

Solutions for Testing of Petroleum Products

made by **ECHⁱ**



ECH Elektrochemie Halle provides customers with a wide array of analytical instrumentation suitable for the analysis of compounds within all different petroleum products. ECH provide a range of instruments suitable for the measurements of parameters such as water content, TAN, TBN, H₂S and more.

All instruments are developed with efficiency and high sample throughput in mind. All ECH instruments designed for petroleum product testing are manufactured in accordance with standards from bodies such as ASTM, ISO & IP.

Water content

Determination according to the three procedures of the standard ASTM D 6304:

- 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 and D 4739 by **Titramax VT TAN/TBN**

Hydrogen Sulphide Content

Determination according to standard IP 570 and ASTM D 7621 (procedures B)
by **Sulfixmax GX Go** or **Lab** with **H₂S Headspace Module**

Hydrogen Sulphide & Mercaptan Content

According to standard UOP 163 by **Titramax VT SULPHUR**

Water Content of Petroleum Products

Water content in petroleum products is a speciality of ECH Elektrochemie Halle and testing to ASTM D 6304 is a method in which ECH have been at the forefront of development with. Testing water content in a specified sample is crucial in understanding the quality and specification level of the product, as well as assisting in the avoidance of any corrosion from excess water content. Comprising of three procedures, ASTM D 6304 covers the spectrum of analysis of water content in petroleum products, ECH has developed a unique solution to all three procedures.

Procedure A - Direct injection

aquamax KF Plus

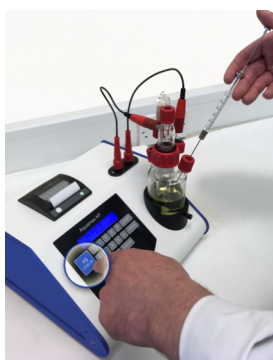
Description

Procedure A is the method of **direct injection**, ideal for light liquids and fuel samples without any known compound interferences.

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 and results calculated and shown in ppm, % or mg/kg.

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

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



Water check button and syringe

Water check

The μg check button allows the operator to simply press go, inject $1\ \mu\text{L}$ or maybe $10\ \mu\text{L}$ of distilled water (as required by some ASTM methods) and verify if the instrument and reagent are working with in 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.

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.

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)

Water Content of Petroleum Products

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 Petroleum Products

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.

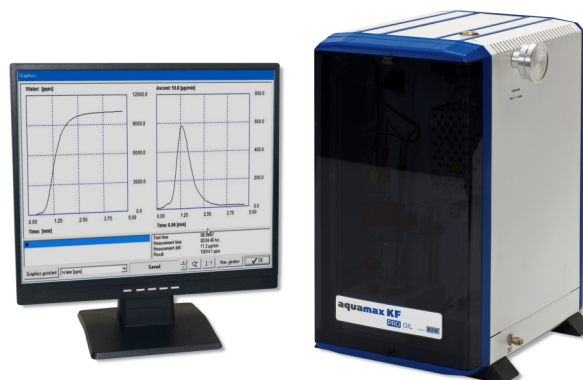
A known volume or mass of sample is to be injected via a gas tight syringe into the heating chamber. The sample should be heated to the point of water vaporization.

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.

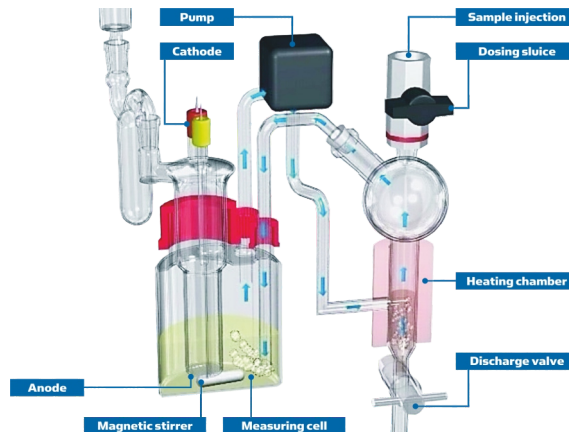
The sample itself remains in the evaporation chamber and is subsequently removed via automatic waste removal pumps. Sample heating must be monitored to avoid the risk of decomposition from the samples itself which could lead to unwanted compounds from the sample entering the titration cell and causing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



Aquamax KF PRO Oil with effective evaporation oven



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 amount low limit of detection (LOD)
- By using of temperature programs it is possible to separate free and chemically bonded water
- Temperature ramping program allows 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)

The analysis of TAN or neutralisation number is the determination of acid components within petrochemical samples, typically petroleum products contain acidic components in the form of additives, or from degradation products such as oxidation. TAN is measured in KOH per gram of sample. Trace amounts of acid in petroleum products are undesirable due to the corrosive nature.

ECH provide a solution by conforming to the well renown

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.

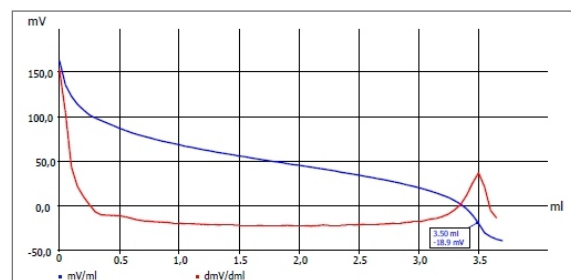
Total Base Number (TBN)

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 engine oil, the more acid it will be able to neutralize during use. TBN analysis in petroleum products has 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

Hydrogen Sulphide Content

Conform to standard IP 570 and ASTM D 7621 (procedures B)

sulfimax GX H₂S Headspace Module

Description

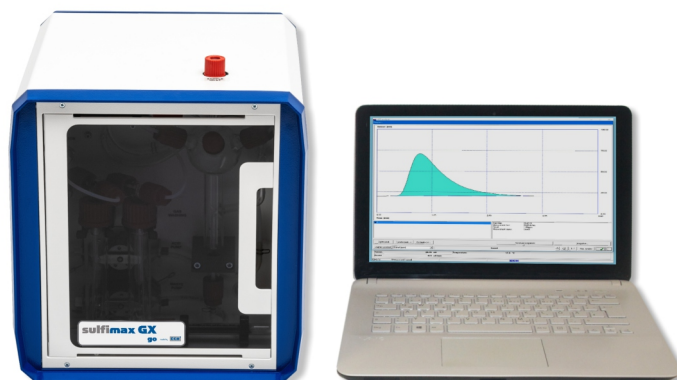
ECH have developed a total H₂S analyzer for the determination of H₂S in all petroleum products such as crude oil, gasoline, diesel fuels and marine fuels according to methods such as IP 570 and ASTM D 7621 (procedures B), through to solid products such as sludges, bitumens, tar & elemental sulphur.

For petroleum and solid products, H₂S gas is heated out using an additional headspace module. Each sample is weighed in a vial, the vial is sealed and placed in the headspace oven which is heated up to 180 degrees C depending on sample composition.

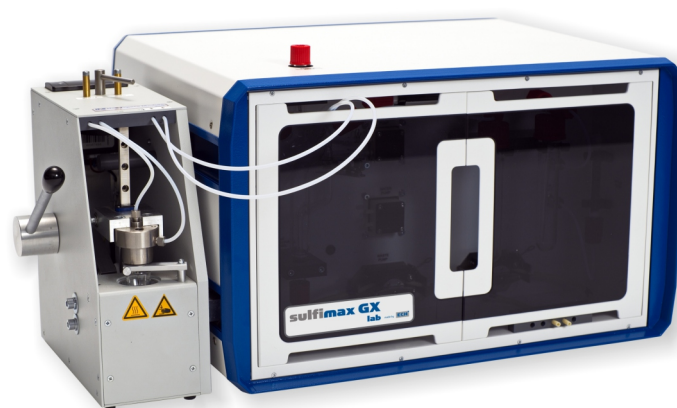
The H₂S gas is extracted from the sample and using a pump/tubing system is transported into the **Sulfimax GX** system where the detection of H₂S occurs down to 0.1 ppm.

Advantages

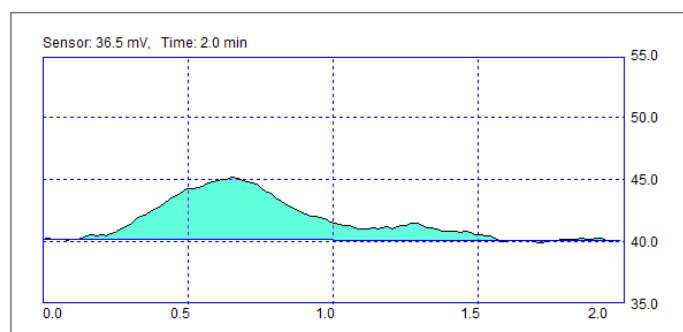
- Easy connection to the H₂S analyzers for gases and liquids Sulfimax GX Lab and Sulfimax GX Go
- No sample preparation necessary for solid samples
- Very low H₂S concentrations detectable
- Measuring time from 2 to approx. 15 min, depending on the H₂S content of the sample
- Manually operated and robust measuring system
- Easy handling for everyone
- No solvent cleaning of extraction vessel necessary (disposable vials)
- Variable temperature adjustment from 30 to 180 °C



Sulfimax GX Go - H₂S analyzer as compact version



Sulfimax GX Lab - H₂S analyzer as laboratory version with connected H₂S Headspace Module



Determination of volatile H₂S from bitumen by headspace technique

Hydrogen Sulphide & Mercaptan Content

Conform to standard UOP 163

titramax VT SULPHUR

Description

Measuring for H₂S concentration is critical. Hydrogen sulphide (H₂S) is a gas which can cause hugely detrimental effects to a refineries production and safety.

The MAC (Maximum Allowable Concentration) of H₂S is as low as 10 ppm, but H₂S will already breach the odour nuisance threshold at 3 - 5 ppm. Anything over 10 ppm will begin to have serious health consequences such as eye defections, loss of sense and smell, and at over 1000 ppm fatality would be certain in only minutes or seconds.

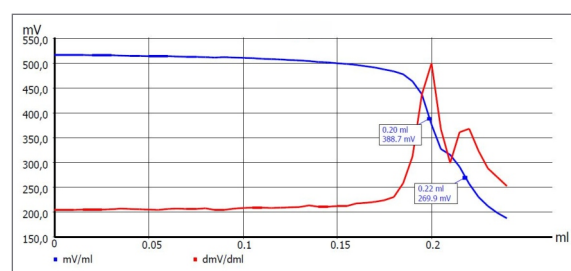
Aside from the health risk, H₂S gas is extremely corrosive and the presence of H₂S can damage technical equipment within the refinery and will corrode concrete at a rate of up to 10 mm per year.

A more traditional method for the determination of hydrogen sulphide comes in the form of titration, specifically, potentiometric titration according to UOP 163. This method details the determination of hydrogen sulphide and mercaptan sulphur in hydrocarbons by potentiometric titration. Typical samples include gasoline, naphtha, light cycle oils and similar distillates. The lower limited of quantification is 0.2 ppm mercaptan and 1 ppm hydrogen sulphide.

ECH offers the Titramax VT SULPHUR which is equipped with the with 10 mL exchangeable unit and metal combination electrode which is suitable for the determination of hydrogen sulphide and mercaptan sulphur content.



Titramax VT SULPHUR



Titration graph of a sample

Advantages of the Titramax VT SULPHUR

- Complete measuring system for the determination of hydrogen sulphide and mercaptan sulphur
- 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
- Gas tight titration vessel with purging fittings

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