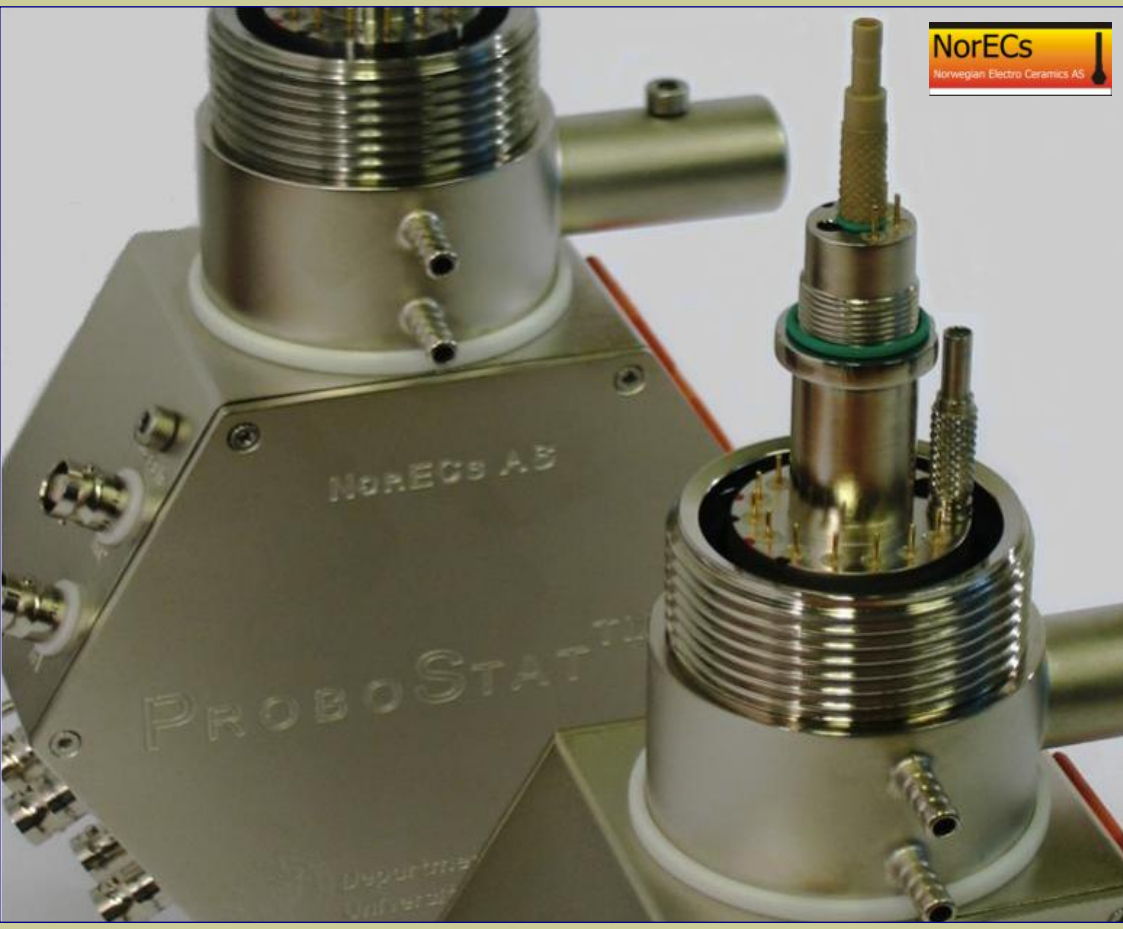
ProboStat™



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

NorECs AS

NorECs Norwegian Electro Ceramics 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 – a full rotameter-based gas mixer
- FCMix – a simple and safe gas control unit for fuel cell tests
- power supply and temperature controller for heating mantles and small furnaces
- measurement software
- test samples
- custom made accessories

In addition, NorECs can supply:

- furnaces
- measuring equipment.

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 20 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 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 (25 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.

ProboStat™ base unit

The central unit in the ProboStat™ system is the base unit assembly, normally made of Ni-plated brass for good heat conduction (stainless steel option for use with corrosive gases)

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

ProboStat™ accessories

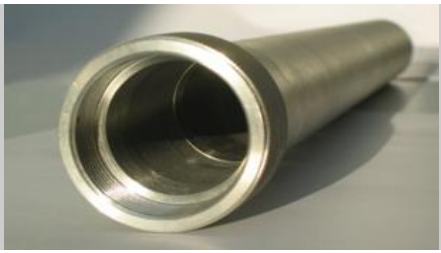
The ProboStat™ system has a wide range of accessories (metals, ceramics, and consumable parts) designed for different types of electrochemical measurements

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.

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 and sample plate holder

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

Internal heater

For creating temperature gradients in Seebeck coefficient measurements.

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.

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 Norwegian Electro Ceramics AS, Oslo Research park, Gaustadalleen 21, NO-0349 Oslo, Norway

Schematical parts list for various methods

	Methods / Accessories	Outer tube, alumina or silica[1]	Support tube assembly for disk sample[2]	Support tube assembly for bar sample	Inner gas tube[3]	Outer gas tube, alumina or silica[1, 4]	Three-rod spring force assembly	Two-rod van der Pauw spring force assembly	TCI[5]	TCC/D[5]	TCC/B[5]	TCT[5]	TCB[5]	H2N#	H1T	H1B	INH2N#[6]	GP2	VDP2	GR#	Gold gaskets	"floor" support plate, "roof" short alumina rod
1	2-electrode impedance spectroscopy and conductivity measurements on disk sample (option 1)	×	×		×	×	×		o	×				×			×					
2	2-electrode impedance spectroscopy and conductivity measurements on disk sample (option 2)	×	×		×	×	×		o	×				××								
3	2-electrode conductivity measurements on disk sample with surface guard	×	×		×	×	×		o	×				×			×			×		
4	van der Pauw 4-point conductivity measurements	×	×		o	×		×	o	×									××			
5	4-point conductivity measurements on bar sample	×		×		×	×				×				×	×		×				×
6	Electrode impedance and voltammetry studies on disk sample with reference electrode – symmetrical cell	×	×		×	×	×		o	×				×			×			×		
7	EMF transport number measurements	×	×		×	×	×		o	×				×			×				×	
8	Seebeck coefficient (thermoelectric power) measurements	×		×		×	×				×	×	×		×	×						×
9	Combined Seebeck coefficient and 4-point conductivity measurements	×		×		×	×				×	×	×		×	×		×				×
10	Fuel cell and electrochemical reactor – asymmetrical cell	×	×		×	×	×		o	×				×			×				×	
11	Permeability measurements – asymmetrical cell	×	×		×	×	×		o	×											×	

× - requered, o - optional

Notes

[1] Silica enclosing and gas tubes are long-term stable to 900 °C, and can be used up to around 1100 °C for short term. Each cooling below 400 °C of silica parts reduces lifetime.
[2] Support tube assemblies for disk sample are produced with outer diameter 10, 12, 16, 20 or 24 mm.
[3] For support tubes with outer diameter 16, 20, and 24 mm a multi-bore inner gas tube is possible and recommended. For 10 and 12 mm supports only a simple 3 or 4 mm gas tube may be used.
[4] Straight alumina or bent silica.
[5] K- or S-type thermocouples.
[6] For support tubes with outer diameter 16, 20, and 24 mm IN2N# is recommended, for 12 mm or less - IN2.
[7] All parts can be also manufactured by users after instructions in the manual.

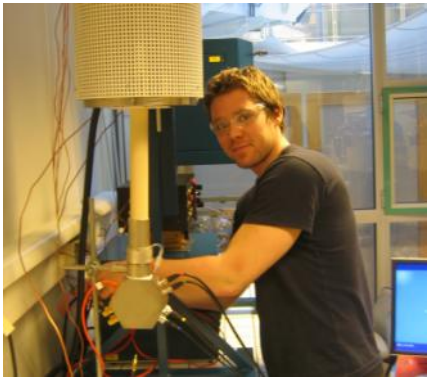
Abbreviations

TCI – inner thermocouple assembly
TCC/D – disk sample control thermocouple assembly
TCC/B – bar sample control thermocouple assembly
TCB – bottom thermocouple assembly for Seebeck coefficient measurements
TCT – top thermocouple assembly for Seebeck coefficient measurements
H2N# – electrode "hand" contact assembly, outer, 2-wire, with net
H1T – electrode "hand" contact assembly, outer, 1-wire, top
H1B – electrode "hand" contact assembly, outer, 1-wire, bottom
INH2N# – electrode "hand" contact assembly, inner, 2-wire, with net
IN2 – electrode contact assembly, inner, 2-wire for 10and 12 mm support tubes
GP2– electrode contact assembly, general purpose, 2-wire
GP1– electrode contact assembly, general purpose, 1-wire
VDP2 – van der Pauw contact assembly
GR# – guard ring
GP2N# – electrode net with 2 contacts

Possible electrode replacements

Electrode	Replacements
H2N#	GP2 + GP2N#
GP2	2 x GP1

Typical components in a setup for materials characterization with the ProboStat™



Temperature control

Alt. 1

Commercial Furnace with Controller

Alt. 2

Heating mantle +
Controller/Power supply

Measurement instrumentation

Impedance Spectrometer
Potentiostat
Multimeter
Multiplexer

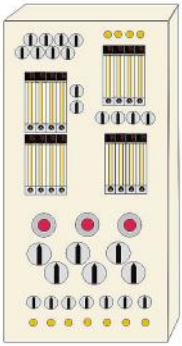
Atmosphere control

Alt. 1

FCMix

Alt. 2

ProGasMix



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

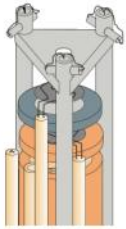


Fig. 1. Setup 1: 2-electrode conductivity & impedance spectroscopy using two H2N# electrode contacts.

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

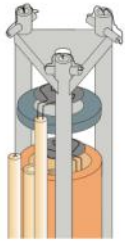


Fig. 3. Setup 3: transport number measurements by EMF of consentration cell or fuel cell testing with seal, H2N# and INH2N# electrode contacts.

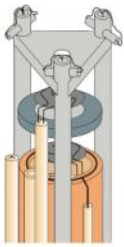
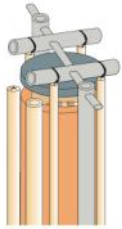


Fig. 4. Setup 4: 2– and 3-electrode conductivity/ impedance spectroscopy/ voltammetry with guard ring, H2N# and INH2N# electrode contacts.

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



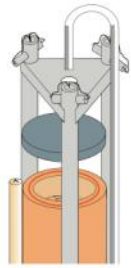


Fig. 6. Setup 6: permeability measurements for use with GC or MS.

Measurement setup; SOFC button cell test

Example of setup schematic from the ProboStat™ manual

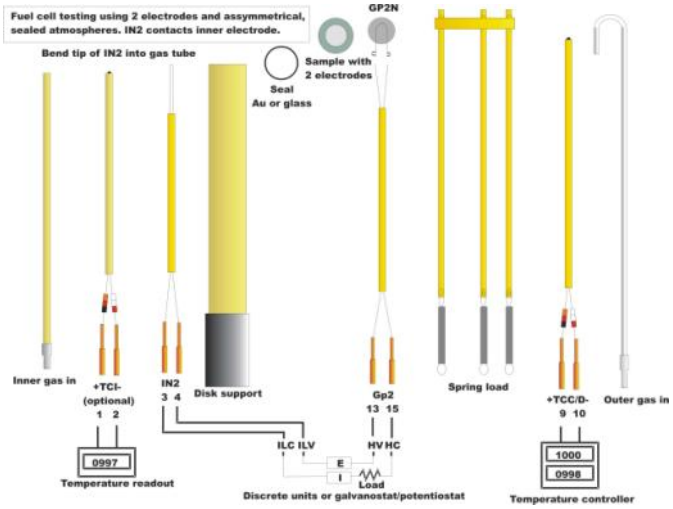


Fig. 7. Fuel cell testing using 2 electrodes and asymmetrical, sealed atmosphere, IN2 inner electrode contact assembly (example).

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.

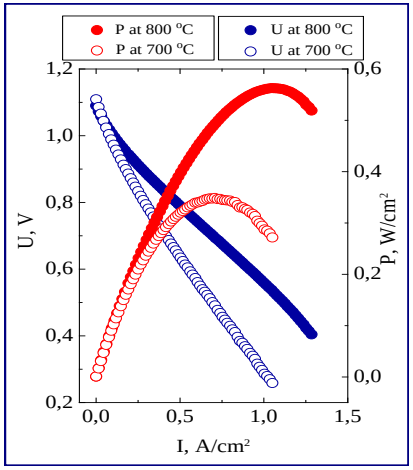


Fig. 8. Fuel cell performance for: wet H_2, Pt | ASC2 | Pt, dry air .

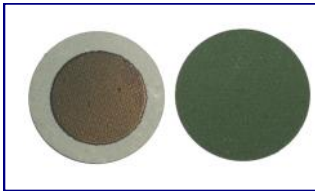
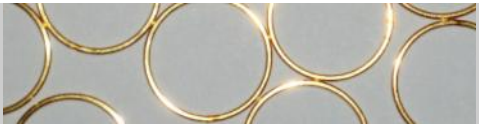


Fig. 9. Commercial button fuel cell ASC2: anode supported cell with 8YSZ electrolyte, porous nickel cermet anode and porous lanthanum cobaltite cathode.



Measurement setup; bar sample

Seebeck coefficient and 4-point measurements

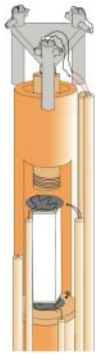


Fig. 10

Fig. 11

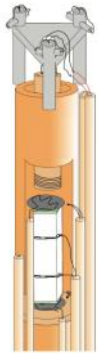


Fig. 12

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

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 to 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 newly designed "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.

"Probble" (pat. pend.)

Simple humidification, overpressure safety guard, and overpressure device.

The bubbler and pressure device supplied with FCMix are also sold separately.

ProGasMix

Versatile gas mixer for wide range control of partial pressures. Includes humidification and drying stages and dual mixture output.

The unique gas mixer developed at the University of Oslo is finally available also to others, in a compact, mobile and appealing unit. It has been used in countless studies of conductivity and other properties as a function of pO_2, pH_2O, etc., and is especially known for its ability to deliver two mixtures suitable for concentration cell measurements (e.g. for separating transport numbers of oxide ions and protons), permeation studies, and fuel cell component tests.

ProGasMix is a versatile manual rotameter-based gas mixer that selects and mixes three gases from a range of connected input gases. A second mixture can be modified from the first for concentration cell measurements. Both are humidified and dried and the two portions mixed to set pH_2O individually in the two mixtures. It is a unique tool for students and researchers in materials science, suitable for use with the ProboStat™ or other systems using variable controlled atmospheres. It 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.

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, ect. We can also deliver custom made tubular heating mantles from Glas-Col suitable for temperatures from ambient up to 650 °C.

Power supply and controller

We offer custom made, functional power supplies with state-of-the-art 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. This enables one to use the cell's thermocouple for accurate control at high temperatures, and the heater's thermocouple for stable heating at close to ambient temperatures. The second display can then monitor the actual sample temperature. An overtemperature guard can also be included, to be connected to the furnace thermocouple.

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 is currently developing software for calculating gas mixer outputs. We are also developing a program for continuously controlling, monitoring and interpreting electrochemical measurements, using one or more ProboStat™ cells or similar and industry standard impedance spectrometers, potentiostats, voltmeters, multiplexers, and temperature controllers.

Standard and custom made test samples

NorECs may deliver test samples of selected materials suitable for demonstration, education, training, and research using ProboStat™ measurement cells. The samples are disks with 0, 1, 2, or 3 Pt electrodes, or bars without electrodes or with Pt end-face electrodes.

Feedthrough repair kit

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

Oxygen sensors

We may deliver closed YSZ tubes—with or without Pt electrodes, suitable for use as combined oxygen sensor and sample support tube. We may also deliver a special topflange feedthrough for insertion of an 8 mm diameter commercial YSZ-based oxygen sensor from above the furnace.

We have representatives worldwide

For details visit *www.norecs.com*

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