm accuracy
- Aquamax KF PRO Oil can be used in the laboratory or used as part of a mobile lab when taking a measurement from the sample point is critical
- Compact and rugged device

Total Acid Number (TAN) and Total Base Number (TBN)

titramax VT TAN/TBN

Description

Total Acid Number (TAN)

TAN analysis in used oils helps maintain equipment due to the corrosive nature of acidic components. The acid levels of oils can increase naturally over time, or when oxidised due to adverse reactions with oxygen. TAN is measured in KOH per gram of sample.

ECH provide a solution by conforming to the well renowned

ASTM method D 664 (Standard Test Method for Acid Number of Petroleum Products by Potentiometric Titration).

The **Titramax VT TAN/TBN** is available with an application guide specifically written for conformity to ASTM D 664 and is available both as manual system, or with 12, 16, 24 and 42 position autosamplers.

Total Base Number (TBN)

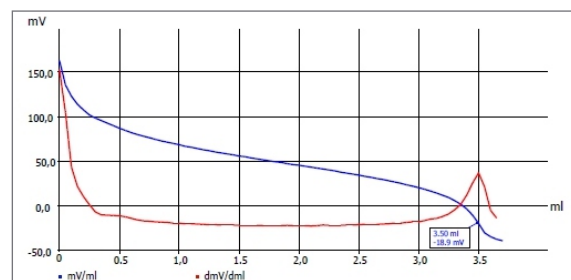
In order to neutralise the acidic components within the oils a certain level of reserve alkalinity additives are applied into the oil, these are monitored and are known as base number.

The base number of oils is defined as the oils ability to neutralise acids that produced during use. The higher the base number in the oil, the more acid it will be able to neutralize during use. TBN analysis in used oil analysis has direct relevance to the total acid number test and therefore often will be tested using the same equipment. Potentiometric titration is used to calculate the TBN.

The **Titramax VT TAN/TBN** can also be equipped with an application package for **ASTM D 2896** („Standard Test Method for Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration“) or for **ASTM D 4739** („Standard Test Method for Base Number Determination by Potentiometric Titration“).



Titramax VT TAN/TBN



Titration graph of TAN determination of an oil sample

Advantages of the Titramax VT TAN/TBN

- Complete measuring system for the determination of TAN/TBN
- Fully-automatic volumetric titration
- Precise adjustment of the titration parameters by control algorithms
- Preset measurement method allows an immediate start
- The result output can be adjusted to your needs by using a formula generator
- Universal titrator for a lot of other methods

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