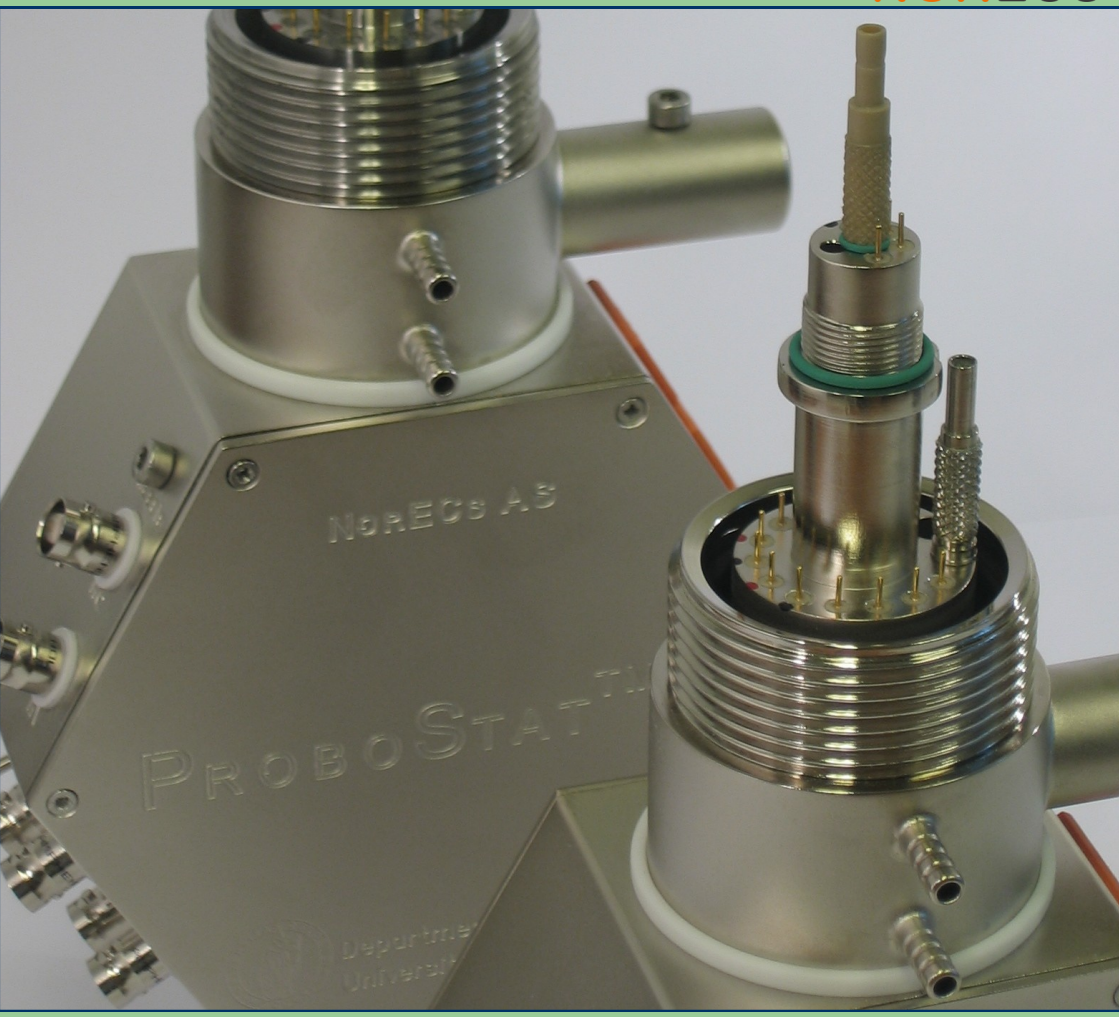
ProboStat™

NORECS



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

NORECS

NORECS AS develops and manufactures equipment and services for high-temperature electrochemical research.

NORECS was established in 2001, based on knowledge and equipment developed in the Kofstad – Norby research group at the University of Oslo. The sales and range of products and services have steadily grown since then. Through a network of representatives, our products are now in use in universities, institutes, and industries in most major countries in America, Asia, and Europe.

NORECS currently manufactures

- ProboStat™ - a versatile system for testing small button or bar samples
- ProGasMix FC – a full rotameter-based gas mixer
- FCMix – a simple and safe gas control unit for fuel cell tests
- Furnace control software
- Measurement software
- Samples and targets
- Custom made accessories

In addition, NORECS can supply:

- Furnaces
- Measuring equipment
- Sample preparation labware
- Oxygen sensors for ProboStat™ integration

We take pride in providing thorough technical support, advice, training, and trouble-shooting help directly to the customer.

ProboStat™

The main product is 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.

The ProboStat™ was developed at the University of Oslo for high-temperature solid-state electrochemical research, and has been in use for more than 30 years. The design has been continuously developed for increased quality, versatility, lifetime, and economy.

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 10-50 mm long bar Tubular (optional)
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

One cell - many applications

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, etc.
- DC, AC, impedance spectroscopy
- Dielectric properties, loss, etc.
- Disk, van der Pauw, and bar geometries
- 2, 3, and 4 electrodes
- Ionic transport number
- Proton transport number
- H/D isotope effects
- Seebeck coefficient
- I-V-characteristics
- Fuel cell components and single (button) cell testing
- Electrode kinetics
- Electrochemical pumping, gas permeation, and electrocatalysis with gas analysis (e.g. GC or MS) on outlets
- Sensor testing
- Poling of ferroelectrics possible with the high-voltage (kV) version
- Annealing and sintering under controlled atmospheres.
- Electrolysis
- In-situ oxygen sensor

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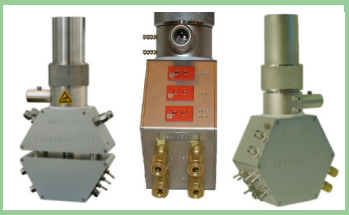
ProboStat™ base unit

The central unit in the ProboStat™ system is the base unit assembly, made of Ni-plated brass or stainless steel



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 Swagelok quick-connects for the two gas chambers
- cooling/heating water hose fittings

Optional:

- High-temperature tolerant version, 165 °C max. Stainless steel construction with gas supply system separated from the electrical connections. Prevents water condensation or carbon precipitation in the internal ProboStat gas lines.

ProboStat™ accessories

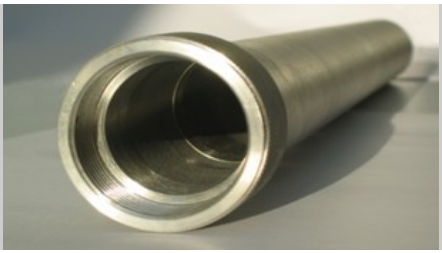
The ProboStat™ system has a wide range of accessories (metals, ceramics, and consumable parts) designed for different types of electrochemical measurements

Sample support tube assemblies

Standard alumina support tube assemblies presently come in the standard designs for 10-24 mm diameter disks and for bar samples. Other materials may be used for special purposes. In addition, special designs for different sample geometries or uses may be made upon order, or assembled by the user.

Outer enclosing tubes

These are 40 mm outer diameter closed tubes of alumina or silica with standard length 60 cm. Use of metals (e.g. stainless steel or superalloys) can give better shielding against noise and improve safety with respect to explosive gases and overpressures, but at the cost of temperature tolerance.



Spring load assembly

The spring load system keeps sample and electrodes in contact and position.

Electrode contact leads

The electrode contact leads for electrical measurements come in a variety of types. Many of them are issued in 2-wire pairs: one for current and one for voltage. The standard material is high purity Pt. Other metals may be applied by us or the user.

Thermocouples

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

Gas supply tubes

These are to be used inside the cell to supply gases to the lower/inner and upper/outer chambers of the cell. They send the gas directly to the sample area, while extraction of the gas takes place at the bottom of each chamber.

A range of sealing materials

Gold or Silver gaskets, Thermiculite.

Top flange and open end outer tube

Open end outer tubes with flange for evacuation and optical access to disk sample top surface/electrode. The optical window may be used for pyrometry, interferometry, luminescence and optical spectroscopy.

Liquid cell assembly

to measure dielectric constant and conductivity of liquids at high temperature.

Crucible holder

Let you use the ProboStat™ to perform annealing of powders in controlled atmospheres.

Internal heater

For creating temperature gradients in Seebeck coefficient measurements.

High-pressure tolerant enclosing tube

Tolerates max. 15 bars. To be mounted outside an alumina enclosing tube (ref. picture).

Standard ProboStat™ packages

Extensively furnished version:

- 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.

Normal version:

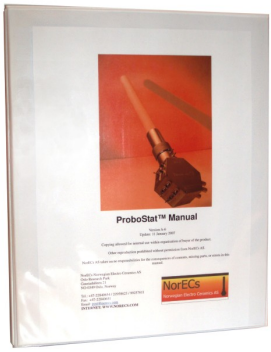
- Disk samples of up to selected diameter, e.g. 12 or 20 mm
- 2 and 3 electrode setups
- Conductivity, impedance spectroscopy, DC measurements
- Concentration cells; transport number, permeability, fuel cell testing, etc.

Normal plus version:

- Contains a normal system plus an extra supply of some commonly consumed spare parts
- Disk samples of varying diameters
- 2 and 3 electrode setups
- Conductivity, impedance spectroscopy, DC measurements
- Concentration cells; transport number, permeability, fuel cell testing, etc.

High voltage normal version:

- Contains a normal system, but with a high voltage (10 kV) base unit
- Polarization of dielectrics, breakdown tests, plasma electrodics etc. using 2 electrodes.
- Disk samples of up to selected diameter, e.g. 12 or 20 mm
- 2 and 3 electrode regular setups
- Conductivity, impedance spectroscopy, DC measurements, etc.
- Concentration cells; transport number, permeability, fuel cell testing, etc.



Minimum version:

Contains base unit and some essentials such as sample support tube, outer tube, and spring load. It requires separate purchase or own fabrication of thermocouple and electrode contacts depending on the methods to be used.

Base unit system:

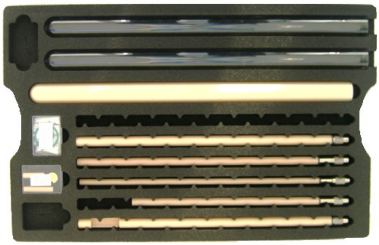
Contains only the base unit and a minimum of sockets to mount your own furnishment in the cell. An option for those who want to make their own accessories and connections from scratch. It may also serve those who want to equip an earlier package with one or more units to increase measurement throughput and make better use of all accessories.

Custom system:

If none of the packages above corresponds exactly to what you want, we can recommend and assemble a system designed specifically for your needs. Please contact us or use the enquiry form found on our webpage www.norecs.com.

Example: Extensively furnished version

Aluminium transport and storage case



Middle layer

- Electrode contacts GP2, GP1, H2N#, H1B, H1T, INH2N#, GR#, VDP2
- Thermocouples TCC/D, TCC/B, TCT, TCB
- Long alumina bars for spring load
- Outer gas tubes
- Inner multibore gas tube
- Alumina caps for thermocouples

Top layer

- Alumina and silica enclosing tubes
- Sample support tube assemblies: up to four for disk samples with different diameter and one with slit for bar sample mounting
- "Floor" and "roof" for bar sample mounting
- Gas connection stems

Bottom layer

- ProboStat™ base unit
- 4 single coax/BNC cables
- 3 thermocouple compensation cables
- Simple bench-top mounting stand set
- Various tools and small parts

NORECS AS, Oslo Science park, Gaustadalleen 21, NO-0349 Oslo, Norway

ProboStat™: systems and measurements

Sample	Disk	Disk	Disk	Disk	Disk	Disk[a]	Disk[a]	Disk[a]	Disk	Bar	Bar	Bar	Disk / Powder[b]
Method / ProboStat™ system	2-point impedance spectroscopy and conductivity	2-point conductivity with surface guard	Electrode impedance studies with ring reference electrode – symmetrical cells	Voltammetry studies with ring reference electrode – symmetrical cells	Van der Pauw 4-point conductivity	EMF transport number	Permeability	Fuel cell and electrolyzer components and single cell	Poling of ferroelectrics	Seebeck coefficient (thermoelectric power)	4-point conductivity	Comined Seebeck coefficients and conductivity	Annealing / sintering under controlled atmospheres
Extensively furnished system	X	X	X	X	X	X	X	X		X	X	X	X
Extensively furnished HV system	X	X	X	X	X	X	X	X	X	X	X	X	X
Normal plus system	X	X	X	X	X	X	X	X		(X)	(X)	(X)	X
Normal system	X	X	X	X	X	X	X	X		(X)	(X)	(X)	X
Normal HV system	X	X	X	X	X	X	X	X	X	(X)	(X)	(X)	X
Bar sample system	(X)	(X)	(X)	(X)	(X)	(X)	(X)	(X)		X	X	X	(X)
ACIS system	X	(X)	(X)	(X)	(X)	(X)	(X)	(X)		(X)	(X)	(X)	(X)
Tubular membrane system	X[c]	(X)	(X)	(X)	(X)	X[d]	X[d]	X[d]		(X)	(X)	(X)	(X)
Minimum system	(X)	(X)	(X)	(X)	(X)	(X)	(X)	(X)		(X)	(X)	(X)	(X)
Base unit system	(X)	(X)	(X)	(X)	(X)	(X)	(X)	(X)		(X)	(X)	(X)	(X)

(X): Available after fabrication or purchase of ProboStat accessories and connections.

a: Requires user sealing

b: Requires suitable crucible

c: Tube sample

d: Closed tube sample

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Typical components in a setup for materials characterization with ProboStat™

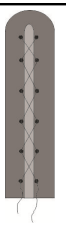
Temperature control

Alt. 1

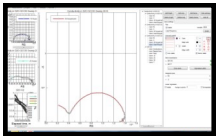
Commercial Furnace with controller

Alt. 2

Heating mantle +
Controller/Power supply



Software for Measurement / Temperature control



Measurement instrumentation

Impedance Spectrometer
Potentiostat
Multimeter
Multiplexer

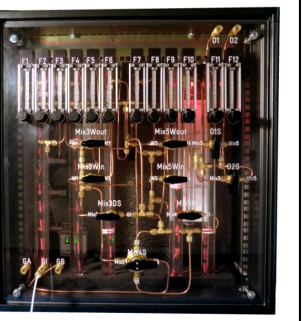
Atmosphere control

Alt. 1

FCMix / Probble

Alt. 2

ProGasMix FC



Measurement setup; Disk samples

2-, 3-, 4-electrode, and gas permeation measurements

Assembling the ProboStat™ for measurements on a disk sample is similar in all methods, with only minor replacements of accessories.

A. Mount inner gas supply tube and sample support tube. Ensure gas tube extends 1 mm over the rim of the support tube.

B. Connect an electrode contact pair H2N# for the lower electrode.

C. Place sample equipped with two centered electrodes.

D. Connect electrode contact pair H2N# for the upper electrode. Attach control thermocouple TCC/D.

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

2-electrode measurements

Here we demonstrate setting -up of the simplest 2-point conductivity method with two H2N# "hand" electrode contacts (only top part of the cell shown, also see Fig.1).

Instead of using the outer electrode connection for the lower electrode in step B, you may use the inner electrode connection for 2-electrode conductivity setup (Fig. 2). In this case you may do concentration cell emf transport number measurements, or fuel cell testing: seal the setup 2 using a gold gasket on the support tube edge before step C. (Fig. 3). The seal and the sample separate the inner and outer gas compartments in the cell. If the sample is smaller than the support tube, add a support plate after step A.

3-electrode measurements

A guard electrode contact used in the combination with the setup 2 (Fig. 4) may be used for 2-point conductivity measurements on disk sample with surface guard (third electrode is optional), 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.

4-electrode measurements

The van der Pauw method requires specially designed electrode contacts VDP2 and two-rod spring-force assembly, but the basis of the setup is the same as for the above-listed setups (Fig.5).

Gas permeation

In typical permeability experiment, a disk sample is sealed gas tight over the support tube (Fig. 6).

Measurement setup; Disk samples

Schematic illustration of assembly of disk sample measurements

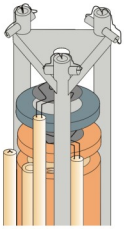
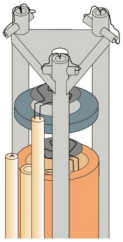


Fig. 1. Setup 1: 2-electrode conductivity & impedance spectroscopy using two H2N# electrode contacts.

Fig. 2. Setup 2: 2-electrode conductivity & impedance spectroscopy using H2N# and INH2N# electrode contacts.



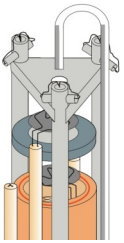


Fig. 3. Setup 3: transport number measurements by EMF of consentration cell or fuel cell testing with seal, H2N# and INH2N# electrode contacts.

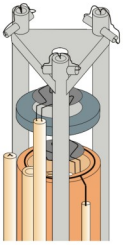
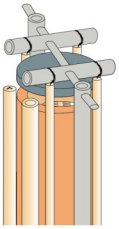


Fig. 4. Setup 4: 2– and 3-electrode conductivity/ impedance spectroscopy/ voltammetry with guard ring, H2N# and INH2N# electrode contacts.

Fig. 5. Setup 5: van der Pauw 4-point conductivity measurements on disk sample using two VDP2.



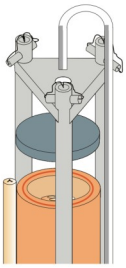


Fig. 6. Setup 6: permeability measurements for use with GC or MS.

Measurement setup; SOFC button cell test

Example of setup schematic from the ProboStat™ manual

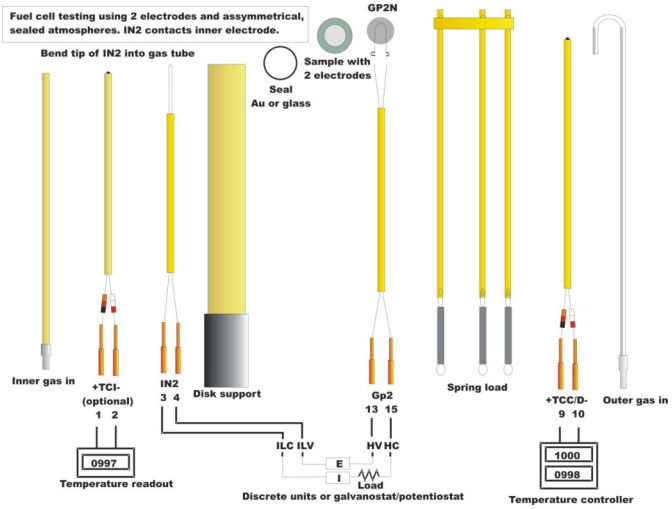
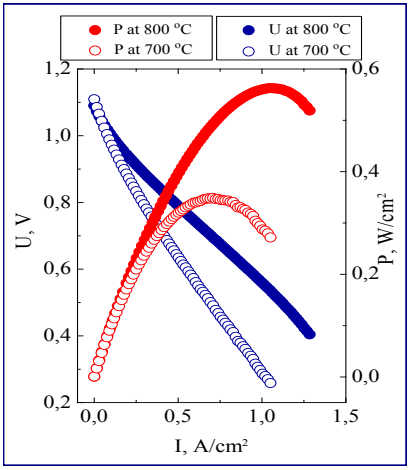


Fig. 7. Fuel cell testing using 2 electrodes and asymmetrical, sealed atmosphere, IN2 inner electrode contact assembly (example).

Use a support tube assembly of suitable diameter and mount inner gas tube, inner electrode contact and seal before placing the sample. For contacting the upper electrode, use H2N# or a combination of GP2/GP2N#. Complete the setup by mounting the thermocouple, spring-load assembly and outer gas supply tube.



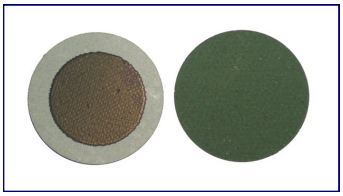


Fig. 9. Commercial button fuel cell ASC2: anode supported cell with 8YSZ electrolyte, porous nickel cermet anode and porous lanthanum cobaltite cathode.

Fig. 8. Fuel cell performance for: wet H_2, Pt | ASC2 | Pt, dry air .

Measurement setup; bar sample

Seebeck coefficient and 4-point measurements

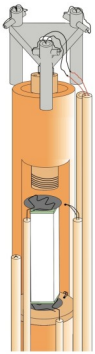


Fig. 10

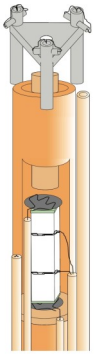


Fig. 11

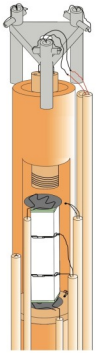


Fig. 12

Fig. 10. Measurement of Seebeck coefficient on bar sample using H1T and H1B electrode connects and internal heater.

Fig. 11. 4-point conductivity measurements on bar sample with wrapped electrodes using H1T, H1B, and GP2 electrode connects.

Fig. 12. Combined measurement of Seebeck coefficient and conductivity on bar sample using H1T, H1B, and GP2 electrode connects and internal heater.

Seebeck coefficient measurements (fig. 10)

Connect the bottom thermocouple (TCB) before mounting the bar sample support tube. Place the "floor" at the bottom of the sample slit and then place the thermocouple tip on top of the floor plate. Connect the sample ends using one short and one long single-wire hand contact assembly (H1B and H1T). Complete the setup by mounting the upper thermocouple (TCT), "roof", spring load assembly, control (middle) thermocouple (TCC/B) and outer gas supply tube.

4-point conductivity measurements (fig. 11)

Connect the two current leads to the end electrodes by using one short and one long single-wire hand contact (H1B and H1T) (fig. 11). For the two voltage probes, use a GP2 with two thin Pt wires wrapped around the sample e.g. 1/3 and 2/3 down its length. Complete the setup by mounting TCC/B, "roof" and spring load assembly.

Combined measurements of Seebeck coefficients and conductivity (fig. 12)

Mount the sample as described for Seebeck coefficient measurements. In addition, a GP2 with thin Pt wires is used for the last two (voltage probe) connections.

Seebeck measurements at increasing gradient can be done by moving the cell stepwise out of the middle of the hot zone of the furnace, while keeping the average temperature constant. Alternatively, we can deliver a modified "roof" with Pt10Rh wire wrapped around the lower part (Internal Heater). By sending current through the wire, a temperature gradient is achieved, enough for doing Seebeck measurements without physically having to move the cell (figs. 10 and 12).

Gas mixers

NorECs is a long term expert in gas flow control and measurements. Our expertise include atmosphere control with mass flow controllers (through USB) and variable area flowmeter mixers (rotameters). Amongst our products sold worldwide are custom built gas mixers, gas handling systems and measurement/calculation software. We offer help with product selection, design and assembly of tailor made systems, based on high-quality Swiss-made Vögtlin products.

FCMix

Simple gas mixer, humidification and overpressure control for small fuel cells.

FCMix is a simple and low cost gas mixer that controls the flows of fuel, oxidant, and one inert gas that can be routed for dilution of the fuel or oxidant for flushing, soft start, slow SOFC anode reduction, and safe operation, as well as tests of gas diffusion limitations. It is compact and easy to place in a 19-inch rack or directly on the lab bench. Two "Probble" combined humidifier and overpressure control units are included – one for each cell chamber. These do humidification, overpressure relief, and fine pressure control in and between the two cell cambers, all in one simple unit that for additional safety contains only pure water and no glass.

ProGasMix FC

Versatile rotameter-based manual gas mixer

ProGasMix FC is a unique tool for students and researchers in materials science, suitable for:

- Fuel cell tests, with humidification of fuel and/or oxidant
- Gas permeation measurements
- Transport number measurements
- Conductivity measurements as a function of pO_2, pH_2O, pH_2, etc.

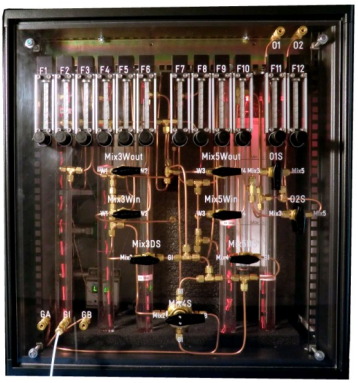
The mixer includes safety features for overpressure (gas blockage). Partial pressures are calculated from flowmeter-readings via accompanying software.
It offers wide range control of partial pressures, and includes humidification and drying stages and dual mixture output.

ProGasMix FC makes gas mixture from three input gases. A second mixture can be modified from the first for concentration cell measurements. pH_2O can be set individually in the two mixtures. The gas mixer is suitable for use with the ProboStat™ or other systems using variable controlled atmospheres, and offers the economy, simplicity, and insight to those that value versatility and wide ranges of mixing.
Partial pressures are calculated from flowmeter readings via accompanying software.

Custom gas control system

Tailor made systems for any customer application.

- Pressure control
- Dilution
- Compact enclosures
- Wetting/drying of gas
- Computer control



Other ProboStat™ related products

Furnaces and heating mantles

We can recommend and deliver furnaces custom made for the ProboStat™ from Elite Thermal Systems or standard furnaces from Lenton, Entech, etc. We can also deliver custom made tubular heating mantles from Glas-Col suitable for temperatures from ambient up to 650°C.

Measuring instrumentation

The ProboStat™ is a passive unit with all required electrode connects and shields and thus works equally well with all instrumentation such as impedance spectrometers, potentiostats, and multimeters from Solartron, PAR, Novocontrol, Autolab, Gamry, HP/Agilent, etc.

Software

NORECS offers Omega measurement software and Omega Temp software for furnace control. Omega measurement software includes and allows to:

- Control instruments such as impedance spectrometers, rotameters, multimeters, furnaces, etc.
- Control and read external equipment such as mass flow controllers, pressure regulators, and other measurement instruments with Modbus or GPIB protocol support.
- Use multimeters to measure sensors and gauges that have analog voltage or current output.
- Include functions to interpret the read values such as thermocouple voltage
- Plot on screen and save to disk the measured data

Samples and targets

NORECS delivers samples of selected materials suitable for demonstration, education, training, and research using ProboStat™ measurement cells. Examples: BZY15, BZY15-Ni, 8YSZ, SOFC single cell MEAs and PLD targets. Available with/without Pt electrodes.

Sample preparation labware

NorECs offers equipment and consumables for ceramic sample preparation from well-known manufacturers.

Measurement services

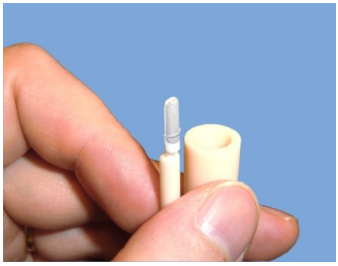
NorECs offers measurement services at elevated temperatures.

Feedthrough repair kit

Feedthrough repair kit for on-site replacement of the ProboStat™ electrical minicontact feedthroughs.

Oxygen sensors

We can now deliver MicroPoas oxygen sensor (Setnag, France), mounted ready for use in any standard ProboStat unit. MicroPoas has built-in metal reference so no reference gas is required. It has high resistance to thermal shocks. This miniaturized sensor (diameter 3mm, length 10mm) is suitable for measuring continuously partial pressure variation from 10^{-35} to 10 atm. Working temperature for the standard version is 500°C to 800°C; high temperature version 600°C to 1050°. Both are made with S–type thermocouple. Instrumentation for reading the signal and measuring the temperature is optional. >1000 Mega-ohm input impedance is required. Picture below shows the tip of the MOSE sensor next to a ProboStat sample support tube.



We may also deliver closed YSZ tubes—with or without Pt electrodes, suitable for use as combined oxygen sensor and sample support tube.

We have representatives worldwide

Contact us for details

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