ProboStat™ ACIS

ProboStat™ ACIS is a sample holder dedicated for AC conductivity measurements and 2-electrode impedance spectroscopy on small disk samples at high temperatures and under controlled atmospheres.

1. Introduction

The ProboStat™ is a well proven system suited for studying a wide range of materials (dielectric, ionic, semiconducting, metallic, etc.) and their interfaces (e.g. grain boundaries and electrodes). The ACIS setup was developed for facile mounting and replacement of test samples compared with the standard ProboStat™ furnishment.

The ProboStat™ ACIS is designed to operate from RT to 1600°C and at near-atmospheric total pressure, but can be pumped to low vacuum, or hold up to 15 bar. The setup is assembled in a single chamber mode and can be fed with virtually any gas.

The sample for the ACIS setup is typically a sintered disk maximum 15 mm diameter and height 10 mm. It is normally required that each sample face is equipped with a painted electrode.

The ProboStat™ ACIS setup is devoted to measure:

- AC conductivity and impedance spectroscopy vs T, pO_2, pH_2O, etc.
- Bulk, grain boundary and electrode impedance measurements
- H/D isotope effect
- Dielectric properties

For detailed methods description please see the ProboStat™ Manual (www.norecs.com).

The system is supplied with a standard ProboStat™ base unit and can be expanded for any sample geometries and measurements that ProboStat™ can support.

2. Contents and assembly

2.1. Contents

The ACIS setup comprises the following parts, illustrated in the figure below:

Parts for ACIS setup:

1. One alumina sample support tube assembly for disk samples with diameter up to 15 mm.
2. One lower "hand" electrode contact 2-wire assembly of Pt/alumina with Pt net (H2BN15).
3. One upper "hand" electrode contact 2-wire assembly of Pt/alumina with Pt net (H2TN15).
4. One control Pt/Pt10Rh thermocouple assembly for ACIS setup (TCC/ACIS-S).
5. One inner Pt/Pt10Rh thermocouple assembly for ACIS setup (TCI/ACIS-S).
6. One alumina cap for thermocouples.
7. One alumina roof for disk sample mounting.
8. One alumina floor disk for sample mounting.
9. One alumina support plate.
10. Three alumina spring force tubes with Pt/alumina locking part.
11. One alumina top triangle plate.
12. Three springs.

2.2. Assembly

Step 1: Insert the inner thermocouple (5) onto feedthroughs no. 1 and 2. Mind the polarity!

	
High temperature zone	Connections to the ProboStat™ base unit

Step 2: Place the cap (6) on top of the inner thermocouple.

	
High temperature zone	Connections to the ProboStat™ base unit

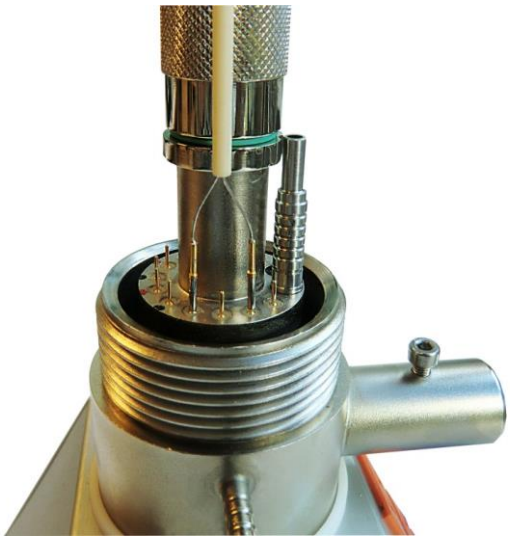
Step 3: Mount the sample support tube (1).

	
High temperature zone	Connections to the ProboStat™ base unit

Step 4: Place the floor disk (8) in the slit of the sample support tube. The top of the alumina cap is centred by the hole of the floor disk. The upper tip of the cap should now be in the same level as the floor disk surface.

	
High temperature zone	Connections to the ProboStat™ base unit

Step 5: Insert the lower "hand" electrode contact (2) onto LC and LV feedthroughs, no. 6 and 8 accordingly. Carefully bend the top part of the electrode.

	
High temperature zone	Connections to the ProboStat™ base unit

Step 6: Place the sample on the lower "hand" electrode contact.

	
High temperature zone	Connections to the ProboStat™ base unit

Step 7: Insert the upper "hand" electrode contact (3) onto HV and HC feedthroughs, no. 13 and 15 accordingly. Carefully bend the top part of the electrode contact.

	
High temperature zone	Connections to the ProboStat™ base unit

Step 8: Place the support plate (9) on the upper electrode.

	
High temperature zone	Connections to the ProboStat™ base unit

Step 9: Insert the roof for disk sample mounting (7) from the top of the sample support tube.

	
High temperature zone	Connections to the ProboStat™ base unit

Step 10: Optional: Insert the outer control thermocouple (4) onto feedthroughs no. 9 and 10, or no. 11 and 12. Mind the polarity!

	
High temperature zone	Connections to the ProboStat™ base unit

Step 11: Hook the springs (12) onto the lower part of the spring force tubes (10). Thread the tubes through the holes in the triangle top plate (11). Hook springs over the base unit pedestal.

	
High temperature zone	Connections to the ProboStat™ base unit

Step 12: Close the sample holder using the enclosing tube

Step 13: Make electrical and gas connections. For more information please see the ProboStat™ Manual, Chapter 8. The Manual can be downloaded at the NorECs web page: http://www.norecs.com

3. Instructions for fabrication of selected parts

3.1. Instruction for fabrication of thermocouple assemblies

3.1.1. General

3.1.1.1. Materials and equipment

The following instructions are valid for a standard cell using **type S (Pt/Pt10Rh) thermocouples**.

For each thermocouple you need:

Alumina capillary tube, 2-bore, 3 mm Ø, 50 cm long or ordered to length (Frialit-Degussit AL23 order number 143 11030 0500)

Pt wire, 0.5 mm Ø
Pt10Rh wire, 0.5 mm Ø

2 female feedthrough connectors

Insulation:

Red (Pt, -) and black (PtRh, +)
or
Blue (Pt, -) and white (PtRh, +)
or
White (Pt, -) and Orange (PtRh, +)
or
White (Pt, -) and Red (PtRh, +)

In addition you need:

Flat-nosed pliers
Solder iron
Solder tin
Tape measure or other length measure
Lab stand with test-tube or burette grip.

In order to check the polarity of the assembly you need a heat source (solder iron, hot air gun, or flame) and a DC voltmeter with mV range.

3.1.1.2. General procedures

In order to prevent contamination of samples, always wash your hands before working with parts that are to touch samples or otherwise will be present in the hot parts of the cell. Preferably wear gloves. We suggest you wash the exterior of parts after assembly using e.g. a suitable cloth and pure alcohol.

After assembly, check the polarity: Heat the tip of the thermocouple while measuring the terminal voltage. The Pt10Rh should become significantly positive. If you use the solder iron for this purpose, do not touch directly the thermocouple with the tool, and also ensure that the voltmeter can measure 0.1 mV's or better to detect the small heating obtained in this case.

3.1.2. Inner Pt/Pt10Rh thermocouple assembly for ACIS setup (TCI/ACIS-S)

3.1.2.1. Procedure

Cut alumina tube to 30.8 cm.
You may drill a hole for the thermocouple weld point in the tip of the tube, using a small diamond ball bore.
Thread Pt and Pt10Rh in the tube.
Weld.
Cut the wires to 32.1 cm.
Insulate 0.5 cm.
Solder to connectors.
Check that Pt10Rh is positive upon heating the tip, as described above.

3.1.3. Outer control Pt/Pt10Rh thermocouple assembly for ACIS setup (TCC/ACIS-S)

3.1.3.1. Procedure

Cut alumina tube to 35 cm.
You may drill a hole for the thermocouple weld point in the tip of the tube, using a small diamond ball bore.
Thread Pt and Pt10Rh in the tube.
Weld.
Cut the wires to 37.7 cm.
Insulate 2 cm.
Solder to connectors.
Check that Pt10Rh is positive upon heating the tip, as described above.

3.2. Instructions for fabrication of electrode contact assemblies

3.2.1. General equipment needed

For these assemblies you will need

alumina capillary tube, 2-bore, 3 mm Ø, 50 cm long or ordered to length (Frialit-Degussit AL23 order number 143 11030 0500)

Pt wire, 0.5 mm Ø
Pt net

In addition you need:

Flat-nosed pliers
Solder iron
Solder tin
Tape measure or other length measure
Lab stand with test-tube or burette grip.
Multi-purpose template bar

3.2.2. General procedures

In order to prevent contamination of samples, always wash your hands before working with parts that are to touch samples or otherwise will be present in the hot parts of the cell. Preferably wear gloves. We suggest you wash the exterior of parts after assembly using e.g. a suitable cloth and pure alcohol.

3.2.3. Electrode "hand" contact assembly, outer, 2-wire, top (H2TN15)

3.2.3.1. Materials

Alumina capillary tube, 2-bore, 3 mmØ, 50/50 cm
Pt wire, 0.5 mmØ, 86.9 cm

Pt net, 1.5×1.5 cm
2 female feedthrough connectors

3.2.3.2. Procedure

Cut 86.9 cm Pt wire.
Cut 1.5 cm Ø Pt net circle.
Cut alumina capillary tube to a length of 35 cm.
Thread through one hole of alumina tube and enough that 1.5 cm protrudes.
Place tube and wire on the template bar so that the threaded end is 40.2 cm from the centre of the stud formation.
Thread the wire over the stud formation in the given or desired pattern.
While threaded over the stud formation, thread the free end of the wire through the other hole of the alumina tube.
Align and cut to same length as the other end.
Loosen from the stud formation.
Place the two feedthrough sockets, with brass-coloured (wire) end up, in two next-neighbour holes in the multi-purpose template bar.
Place the two Pt wire ends in the two sockets, and support with the test-tube/burette grip.
With the solder iron, heat both wire and socket. Apply solder tin and let it float down into the socket until it wets the sockets inside and outside as well as the Pt wire. Do not move wire until tin is solidified.
Repeat for the other wire.
Check for visual signs of dry-soldering, and reapply heat and solder tin if it appears necessary.
Remove assembly and place electrode contact area between (clean) steel pistons in a hydraulic press, such that at least 1.5 cm from the centre of the electrode formation is pressed.
Press at 3 metric tons for a few seconds.
Check that Pt formation looks OK. For use with Pt net, attach the net and press once more at 5t.

3.2.3.3. Package and storage

Pack or store assembly as it is, i.e. without bending the electrode contact.

3.2.3.4. Assembly

Bend 90° 1.1 cm from the centre of the electrode contact formation.
Contact to HV (13) and HC (15).
Adjust height to account for sample thickness by bending wires at bottom and top if necessary.
Check that wires do not touch base unit block.

3.2.3.5. Use

Use as electrode contact to top electrode of disk sample.

3.2.4. Electrode "hand" contact assembly, outer, 2-wire, bottom (H2BN15)

3.2.4.1. Materials

Alumina capillary tube, 2-bore, 3 mmØ, 50/50 cm
Pt wire, 0.5 mm Ø, 83.6 cm
Pt net, 1.5×1.5 cm
2 female feedthrough connectors

3.2.4.2. Procedure

Cut 83.6 cm Pt wire.
Cut 1.5 mm Ø Pt net circle.
Cut alumina capillary tube to a length of 33.5 cm.
Thread through one hole of alumina tube and enough that 1.5 cm protrudes.
Place tube and wire on the template bar so that the threaded end is 39 cm from the centre of the stud formation.
Thread the wire over the stud formation in the given or desired pattern.
While threaded over the stud formation, thread the free end of the wire through the other hole of the alumina tube.
Align and cut to same length as the other end.
Loosen from the stud formation.
Place the two feedthrough sockets, with brass-coloured (wire) end up, in two next-neighbour holes in the multi-purpose template bar.
Place the two Pt wire ends in the two sockets, and support with the test-tube/burette grip.
With the solder iron, heat both wire and socket. Apply solder tin and let it float down into the socket until it wets the sockets inside and outside as well as the Pt wire. Do not move wire until tin is solidified.
Repeat for the other wire.
Check for visual signs of dry-soldering, and reapply heat and solder tin if it appears necessary.
Remove assembly and place electrode contact area between (clean) steel pistons in a hydraulic press, such that at least 1.5 cm from the centre of the electrode formation is pressed.
Press at 3 metric tons for a few seconds.
Check that Pt formation looks OK. For use with Pt net, attach the net and press once more at 5t.

3.2.4.3. Package and storage

Pack or store assembly as it is, i.e. without bending the electrode contact.

3.2.4.4. Assembly

Bend 90° 1.1 cm from the centre of the electrode contact formation.
Contact to LC (6) and LV (8).
Adjust height to account for sample thickness by bending wires at bottom and top if necessary.
Check that wires do not touch base unit block.

3.2.4.5. Use

Use as electrode contact to lower electrode of disk samples.