

ProboStat™

Sample holder cell system for electrical and other characterization at high temperatures and under controlled atmospheres



NORECS

Outline

1. ProboStat™

2. Base unit and accessories

3. Measurement setups

4. Other ProboStat™ related products

ProboStat™

ProboStat™ - a versatile sample holder for measurements of electrical properties, transport parameters, and kinetics of materials, solid/gas interfaces and electrodes at high temperatures under controlled atmospheres.

Main features:

Overall design: Single end fixture of all parts with closed end enclosing tube 40 mm outer diameter, 30-60 cm long

Sample: 10-24 mm diameter disk
25-50 mm long bar

Electrodes: 2, 3, or 4

Temperature: Typical long term: <1400°C
shorter term: <1600°C

Atmosphere: Oxidizing, inert, reducing, corrosive;
Wet or dry
Low vacuum 10^{-2} mbar to atmospheric pressure
(15 bar with enclosing steel tube)
Single or dual chamber modes





ProboStat™

The ProboStat™ excels in easy exchange of samples and electrodes, and in versatility for many different methods

Materials properties measured and applicable methods:

- Conductivity vs. T , pO_2 , pH_2O , ect.
- DC, AC, impedance spectroscopy
- Dielectric properties, loss, ect.
- Disk, van der Pauw, and bar geometries
- 2, 3, and 4 electrodes
- Ionic transport number
- Proton transport number
- H/D isotope effect
- Seebeck coefficient
- Annealing or sintering under controlled atmospheres
- I-V characteristics
- Fuel cell component and single cell testing
- Electrode kinetics
- Electrochemical pumping, gas permeation and electrocatalysis with gas analysis (e.g. GC or MC) on outlets
- Sensor testing
- Poling of ferroelectrics possible with the high-voltage (10 kV) version

ProboStat™ base unit

The base unit is the central unit in the ProboStat™ system

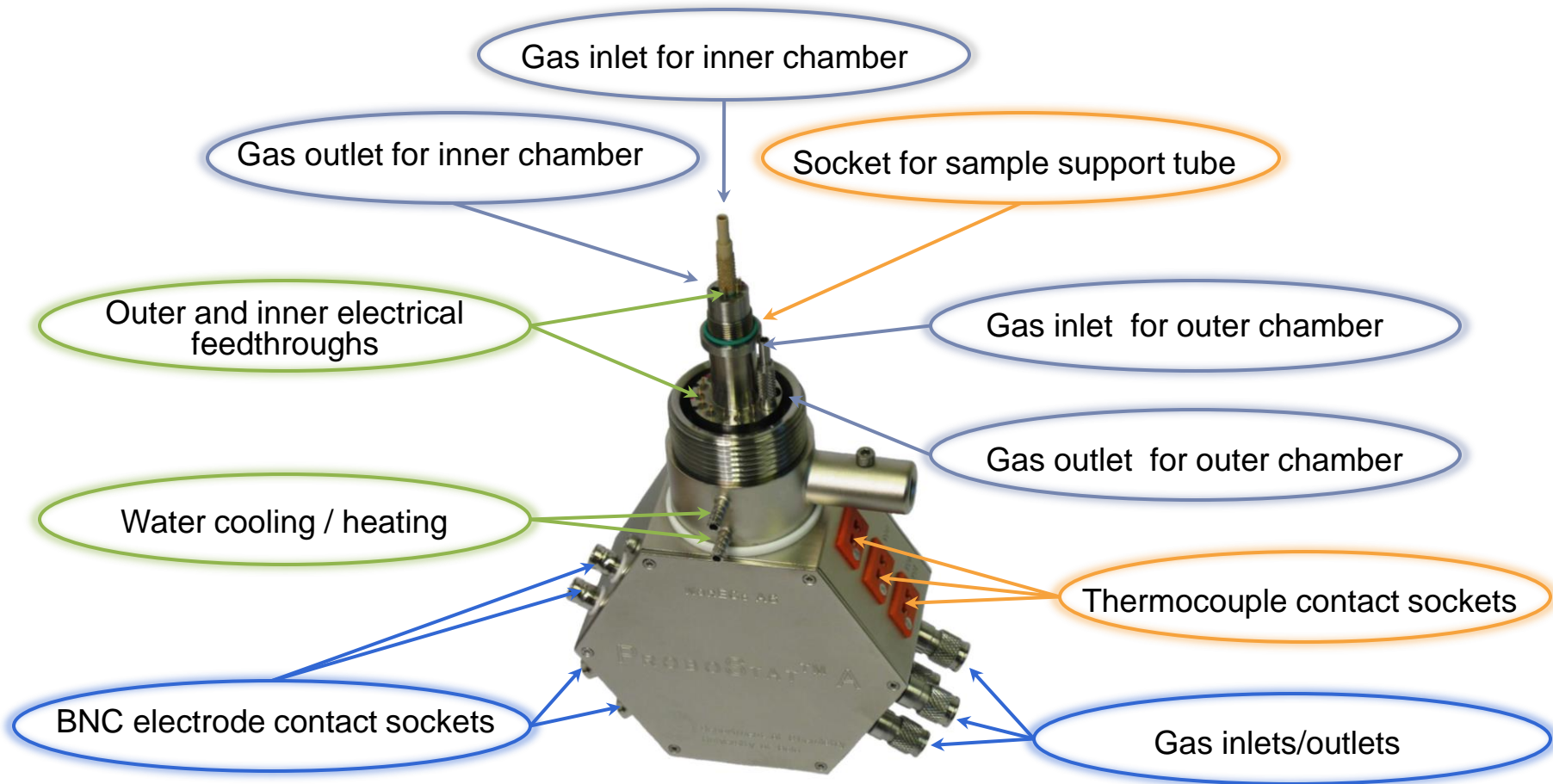
The ProboStat™ base unit features:

- Ni-plated brass construction for good heat conduction (stainless steel option)
- O-ring sealed fixation of sample support tube and outer enclosing tube
- Spring load fixation collar
- 16 electrical feedthroughs (6 for electrodes, 4 for shields and 6 for thermocouples)
- 6 BNC electrode contact sockets
- 3 thermocouple contact sockets
- 3 toggle switches for grounding and shielding options
- 4 gas inlets/outlets with Swagelock quick-connects for two gas chambers
- cooling/heating water hose fittings



ProboStat™ base unit

ProboStat™ base unit





ProboStat™ accessories

The ProboStat™ system has a wide range of accessories designed for different types of electrochemical measurements

ProboStat™ accessories

Outer enclosing tubes

40 mm outer diameter closed tubes of alumina or silica.

Sample support tube assemblies

Presently come in the standard designs for 10 – 24 mm diameter disks and for bar samples.

Spring load assemblies

Keeps sample and electrodes in contact and position.

Electrode contact leads

Come in variety of types. The standard material is high purity Pt. Other metals may be applied.

Thermocouples

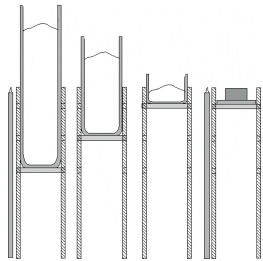
Up to 3 thermocouples can be used. The type (commonly S or K) can be chosen by the user.

Gas supply tubes

Send gas directly to the sample area. Supply gases to the inner and outer chambers of the cell.

ProboStat™ accessories (optional)

Crucible and sample plate holder

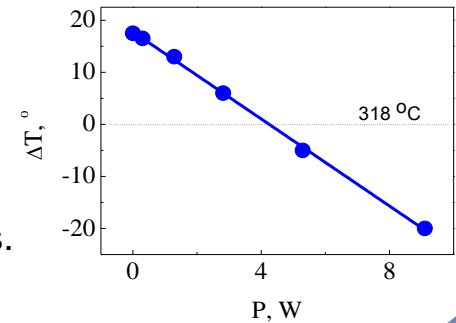


A special support tube with a stop plate to hold crucibles for annealing powders or samples, and sintering under controlled atmospheres or vacuum.

Internal heater

Can be used for creating temperature gradient in Seebeck coefficient measurements.

Temperature gradient across a bar sample as a function of power supplied to the internal heater.



Liquid cell assembly



The ProboStat™ system can be equipped with a liquid cell assembly to measure the dielectric constant of liquids at RT – 200 °C.

Top flange and open end outer tube

Open end outer tube with flange for evacuation and optical access to disk sample top surface/ electrode.



... and more...



Standard ProboStat™ packages

WE CAN RECOMMEND AND DELIVER

- *Extensively furnished version*

- *Normal plus version*

- *Normal version*

- *High voltage normal version*

Options suitable for laboratories which value the cost-effectiveness of direct acquirement of new techniques without trial and error and development time. These examples may also be suitable systems for projects where a large range of techniques are expected to be applied in the studies of a material or class of materials.

- *Minimum version*

- *Base unit system*

Options for those who want to make their own accessories and connections. It may also serve those who want to equip an earlier package with one or more units to increase measurement throughput and make better use of all accessories.

- *Custom systems*

A system designed specifically for your needs.

Standard ProboStat™ packages

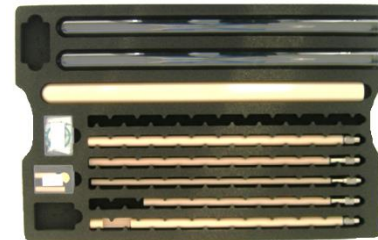
Example: Extensively furnished system package

Typical use:

- Disk samples of varying diameters
- 2, 3 and 4 electrode setups
- Van der Pauw 4-point measurements
- Bar samples (4-point and Seebeck coefficient measurements)
- Conductivity, impedance spectroscopy, DC measurements
- Concentration cells; transport number, permeability, fuel cell testing etc.

System contains:

- Ni-plated brass base unit
- 2 outer tubes (alumina and silica)
- 3 alumina sample support tube assemblies
- 2 alumina spring load assemblies
- 3 gas supply tubes (alumina and silica)
- 4 thermocouples (Pt/Pt10%Rh)
- 15 Pt/alumina electrode connection assemblies
- Pt net electrodes, Au seal rings, manual, storage case, stand and more.





Measurement setups

Assembling the ProboStat™ for measurements is similar in all methods, with only minor replacement of accessories

Measurement setup; Disk sample

Here we demonstrate setting-up of the simplest 2-point conductivity method with two electrode contact pairs from the outer chamber.



A. Mount inner gas supply tube and sample support tube .



B. Connect an electrode contact pair for the lower electrode.



C. Place sample equipped with two centered electrodes.



D. Connect an electrode contact pair for the upper electrode. Attach control thermocouple.



E. Mount spring-force assembly and the outer gas supply tube.

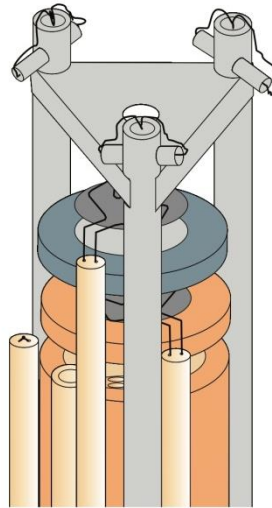
Measurement setup; Disk sample

Schematic illustration of assembly of disk sample measurements

Setup 1

2-electrode conductivity & impedance spectroscopy using two outer electrode contacts

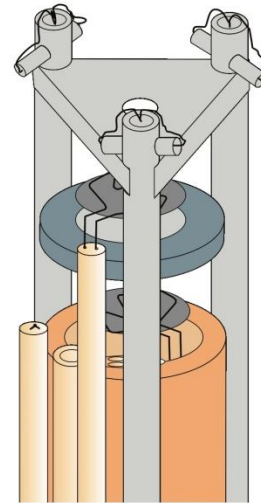
2-point conductivity method with two outer "hand" electrode contacts (only top part of the cell is shown).



Setup 2

2-electrode conductivity & impedance spectroscopy using inner and outer electrode contacts

Instead of using the outer electrode connection for the lower electrode, you may use the inner electrode connection for 2-electrode conductivity measurements.



Setup 2 is changed into the setup 3 by using gold gasket for sealing sample on the support tube edge.

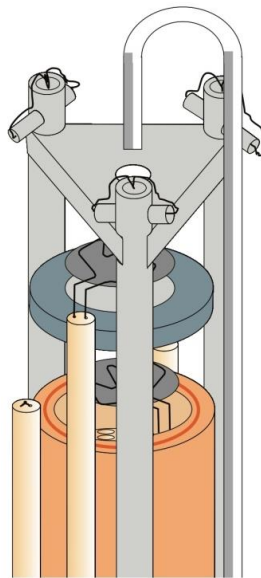
Measurement setup; Disk sample

Schematic illustration of assembly of disk sample measurements

Setup 3

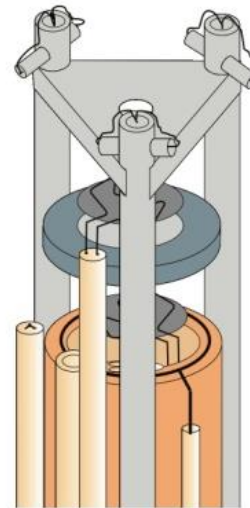
2-electrode conductivity & impedance spectroscopy, transport number measurements by emf of concentration cell or fuel cell testing with seal

For the concentration cell emf transport number measurements, or fuel cell testing: seal the setup 2 on the support tube edge. The seal and the sample separate the inner and outer gas compartments in the cell.



Setup 4

2- and 3-electrode conductivity / impedance spectroscopy / voltammetry with guard ring



A guard electrode contact used in the combination with the setup 2 may be used for 2-point conductivity measurements on disk sample with surface guard, electrode impedance

studies with ring reference electrode – symmetrical cell, and voltammetry studies with ring reference electrode – symmetrical cell. The guard electrode contact can also have sealing function.

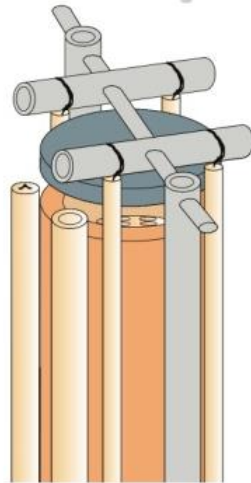
Measurement setup; Disk sample

Schematic illustration of assembly of disk sample measurements

Setup 5

Van der Pauw 4-point conductivity measurements on disk sample

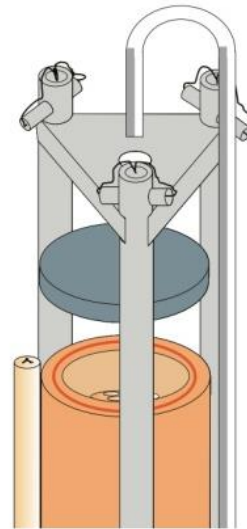
The van der Pauw method requires specially designed electrode contacts and two-rod spring-force assembly, but the basis of the setup is the same as for the above-listed setups.



Setup 6

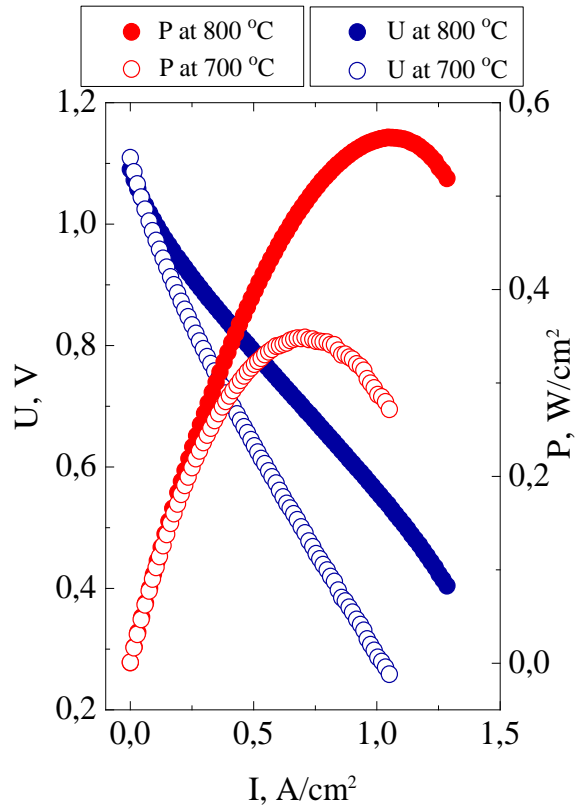
Permeability measurements for use with GS or MC

In typical permeability experiment, a disk sample is sealed gas tight over the support tube.



Measurement setup; Disk sample

Example: Fuel cell test (setup 3)



Fuel cell performance for:
wet H_2 , Pt | ASC2 | Pt, dry air

Fuel Cell:

ASC2

anode: porous NiO/8YSZ

electrolyte: dense 8YSZ

cathode: porous LSCF

Fuel: wet H_2 ;

Oxidant: dry air;

Gas flow rate 30 ml/min.

Sealing:

Gold gasket

$\text{OCV}_{\text{theor}} = 1.102 \text{ V}$

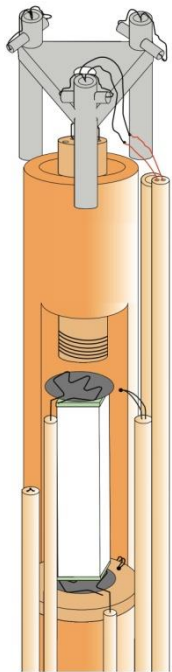
$\text{OCV}_{\text{exp}} = 1.091 \text{ V}$



SOFC test with the ProboStat™
and FCMix – a simple gas mixer
for fuel cell research (University
of Oslo)

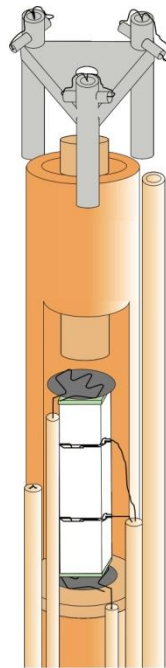
Measurement setup; Bar sample

Schematic illustration of assembly of bar sample measurements



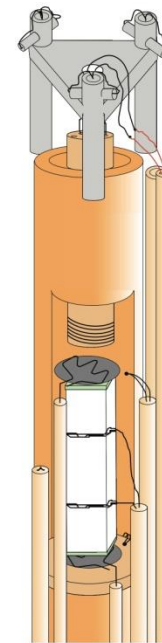
Setup 7

Seebeck coefficient measurements on bar sample using two "hand" electrode connects and internal heater



Setup 8

4-point conductivity measurements on bar sample with wrapped electrodes using two "hand" electrode connects and two general purpose electrode connects



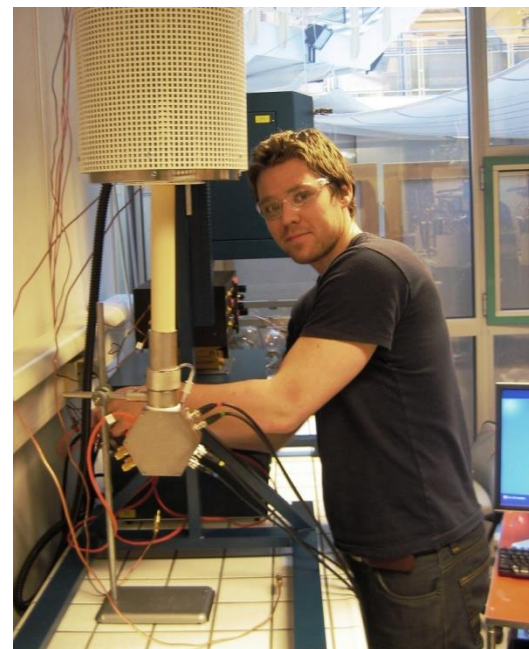
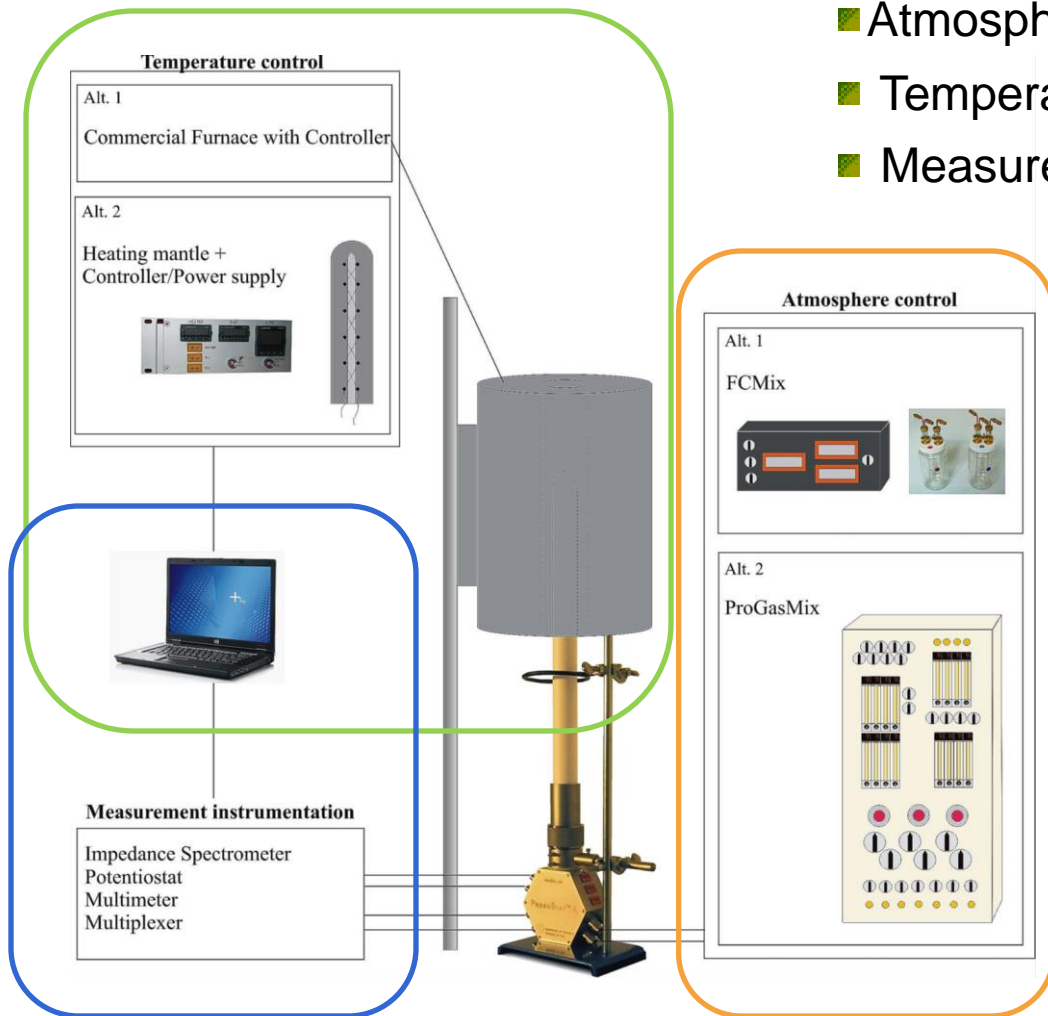
Setup 9

Combined measurement of Seebeck coefficient and 4-point conductivity measurements on bar sample with internal heater

What are typical components in a setup
for materials characterisation
with the ProboStat™?

WE CAN RECOMMEND AND DELIVER

- Atmosphere control
- Temperature control
- Measurement instrumentation



Atmosphere control

ProGasMix

A versatile manual rotameter-based gas mixer that selects and mixes gases from a range of connected input gases.

- Up to 7 input gases
- Selection of 3 input gases for mixing
- 2 individual mixtures routable to 4 outlets
- Wetting and drying stages



FCMix

A simple gas mixer for gas supply and humidification for fuel cell test

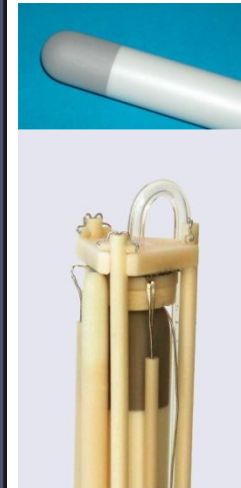


- Suitable for button-sized fuel cell tests
- Input gases: air, Ar, H₂ (other by order)
- No main power needs

Combined humidifier and overpressure control unit is included.

Oxygen sensor

Closed YSZ tube with or without Pt electrodes suitable for use as combined oxygen sensor and sample support tube.



We may also deliver a special top flange feedthrough for insertion of an 8 mm diameter commercial YSZ-based oxygen sensor.

Temperature control and measuring instrumentation

Furnaces

Custom made: a) furnaces for the ProboStat™ from Elite Thermal Systems and b) heating mantles from Glas-Col; or standard furnaces from Lenton, Entech, etc.

Measuring instrumentation

The ProboStat™ works equally well with all instrumentation such as impedance spectrometers, potentiostats, and multimeters from Solartron, Novocontrol, Autolab, PAR, Gamry, HP/Agilent, etc.

Power supply and controller

We offer custom made, functional power supplies with Eurotherm controllers. One important part of the product is the possibility to use more than one thermocouple, e.g. one in the sample holder and one in the heater.

Software

NORECS has developed software for:

- Calculating gas mixer outputs
- Continuously controlling, monitoring and interpreting electrochemical measurements, using one or more ProboStat™ cells.

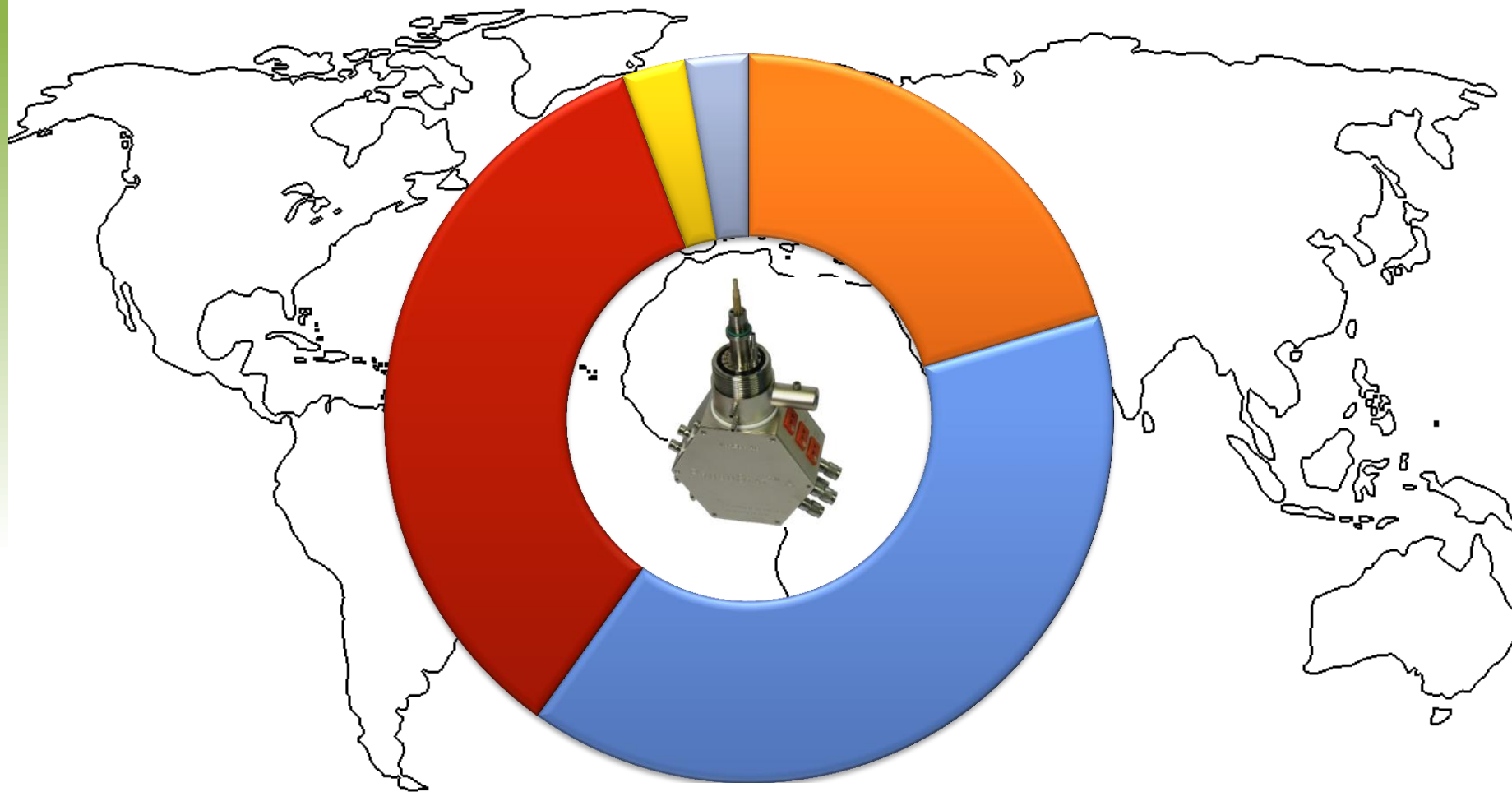
Summary

- The ProboStat™ is a versatile sample holder cell system for electrical and other characterisation at high temperatures and under controlled atmosphere;
- The ProboStat™ is designed for:
 - Disk and bar geometries; 2, 3, or 4 electrodes;
 - Temperature range RT – 1600°C;
 - All atmospheres: Oxidising, reducing, wet, dry, inert, corrosive, vacuum, overpressure;
 - Mini-contacts and spring loads for replacement of setups and samples;
 - Large range of electrical methods;
 - Superior lifetime and user economy.
- The ProboStat™ is compatible with all instrumentation;
- Each ProboStat™ system may be designed specifically for the customers needs;
- We can supply gas mixers, furnaces, measuring equipment and other ProboStat™ related products.
- We take pride in providing technical support, advice, training, and troubleshooting help directly to the customer.



ProboStat™ worldwide

■ USA ■ Europe ■ Asia ■ Canada ■ South America



NORECS AS

NORECS



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