

## Solutions for Testing of **Battery Components and Raw Materials**

made by **ECH<sup>i</sup>**



ECH Elektrochemie Halle supports customers involved with the testing of battery components and their raw materials.

ECH offers analytical systems for the determination of water content in plastics, metals and liquids, for the analysis of interfering components such as H<sub>2</sub>S in battery electrolytes and alkali in cathode materials as well as for the testing of electronic assemblies.



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# Water Content of Battery Components

Determination according to ASTM D 6304

# aqua vario

40.00

Manual version

## Description

The development of high-performance batteries is seen as the key to progress in electromobility and the widespread use of renewable energies. Lithium-ion batteries have considerable advantages over conventional batteries. They can be produced in compact, lightweight designs and have a high degree of efficiency.

The entire construction of the lithium-ion accumulator must be done in such a way that the water content is very low (content of  $H_2O < 20$  ppm), otherwise the water reacts with the conductive salt  $LiPF_6$  to HF (hydrofluoric acid). The hydrofluoric acid leads to a strong reduction in capacity and service life. For production and development, this means constant control of the water content in all raw materials and assemblies used.

The **AQUA 40.00 Vario** measures the water content of samples of all kinds precisely and accurately. Here, the coulometric Karl Fischer titration is combined with the headspace technique.

With this technique the sample is placed into a headspace vial then weighed, crimped and placed into the headspace oven. Using constant temperature or intuitive temperature gradient heating programs ranging from 35 - 300 degrees C, the water content is extracted from the sample. From there, only the water is carried into the titration cell by a closed loop gas transfer. Once the water reaches the titration cell, the coulometric reaction occurs allowing accurate water content measurements down to 1 ppm. Unlike the alternative methods, loss on drying, Karl Fischer titration is only sensitive to moisture, so any potential cross contamination is avoided. Not only does this technique provide more accurate results, but it also uses far less sample (often less than 1 gram) for analysis.

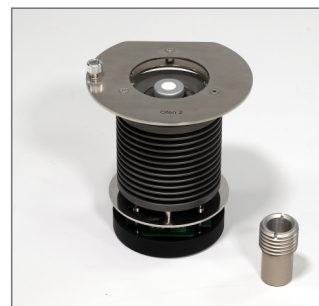
## Features

- Closed loop gas circulation without external gas supply
- Configurable temperature programmes
- Manual oven module for small numbers of samples
- Automatic identification of interchangeable oven
- Prioritized express samples can set individually by user
- Software complies with requirements of FDA to 21 CFR Part 11 (Software with userspecific access, routine methods for individual and definable user levels, profound documentation and archiving of all measured data)



AQUA 40.00 Vario as manual version

## Flexible for different vial sizes



Easily exchangeable oven unit for different vial sizes with automatic identification

# Water Content of Battery Components

## Determination according to ASTM D 6304

# aqua vario

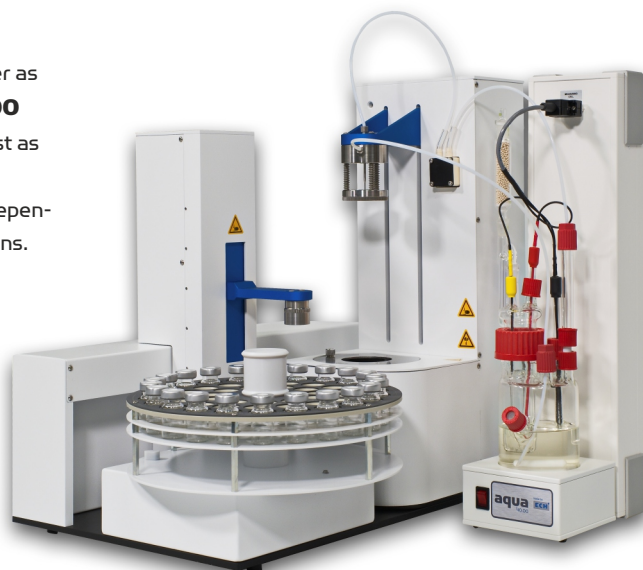
40.00 PLUS

Automatic version  
with autosampler

### Description

The manual version is subsequently expandable with an autosampler as the number of analyses grows. The automatic version **AQUA 40.00 Vario PLUS** has the same features as the manual version and is just as flexible in terms of vial sizes that can be used.

The oven unit and sample plate used are automatically identified. Depending on the size of the vials, the sample plates have up to 50 positions.



AQUA 40.00 Vario PLUS - automatic version with autosampler

### Flexible for different vial sizes



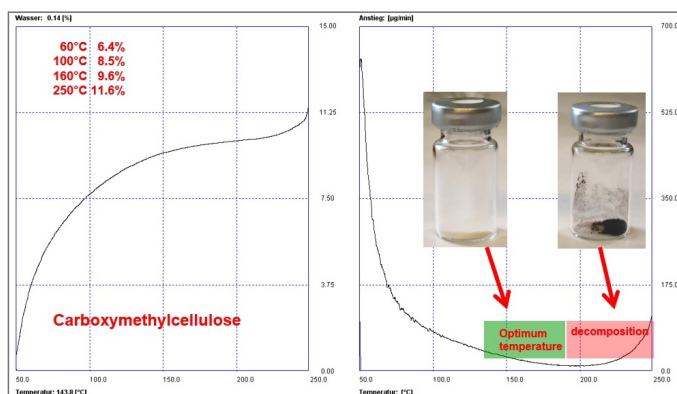
Easily exchangeable sample plate for different vial sizes with automatic identification



SWOP Box - universal reagent exchange module for all Karl Fischer titrators (regardless of manufacturer) for contactless exchange of the reagents without air moisture entry

### Advantages of both versions

- 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, correspond to 0.1 to 40 mL sample volume)
- Complete plastic bodies are analyzable
- Automatic identification of interchangeable oven unit and sample plate of the autosampler
- Compact design



Water release of carboxymethyl cellulose as a function of temperature (temperature programme 50 - 250 °C, 3 K/min)

# Water content of battery electrodes

Water determination at temperatures up to 1300 °C

## aqua HT 1300 40.00

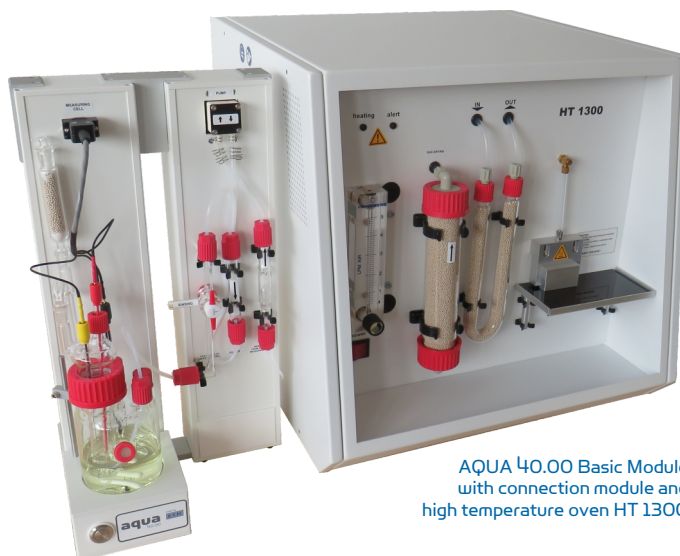
### Description

The high temperature oven **HT 1300** is an extension module with high performance for the Karl Fischer titrator **AQUA 40.00** for extracting the water from samples which release the water at higher temperatures (300 to 1300 °C). Therefore, chemical reactions forming water as a reaction product can be controlled.

Samples are put into small sample boats into the hot area of the oven. A special valve system transports the sample into the oven without interrupting the carrier gas flow.

In the inert gas flow, the water is completely released from the sample and transferred via the connection module to the measuring cell of the **AQUA 40.00 Basic Module**.

Due to the two-stage pre-drying of the inert transport gas in the integrated drying tower a very low background drift is achieved.



AQUA 40.00 Basic Module with connection module and high temperature oven HT 1300

### Applications

Water determination in solid, pasty and liquid samples at heating temperatures up to 1300 °C, e. g.

- Battery electrodes
- Hydroxide contents of oxides
- Metals and alloys
- Powder mixtures

### Advantages

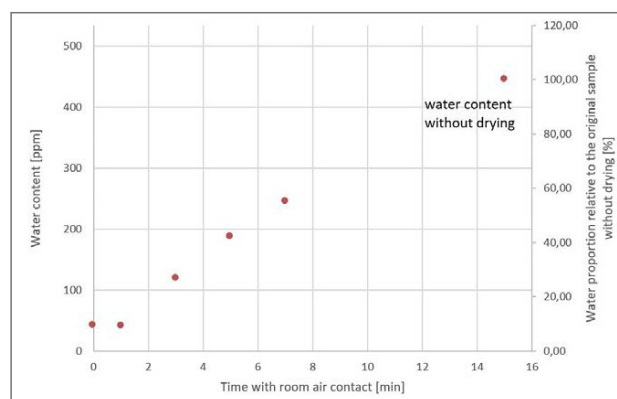
- No sample pre-treatment necessary
- Short measurement time
- Easy to operate manual feeder
- AQUA 40.00 software with extension to control high temperature oven

### Features

- High temperature heating for trace amounts of water
- Can be coupled directly with the AQUA 40.00 Basic Module
- Uniform software with integrated oven temperature control
- Sample supply in ceramic or quartz glass boats
- Gas supply with nitrogen or argon
- Isothermal heating or temperature program



Sample dosing with boats of ceramic or quartz glass



Measurements of the water content of powdered electrode material ( $\text{LiCoO}_2$ ) after removal from the drying process (20 min, 600 °C)

## Working under controlled conditions

### Weighing and measuring samples in a closed atmosphere

# gloveBOX

## Description

The **GloveBox** is designed for easy and functional work under defined conditions. It offers the possibility of comfortable sample preparation, e. g. without the influence of oxygen or under low humidity.

The **GloveBox** is designed and manufactured according to customer requirements.

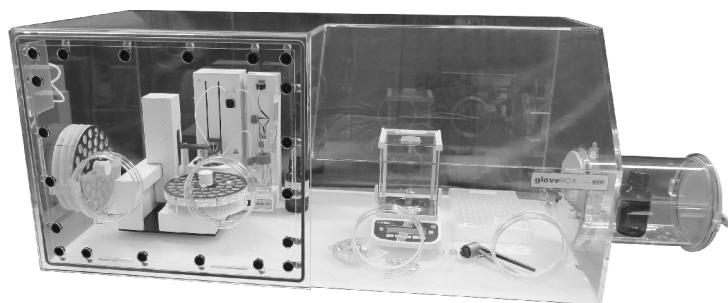
The chamber is made entirely of acrylic glass, which ensures a clear view into the working area. The gloves (made of butyl, neoprene, latex) are attached to the front with flanges and can be changed easily. The number of gloves depends on the length of the **GloveBox**, so that the entire interior can be easily operated.

On the side (optionally on the left or right) is a large sample lock, which is integrated into the maintenance hatch. The maintenance hatch contains two gas connections for gas supply and gas drain.

The gas supply hits a gas baffle plate to prevent gas turbulence for precise weighing operations. Connections for the passage of electrical cables, e. g. for a scale, are possible.



GloveBox version with 1400 x 700 x 600 mm offers plenty of space and a large maintenance hatch for easy loading of the the box with equipment



GloveBox version with 4 glove openings and large maintenance hatch on the front side, here equipped with balance and Karl Fischer titrator, integrated holders for sample plates on the left side wall

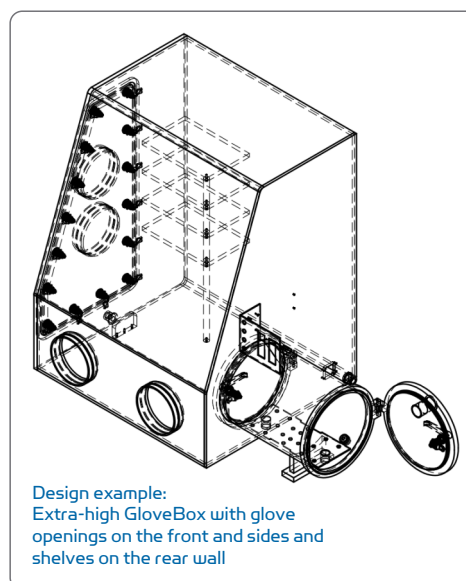
## Applications

Sample preparation for

- Analysis of battery components
- Disassembly of battery samples
- Filling for safe storage of samples

## Advantages

- In-house design department for customised design
- Different sample locks available
- Adaptation to working with scales
- Free view of the work surface as there is no frame construction
- Gloves can be easily attached and changed as needed
- Inexpensive, robust design made of acrylic glass



Design example:  
Extra-high GloveBox with glove openings on the front and sides and shelves on the rear wall

# Residual alkali content in cathode materials

Determination of the carbonate and CO<sub>2</sub> contents according to ISO 11045-1

## titramax VT ALKALINITY

### Description

The **Titramax VT ALKALINITY** can be used to determine the content of metal carbonates and soluble bases in cathode materials.

The hydroxides and carbonates influence the properties of the batteries and can lead to swelling. They are important surface parameters in the manufacturing process.

The measurement uses a volumetric titration method with hydrochloric acid (0.01 – 0.1 mol/L). The powder sample is extracted with water under exclusion of air, then filtered off and the water phase is titrated automatically with the acid.

The user has to enter the sample weight into the menu. The titration speed is precisely adjusted to the reaction rate by control algorithms. The titration is carried out automatically up to two end points of the measurement. The results at pH 8.5 and 4.5 are determined.

The titrator also meets the requirements of the standards ASTM D 1121, DIN EN ISO 787-4, DIN ISO 125, ISO 10539.



Titramax VT ALKALINITY

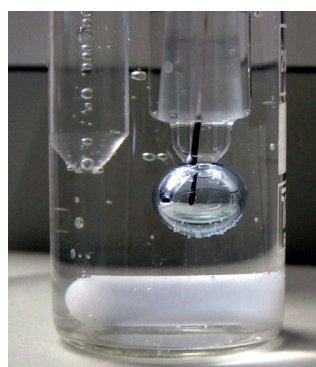
### Applications

The **Titramax VT ALKALINITY** titrator is suitable for analysing

- the residual alkali content of battery materials,
- the purity of lithium salts,
- of boiler water, cooling water, drinking water

The titrator can also be used for other titrations in battery production,

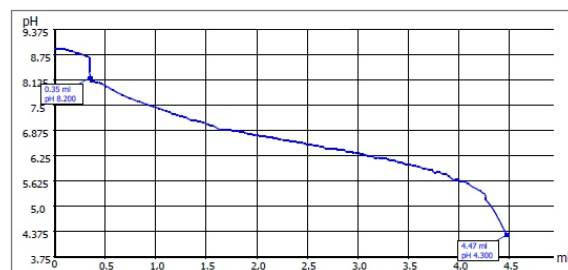
- pH titrations (e. g. phosphoric acid, hydrofluoric acid),
- complexometric titrations (metal ions such as nickel, cobalt, manganese),
- redox titrations



Titration tip and pH-electrode in sample solution

### Advantages

- Complete measuring system for the determination of alkalinity
- 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



Titration graph of a sample



# H<sub>2</sub>S content of battery electrolytes

## Determination of interfering influences

# sulfixmax GX go

## Description

The **Sulfixmax GX Go** determines hydrogen sulphide and volatile sulphides in liquids and gases. The sample can be dispensed directly by syringe without pretreatment. By effective gas extraction the H<sub>2</sub>S is completely expelled from the sample. Interferences due to the sample matrix practically do not occur. The released H<sub>2</sub>S gas is conducted to the highly sensitive sensor, which detects H<sub>2</sub>S in the range of 0.01 to 10,000 ppm. A typical measurement takes 5 min, depending on the sample composition. If the **Sulfixmax GX Go** is extended with the optionally available H<sub>2</sub>S Headspace Module, solid and pasty samples can also be measured.



H<sub>2</sub>S analyzer Sulfixmax GX Go as compact version with small footprint



## Advantages

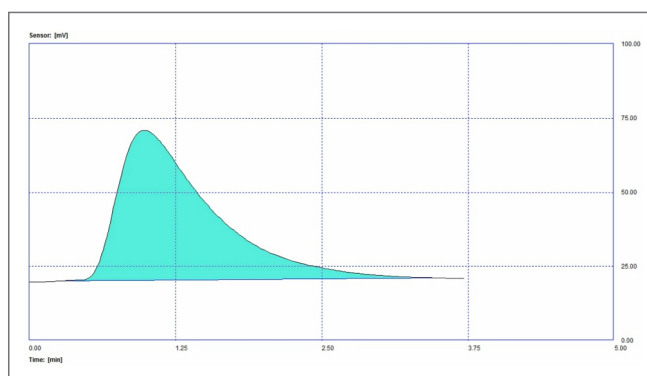
- Analysis of the original sample
- No sample preparation
- Minimized cross sensitivity through the indirect method
- Suitable for on-site use



Sulfixmax GX Go as version with integrated control unit and battery operation

## Features

- Complete separation of H<sub>2</sub>S from the sample by effective gas extraction
- Typical measuring time 5 min (depending on sample properties)
- Automatic addition of acid or buffer
- Automatic emptying after end of measurement
- Simple calibration
- Software: simple, clear, intuitive
- Dosing manually by syringe
- Definition of own methods for device control



Typical measurement - automatic peak analysis / interpretation

# Layer thickness of metal surfaces

## Determination of thickness of metal layers on wires and strips

# SnLAYER

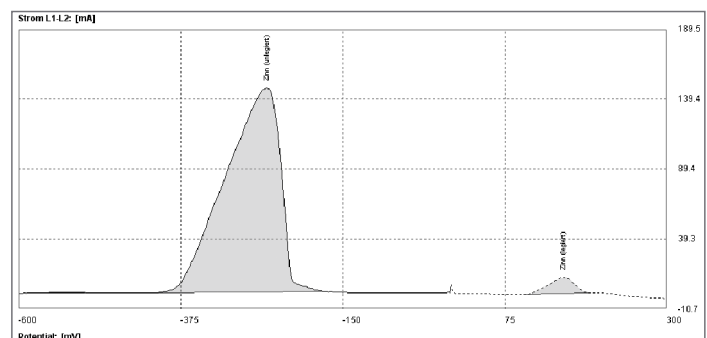
## Description

When coating metals, wafer-thin layers are often sufficient to achieve the necessary effect. At the same time, homogeneous thin layers should be achieved that fulfil the required electrical properties and stability over many years. A few additional micrometres mean an increased, useless consumption of valuable material and thus unnecessary costs.

With ECH's **SnLAYER** analysis system, the coating thickness on metals is determined quickly and with high precision with only one single measurement. For this purpose, a new electrochemical method was developed, which is based on coulometric voltammetry and is oriented towards the standards DIN 1787 and DIN 40500, Part 5. The patented potential scan method enables the simultaneous determination of free and alloyed parts of coatings, e.g. tin on copper. A typical measurement takes 8 min (non-alloyed and alloyed tin).



SnLAYER for determination of thickness of metal layers



Typical measurement curve



Application: Tin on copper sheet strip

## Advantages

- Complete measurement system for precise determination of the layer thickness of metal coatings
- Differentiation of free and bounded tin
- Customer-friendly handling, intuitive software
- Comprehensive statistic module
- Wide dynamic range for various wire-diameters and layer thickness
- Universally applicable for different types of layers

## Features

- Typical duration: 8 min (non-alloyed and alloyed tin), < 2 min (non-alloyed tin)
- Pre-defined methods specified for certain wire types
- Generation of individual methods
- Determination of the mass of the coating by integration of the current (amount of electric charge)
- Determination of the thickness with only one measurement

## Applications

- Measurement of tin layer thickness on copper wires, copper sheets, wire strands
- Determination of alloyed and unalloyed tin
- Analysis of nickel, silver, copper and alloys on copper, steel, Percon and others
- Quality control in the production of circuit boards, cables and wires

# Micro test method for electronic assemblies

## Evaluation of the quality of the sinterability of surfaces

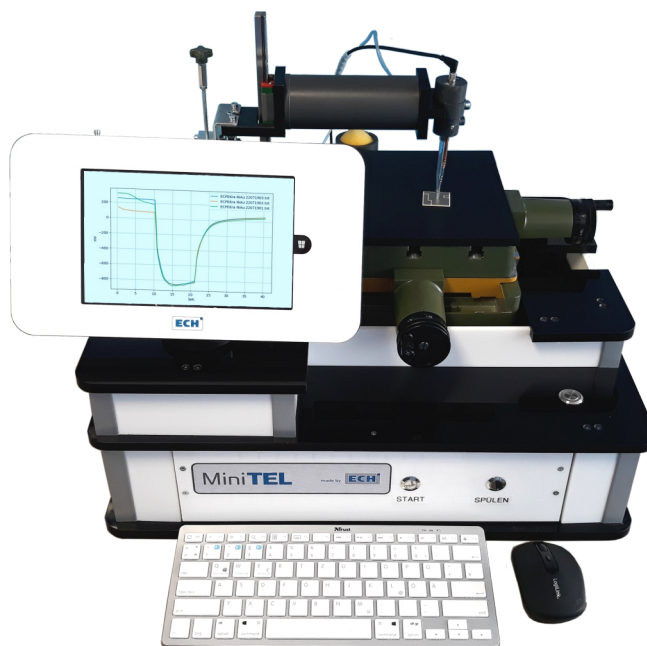
# MiniTEL

## Description

Fail-safe electronic assemblies become increasingly important. They play a crucial role in autonomous mobility and power electronics where high currents flow, such as wind turbines, solar modules, energy storage systems and batteries. An early detection of corrosive processes on metallic surfaces is essential to avoid an impair of function of the assemblies.

Use the **MiniTEL** to characterise corrosion properties of PCB metallisations, to test sintered contacts of power electronics, to check incoming metallisations and to evaluate solderability and sinterability.

With the **MiniTEL** micro test method, it is now possible for the first time to evaluate the quality of the sinterability of surfaces easily, quickly and reliably during the production process. Defective material can be detected and sorted out.



MiniTEL - Measuring station with electrochemical mini measuring cell

## Applications

Electrochemical testing of micro- and macrostructures of metallic surfaces, e. g.

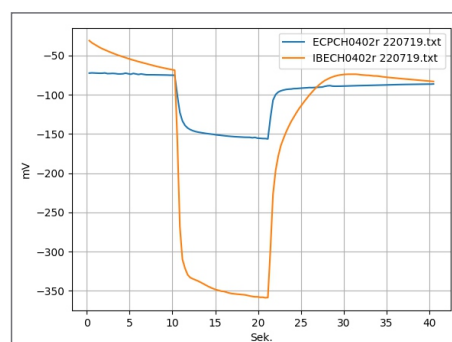
- classic printed circuit board coatings
- sintered surfaces
- wires and strands

## Advantages

- Different sample shapes measurable (plates, wires, contacts)
- Easy handling
- Ready for operation quickly after switching on
- Very short analysis times
- Minimal chemical consumption
- Automation possible
- Adapter for motor vehicles for on-site use
- Measuring head can be implemented in flow lines for online production monitoring

## Features

- Suitable for different sample shapes (plates, wires)
- Very short analysis times: 30 to 60 s per measurement
- Low chemical consumption: 100 µL per measurement
- Operation via integrated touch screen computer
- Editable control software with export function
- Coloured result display depending on quality level



Detection of contaminant layers (sulphur) on solder contact

## Global Sales Division



### **ECH Scientific Limited**

Building 69, Wrest Park, Silsoe

Bedfordshire, MK45 4HS

United Kingdom

Tel.: +44 (0) 1525 404747

Fax: +44 (0) 1525 404848

E-Mail: [info@echscientific.com](mailto:info@echscientific.com)

Web: [www.ech.de](http://www.ech.de)

## Manufacturer



ELEKTROCHEMIE HALLE

### **ECH Elektrochemie Halle GmbH**

Otto-Eißfeldt-Str. 8

D-06120 Halle (Saale)

Germany

Tel.: +49 (0) 345 279570-0

Fax: +49 (0) 345 279570-99

E-Mail: [info@ech.de](mailto:info@ech.de)

Web: [www.ech.de](http://www.ech.de)