

## A versatile measuring setup for the electrochemical characterization of materials developed for energy storage devices

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### A measuring setup offering unique features - the Microcell HC

The **Microcell HC** setup is suitable for the **electrochemical characterization** of liquids, gels, and polymers with a low to high viscosity [1].

Depending on the measuring cell, the measurements can be performed in both a **two- and three-electrode setup**, during which the **temperature of the sample** can be controlled quickly and precisely.

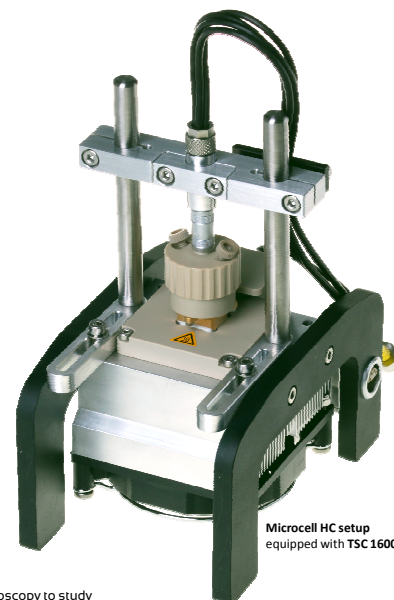
#### Essential features

- Large **temperature range** between **-40 °C** and **+100 °C**; possible limitations to the range depend on the measuring cell used and measuring conditions.
- Quick temperature control with maximum **temperature ramping rate up to 60 °C/min** depending on the measuring cell used.
- Precise **temperature control** with a **tolerance of ±0.1 °C**.
- Measurement of **volatile samples** when using a sealed measuring cell.
- **Small sample volume**, varying with the design of the measuring cell, ranging from 70 µl to 1.6 ml.

Only a **small sample volume** (milligram range) is required, which allows for electrochemical analyses of substances that are only available in small amounts and/or extremely expensive.

A special connecting system ensures a fast exchange of sample cells between measurements. This guarantees a high sample throughput and allows for measuring different components of energy storage devices in a short time.

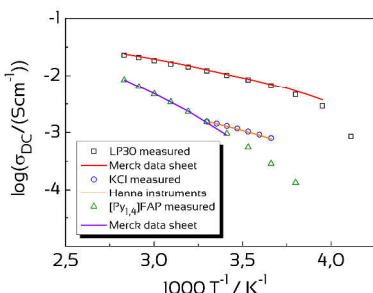
- **Fast and comfortable assembly.**
- **Easy-to-change electrode system** (gold, platinum, glassy carbon).
- Quick exchange of special **micro-reference electrode**; pseudo-reference electrodes as well as electrodes of the second kind available [2].
- Measurements possible **outside or inside a glove box**.
- **Turn key system** for fully-automated measurements under temperature control in combination with **METROHM** devices.
- Easy-to-use, time efficient and flexible software for impedance data analysis available (**RelaxIS - Impedance Spectrum Analysis**).



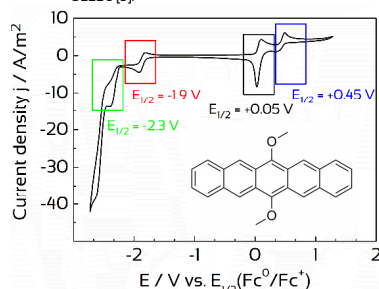
Microcell HC setup equipped with TSC 1600

### Electrochemical characterisation of liquid samples

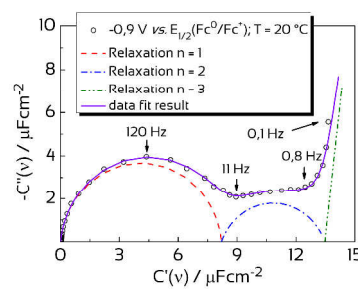
A) Impedance spectroscopy to study the sample's temperature dependent dc ion-conductivity.



B) Cyclic voltammetry studies of the **electrochemical stability** of electrolyte systems, electrochemical reaction kinetics or the **HOMO-LUMO gap** of dyes for OLEDs [3].

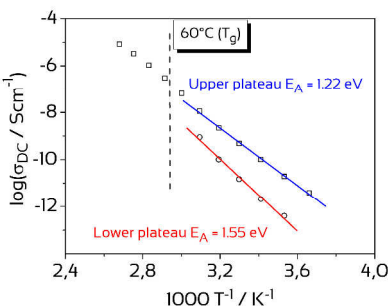


C) Electrochemical impedance spectroscopy to study the differential **capacitance** of electrode / electrolyte interfaces [4].

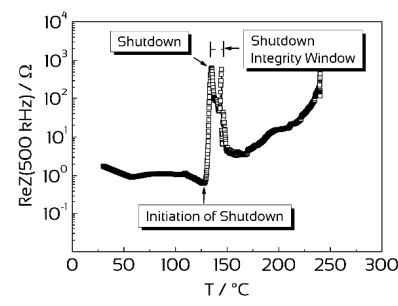


### Electrochemical characterisation of polymeric and solid samples

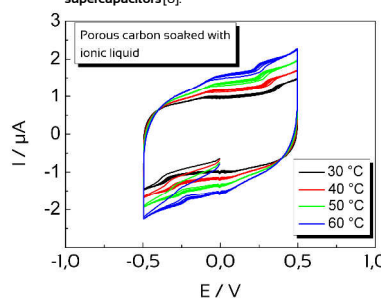
A) Impedance spectroscopy to study the sample's temperature dependent dc ion-conductivity.



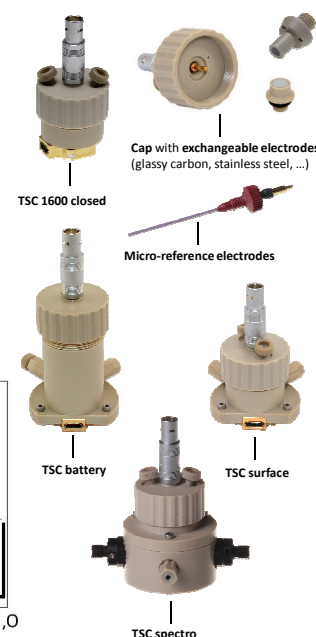
B) **Hot electrical resistance** studies of separator materials soaked with battery electrolyte [5], determination of **MacMullin numbers**.



C) Electrochemical impedance spectroscopy to study the electrochemical properties of porous carbon / electrolyte systems used for **supercapacitors** [6].



### Available measuring cells



### Outlook

- Development of **further applications** for established measuring cells, e.g. coupling with gas analytics [7] allowing for detecting decomposition products during cycling.
- Enhancement of the **automation** degree by embedding the Microcell HC in standardised liquid handling and sample preparation systems.
- Design of **novel measuring cells** compatible with the Microcell HC setup for special applications, e.g. the investigation of active materials for solid oxide fuel cells at high temperatures.

**Aim: One measuring setup** (Microcell HC) offering compatibility with a large variety of measuring cells for **almost all electrochemical issues**.

- Development of **standard measuring routines** for scrutinizing material properties like dc ion-conductivity or electrochemical stability.

### References

- [1] B. Huber, M. Drüscher, B. Roling, *Nachrichten aus der Chemie* **60** (2012) 1213-1214.
- [2] B. Huber, B. Roling, *Electrochim. Acta* **56** (2011) 6569-6572.
- [3] J. Schwaben, N. Münster, T. Breuer, M. Klues, K. Hams, G. Witte, U. Koert, *Eur. J. Org. Chem.* **2013** (2013) 1639-1643.
- [4] M. Drüscher, N. Borisenko, J. Wallauer, C. Winter, B. Huber, F. Endres and B. Roling, *Phys. Chem. Chem. Phys.* **14** (2012) 5090-5099.

- [5] E. P. Roth, D. H. Doughty, D. L. Pile, *J. Power Sources* **174** (2007) 579-583.
- [6] Data provided with courtesy by Dipl. Chem. Thomas Jänsch (Working group of Prof. Dr. B. Roling, Philipps-university of Marburg)
- [7] Z. Peng, S. A. Freudenberger, Y. Chen, P. G. Bruce, *Science* **337** (2012) 563-566.
- [8] www.rhd-instruments.de