# Liquid Cell Setup for ProboStat™

Supplementary Material to the ProboStat™ Manual
Update: April 27, 2018



NORECS AS
Gaustadalléen 21
NO-0349 Oslo, Norway

Tel.: +47  45916188
E-mail: post@norecs.com
Web: www.norecs.com

# 1. Introduction

The liquid cell accessories for ProboStat™ have been developed to accomplish the following:

- full compatibility with a standard ProboStat™ base unit;
- controlled atmosphere or low-vacuum, temperature range RT - 200°C;
- possibility of disassembling parts for cleaning and replacement;
- simple construction to enable affordable in-house production or replacement orders of spare parts;
- reasonably high geometric factor of sample volume;
- good shielding to minimise parasitic capacitance of cell.

# 2. Contents and assembly

## 2.1. Contents

The liquid cell accessories (version delivered in 2005 or later) comprise the following parts, illustrated in fig. 1:

- One 30 mm outer diameter support tube LQST30 made of brass. Its socket fits on the pedestal of the ProboStat™ base unit.

- One sample container cup assembly LQCA30 consisting of an outer brass cylinder, an insulator cylinder, and a bottom electrode. The three parts are fitted without threads, just held and sealed by Viton O-rings. The unit can be disassembled when necessary by pushing or gentle hammering out the bottom electrode and insulator cylinder from the bottom. It is reassembled by pressing or gently hammering the parts back in place using suitable tools. In this process, ensure that the electrode surface stops perfectly aligned with the insulator ring.

- One brass top electrode LQTE with receptacle for outer electrode contact wires.

- Three Teflon sample compartment rings LQSR# with a groove on the top side and specific thicknesses of the thin part.

- One Teflon cylinder LQTC with cut-outs for spring load and outer electrode contact wires.

- One spring load assembly consisting of two-legged insulated Kanthal wire and two steel springs.

- One Ni/alumina inner electrode contact for the inside of the support tube LQIN2, to contact the bottom electrode.

- One supporting tube (alumina tube with a silicone hose) – for pressing the inner electrode contact into contact with the bottom electrode.

- One Ni/alumina outer electrode contact LQT2, to contact the top electrode.

- One thermocouple for liquid cell TCC/LQ, K- or S-type.

- One brass centering plate with various holes, used for centering the inner electrode contact.

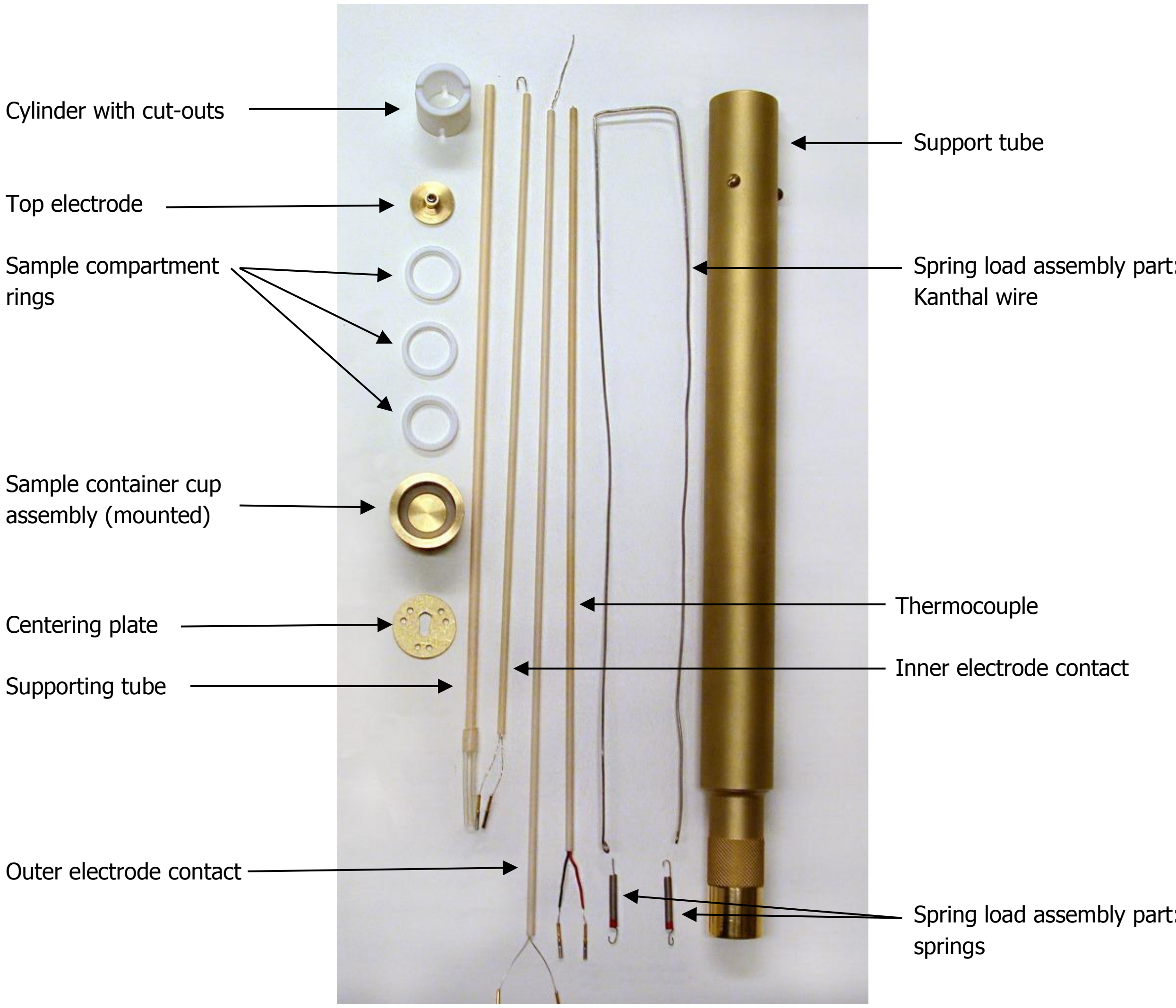



**Fig.1.** *Overview of parts for liquid cell setup.*

## 2.2. Use of liquid cell electrode contacts and thermocouple on ProboStat™ base unit with compensation wiring for other metals

Your ProboStat™ base unit may have compensation wiring for e.g. Pt wires, while the liquid cell may be equipped with Ni or Cu wires. This may cause small thermovoltages at the junctions in case of large thermal gradients through the system, but this has no influence on AC measurements (as in dielectric measurements).

Your ProboStat™ may be wired for S- or K-type thermocouples. The liquid cell accessories should come with the same type of thermocouple.

*If the thermocouple is used for temperature control or monitoring, the device must be programmed for the thermocouple type in use.*

## 2.3. Assembly

In order to assemble the liquid cell, follow general instructions for the ProboStat™ until you have the base unit mounted to a stand, with the outer tube and flange removed (see chapter 4 in the main ProboStat™ manual).

We recommend that you have the O-ring in place beneath the threads on the base unit pedestal (see fig. 4-3 in the main manual).

Also ensure that the four shields feedthrough terminals are connected together either with a shield bridge or switch (see chapter 4 in the main manual). This connection is required for some impedance spectrometers. You should disconnect shield feedthrough terminals if you positively know that your measuring instrument does not need or want it.

*Assembling the setup:*

**Step 1:** Press the silicone hose of the supporting tube onto the inner gas inlet on the base unit pedestal.

| | |
|---|---|
|  |  |
| High temperature zone | Connections to the ProboStat™ base unit |

**Step 2:** Attach the two legs of the inner electrode contact to feedthrough minicontacts 3 and 4 on the pedestal (see Wiring overview, chapter 14 in the main manual). These connect to the base unit ILC and ILV terminals. Let the top of the wire enter down in the top hole of the supporting tube.

| | |
|---|---|
|  |  |
| High temperature zone | Connections to the ProboStat™ base unit |

**Step 3:** Screw the support tube onto the pedestal and fasten it gently against the O-ring.

| | |
|---|---|
|  |  |
| High temperature zone | Connections to the ProboStat™ base unit |

**Step 4:** Feed the metal centering plate down around the inner electrode contact into the support tube, until it rests against the three small screws. (If you need to adjust it – and when you later need to remove it – use tweezers in the extra hole pair in the plate.)

| | |
|---|---|
|  | The same figure as above. |
| High temperature zone | Connections to the ProboStat™ base unit |

**Step 5:** Find and inspect the sample container cup assembly. If it is pre-assembled, just check that the electrode and insulator surfaces are aligned, smooth and clean. If it is disassembled, check that the two Viton O-rings are in place and in good shape, consider lubricating the O-rings slightly using high temperature tolerant grease, push or hammer gently the parts together and ensure good alignment of the electrode and insulator surfaces.

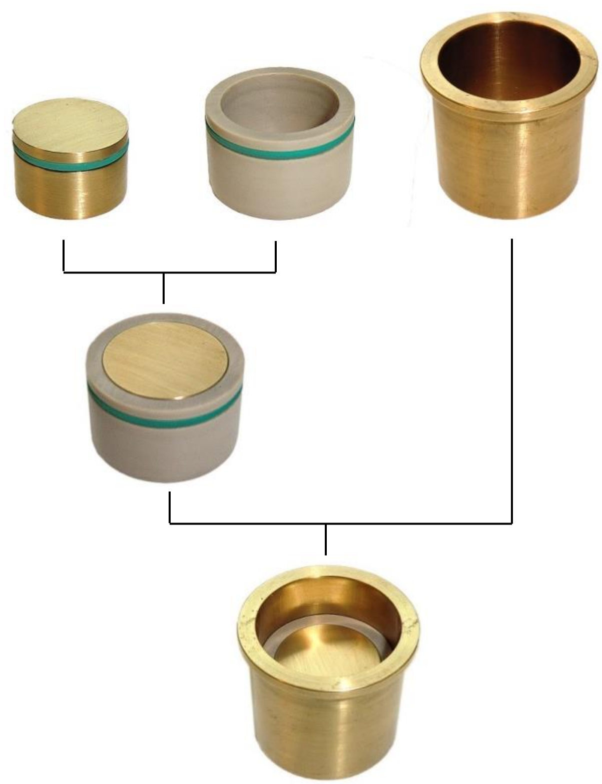


Fig. 2. *Sample container cup assembly mounting.*

**Step 6:** Place the sample container cup assembly into the support tube. In this process the inner electrode contact should find its way into the centre hole of the underside of the bottom electrode, and be pressed against it by the compressed silicone hose. At this stage, check that there is electrical contact between the IHC or IHV terminals of the base unit and the top face of the bottom electrode using a multimeter. If there is no contact, the electrode contact assembly did not hit the centre hole of the electrode, or the hose is not adjusted to press the electrode high enough.

| | |
|---|---|
|  | The same figure as above. |
| High temperature zone | Connections to the ProboStat™ base unit |

**Step 7:** Select a sample compartment ring (note its gap distance) and place it on the bottom of the cup, with the flat side down and the groove facing up.

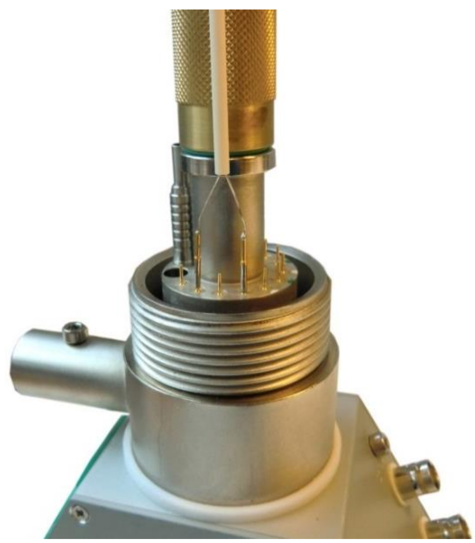
| | |
|---|---|
|  | The same figure as above. |
| High temperature zone | Connections to the ProboStat™ base unit |

At this stage you may insert the liquid to be measured (to a level where it just fills up the spacer ring) but it is suggested that you do a test assembly and measurement with an empty cell the first time.

**Step 8:** Place the top electrode in the groove of the sample compartment ring.

| | |
|---|---|
|  | The same figure as above. |
| High temperature zone | Connections to the ProboStat™ base unit |

**Step 9:** Connect the outer electrode contact to feedthrough terminals 13 and 15. These connect to the base unit HV and HC terminals. Insert the top wire pair into the receptacle at the electrode and fasten the screw gently. Ensure that the insulating ceramic tube extends past the rim of the sample container cup so that the wires don't touch. Also, check that the wire legs do not touch metal at the base.

| | |
|---|---|
|  |  |
| High temperature zone | Connections to the ProboStat™ base unit |

**Step 10:** Insert the Teflon cylinder, letting the largest cut-outs point downwards, and so that the wires of the outer electrode contact pass in the groove. This part holds the upper electrode down.

| | |
|---|---|
|  | The same figure as above. |
| Connections to the ProboStat™ base unit | High temperature zone |

**Step 11:** Attach the thermocouple onto the appropriate feedthroughs. (Mind the polarity!) Use feedthroughs 9 and 10 and connect the temperature controller to the TCT socket, or use feedthroughs 11 and 12 and the TCC thermocouple socket.

| | |
|---|---|
|  |  |
| High temperature zone | Connections to the ProboStat™ base unit |

**Step 12:** Mount the spring load wire in the cut-outs of the insulator ring. Attach the springs in the 360° groove on the side of the pedestal.

| | |
|---|---|
|  | To appear in future updates. |
| High temperature zone | Connections to the ProboStat™ base unit |

**Step 13:** Mount the outer enclosing tube, with its O-ring, and fasten using the flange.

| | |
|---|---|
|  | To appear in future updates. |
| Connections to the ProboStat™ base unit | High temperature zone |

Control of temperature and atmosphere, and the measurements themselves, can be done using procedures described elsewhere.

It is suggested that the cell is connected to a vacuum pump so that vacuum can be used to evacuate bubbles from the liquid and otherwise degas the liquid and the cell (assuming that the liquid under test is fairly non-volatile).

For exchange of liquid sample, the parts are disassembled in the reverse order to the extent necessary for emptying and cleaning. Normally, the thermocouple, the support tube, and the bottom electrode contact assembly may be left mounted, and the top electrode can be left connected to the wire pair. Use a mounting ring, a wrapping metal wire or some other means to hold the long outer items (top electrode contact assembly and thermocouple) upright along the cell while working on this.

## 3. Maintenance

There are no special needs here, except those that arise from the use of individual liquid samples and their possible interaction with the parts they are in contact with.

We anticipate that the need for replacements of parts is strongly dependent on combinations of the nature of the sample liquids, the atmospheres, and the temperatures applied.

We suggest that the sample compartment cup parts are not disassembled more often than necessary, but that it is rinsed and dried in the assembled state.

Should the cell become leaky during use because of failure of the O-ring seals, or if liquid is spilt because of other failures or mishaps, the bottom of the cell will be tolerant for most liquids. When cleaning up, be sure to be very gentle on the electrical feedthroughs, and consider flushing a rinsing liquid down the gas outlet holes.

## 4. Specifications

### 4.1. Materials and maximum temperature

The materials used which are in contact with the liquid are brass, Teflon, Viton, and PEEK. This normally allows an operating temperature in the hot part of the cell of at least 200°C, of course depending on sample and exposure time.

### 4.2. Electrical specifications

The top electrode is 18 mm in diameter. The bottom electrode is 17 mm of diameter. The sample compartment rings have inside diameters (and thus nominal liquid sample diameter) of 16 mm and thus sample area of 2.0 $cm^2$. They are further made in heights of 0.5, 1.0, and 2.0 mm. If we take 16 mm as the effective sample diameter, the cell gets nominal geometric constants ($K_g$) of 0.4, 0.2, and 0.1 m. This gives, in turn, nominal empty (vacuum) cell capacitances of 3.5 pF, 1.8 pF, and 0.88 pF.

In addition, the cell contributes parasitic capacitance caused by proximity of parts and through the Teflon spacer.

However, the shielding and guarding effect of the outer metal walls of the sample container cup and of the metal support tube and of the base unit metal chassis bulk, ensures that the measured parasitic capacitance is minimised. In order for this to work, the metal parts of the shield system – all connected to the base unit chassis bulk – must be connected to the measuring instrument's guard terminal. For impedance spectrometers like Solartron 1260FRA and HP 4192A this is the same as the shields of the measuring leads – that are connected together by the shields bridge. Be sure that the switch "Ch+HCS" is in position "down" to connect the shields to the chassis.

Measurements on empty cells indicate that the measured capacitance is 0.4 pF above the theoretical ones, and this can be taken as the sum of parasitics including the effect of the Teflon spacer.

Checks of the accuracy of the geometrical factor have been made using water as dielectric. This test is better than using air or vacuum since the capacitance of water is high. Measurements at 100 kHz showed capacitances (after subtraction of 0.4 pF parasitics) of 28.0, 13.6, and 6.6 pF for the three spacers, as compared with theoretical values of 28.5, 14.2 and 7.1 pF. This indicates that the absolute geometrical factors are within 90% of the nominal ones. Still, the accuracy can be improved by calibration the effective diameter and/or of the height of each spacer by using measurements of air and water, as here.

We conclude: The user should obtain the parasitic capacitance by measuring the capacitance of empty cells and subtract the calculated value from the measured value. The obtained parasitic value should be subtracted from all subsequent measurements. Then the cell should be measured with a standard liquid, e.g. water, the parasitic value subtracted, and the geometrical factor of the cell calibrated by dividing with the theoretical value.

## 5. Methods and measurements

Assemble the liquid cell as described in a preceding chapter.

Connect the high current and voltage terminals of the measuring instrument to the HC and HV terminals of the base unit and the low current and low voltage terminals of the instrument to the ILC and ILV terminals of the base unit. (The LC and LV terminals are not used.)

The Ch+HCS switch should be engaged (down) to include the base unit chassis, the metal support, and the outer part of the sample container cup in the shield system.

The other switch (LC+HCS) should be left up unless you want grounded measurements for some reason.

The following procedure for calibration and measurement is suggested:

Choose one sample compartment ring. Assemble an empty liquid cell and measure its capacitance (in vacuum or dry air). We suggest you use as high frequency as possible, but not above 1 MHz. The capacitance should be

$$C_v = C_0 + \varepsilon_0 K_g$$

where $C_0$ is the parasitic capacitance and $K_g$ is the geometric factor of the sample compartment (e.g. 0.4, 0.2, or 0.1 m). Next measure the same but with a known liquid sample in place:

$$C_s = C_0 + \varepsilon_0 \varepsilon_{r,s} K_g$$

where $\varepsilon_{r,s}$ is the relative permittivity of the sample liquid. Insert the known parameters and eliminate/combine to find $K_g$ and $C_0$:

$$K_g = \frac{C_s - C_v}{\varepsilon_0(\varepsilon_{r,s} - 1)}$$

$$C_0 = C_v - \varepsilon_0 K_g$$

The actual measurements of unknown samples then yield:

$$C_u = C_0 + \varepsilon_0 \varepsilon_{r,u} K_g$$

and

$$\varepsilon_{r,u} = \frac{C_u - C_0}{\varepsilon_0 K_g}$$

The above describes capacitance. A similar procedure can be used for conductance (real part of admittance, or imaginary part of capacitance) and the resulting loss parameters of the dielectric.

# 6. Instructions for fabrication of selected parts and samples

## 6.1. Inner electrode contact assembly (LQIN2)

### *6.1.1. Materials*

Alumina capillary tube, 2-bore, 3 mmØ, 24 cm
Metal wire, 0.25 - 0.5 mmØ, 57.4 cm
2 female feedthrough connectors

### *6.1.2. Procedure*

Cut 57.4 cm metal wire.
Cut alumina tube to 24 cm.
Thread the wire ends through both holes of alumina tube from the same side, enough that 3 cm protrudes on both wire ends.
Solder feedthrough connectors to the two open ends.
On the high-temperature end, narrow in the closed wire loop.

## 6.2. Outer electrode contact assembly (LQT2)

### *6.2.1. Materials*

Alumina capillary tube, 2-bore, 3 mmØ, 29 cm
Metal wire, 0.25 - 0.5 mmØ, 74 cm
2 female feedthrough connectors

### *6.2.2. Procedure*

Cut 74 cm metal wire.
Cut alumina capillary tube to 29 cm.
Thread the wire ends through both holes of alumina tube from the same side, enough that 4.6 cm protrudes on both wire ends.
Solder feedthrough connectors to the two open ends.
On the high-temperature end, narrow in the closed wire loop as much as possible.

## 6.3. Thermocouple assembly for liquid cell (TCC/LQ), K-type*

### *6.3.1. Materials*

Alumina capillary tube, 2-bore, 3 mmØ, 29 cm
NiCr wire, 0.5 mmØ
NiAl wire, 0.5 mmØ
2 female feedthrough connectors

### *6.3.2. Procedure*

Cut alumina capillary tube to 29 cm.
Thread the NiAl and NiCr wires in the tube and weld. Cut wires to 33.5 cm.
Insulate all but 1 cm. Use white insulation for NiAl and green for NiCr.

Solder to connectors.
Check that NiCr is positive upon heating the tip.

*Use Pt and Pt10%Rh wires for making S-type thermocouple. Use red insulation for Pt and black for Pt10%Rh.

## 6.4. Other parts

Not described at present – “factory made”.

## 6.5. Spring force assembly for liquid cell

Cut the Kanthal wire (1 mm) to 60 cm.
Make a sharp U-form at the mid-point of the wire by bending it over the top of a 30 mm tube.
Thread a 15 cm high temperature shrink tube onto the wire to cover the middle part, and heat it up.
Make an eye in each end of the wire for hooking the springs.