



Large Sample Setup for ProboStat™

Supplementary Material to the ProboStat™ Manual
Update: October 31, 2018

Copying allowed for internal use within organization or buyer of the product.

NORECS AS
Gaustadalléen 21
NO-0349 Oslo, Norway

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

NORECS

This is a supplement to the ProboStat™ manual, to be used together with (and not instead of) the manual.

1. Introduction

The large sample setup for ProboStat™ has been developed to accomplish the following:

- measurements on disk samples with diameter up to 60 mm,
- full compatibility with the standard ProboStat™ base unit,
- controlled atmospheres or low vacuum, temperature range RT - 1600°C.

The setup is operating in the single atmosphere mode and can be used for:

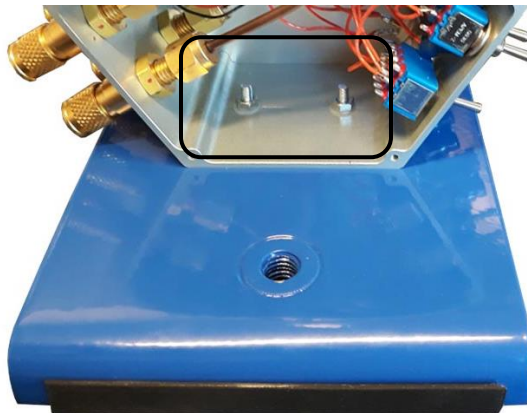
- 2-point impedance spectroscopy and conductivity measurements on large disk samples vs T, pO₂, pH₂O, etc.
- 2-point conductivity measurements with surface guard,
- electrode impedance studies with ring reference electrode,
- voltammetry studies with ring reference electrode.

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

The setup utilizes adapter that allows standard ProboStat™ to mount 80 mm enclosing tube (with 70 mm inner diameter).

Important:

The large sample enclosing tube makes the setup top heavy, and the normal stand system is inadequate to be used in the traditional fashion. The large sample set includes additional way of supporting and stabilizing the ProboStat cell. The blue base should be attached to the bottom of the ProboStat using the supplied screws. Open the side plate of the ProboStat base unit, add and tighten the screws and nuts (use the washers) as illustrated in the Figure below.



2. Contents and assembly

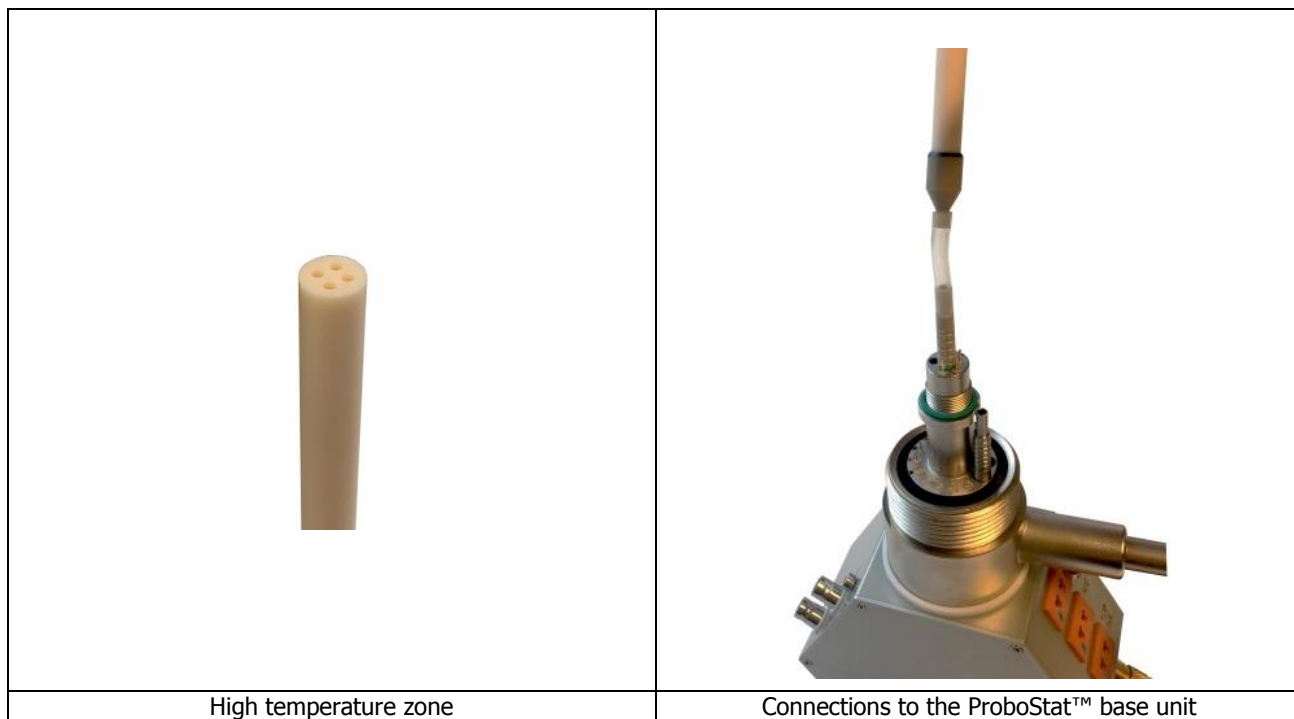
2.1. Contents

- One set of neck adapter and flange made of brass,
- One alumina enclosing tube with outer diameter 80 mm, closed one end,
- One enclosing quartz/metal ring with outer diameter 40 mm used to avoid gas leakage through the adapter,
- One standard ProboStat™ sample support tube assembly with outer diameter 24 mm,
- One alumina support plate / sample support tube adapter for sample stabilization and resting of the ring electrode contact,
- One spring load assembly consisting of one alumina support plate with central hole, three standard long alumina bars with holes, one top triangle made of Pt10%Rh wire \varnothing 1 mm, and three soft springs,
- One standard alumina inner multi-bore gas supply tube with 3.9 mm length silicone hose,
- One standard outer alumina gas supply tube,
- One thermocouple for disk sample, S-type, alumina,
- One Pt/alumina guard-reference-ring electrode with 58 mm \varnothing ring.
- One Pt/alumina inner "hand" electrode contact,
- One Pt/alumina outer "hand" electrode contact with 20 mm \varnothing net,
- One Pt net, \varnothing 20 mm,
- Two Viton O-rings, 80×3 mm and standard 40×3 mm.

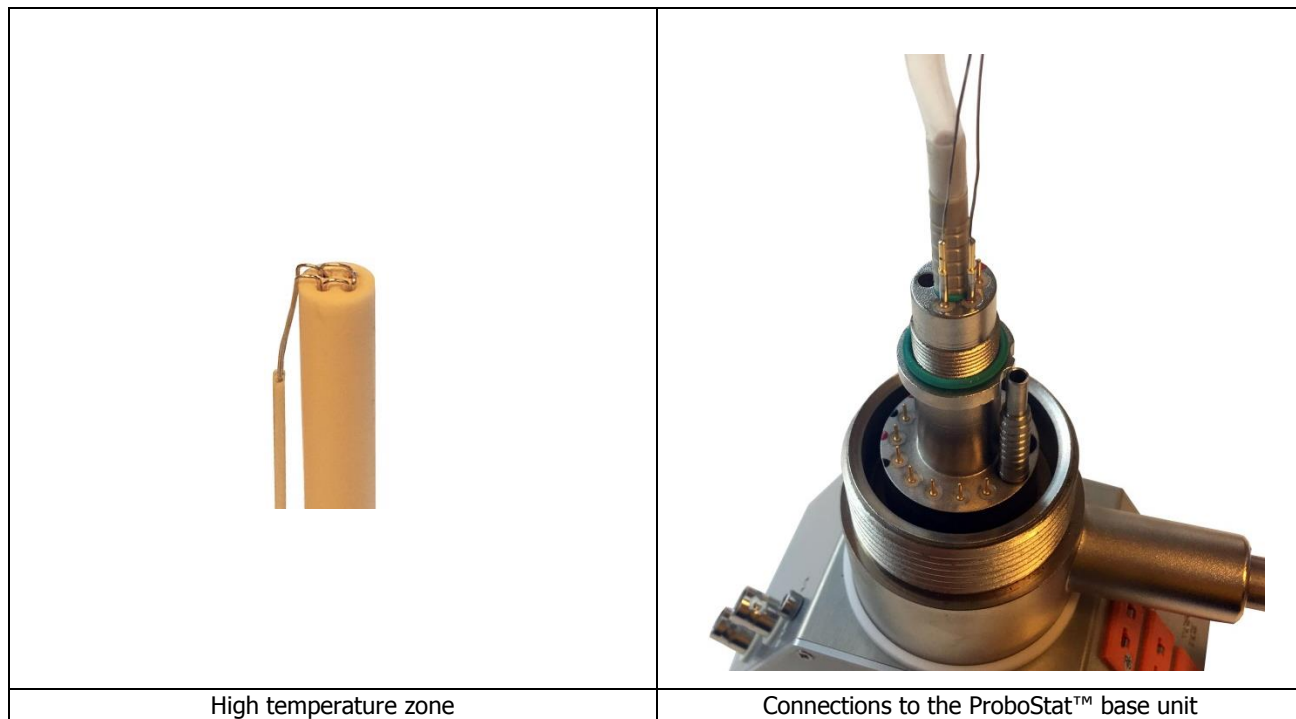
2.2. Assembly

The given example is related to Part 8.2.3 in the ProboStat™ manual: 2-point conductivity measurements on disk sample with surface guard. For other methods please refer to the corresponding chapter in the ProboStat™ manual.

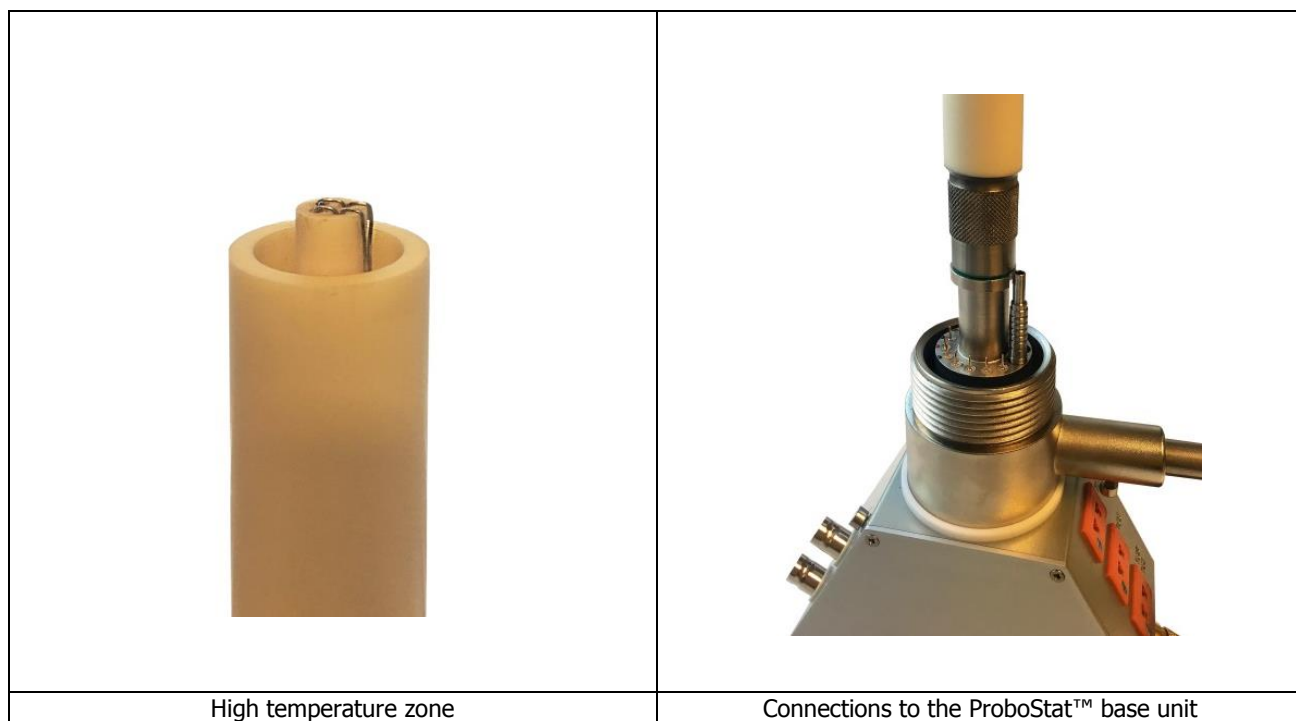
Step 1: Mount the multi-bore gas tube onto the inner PEEK gas socket.



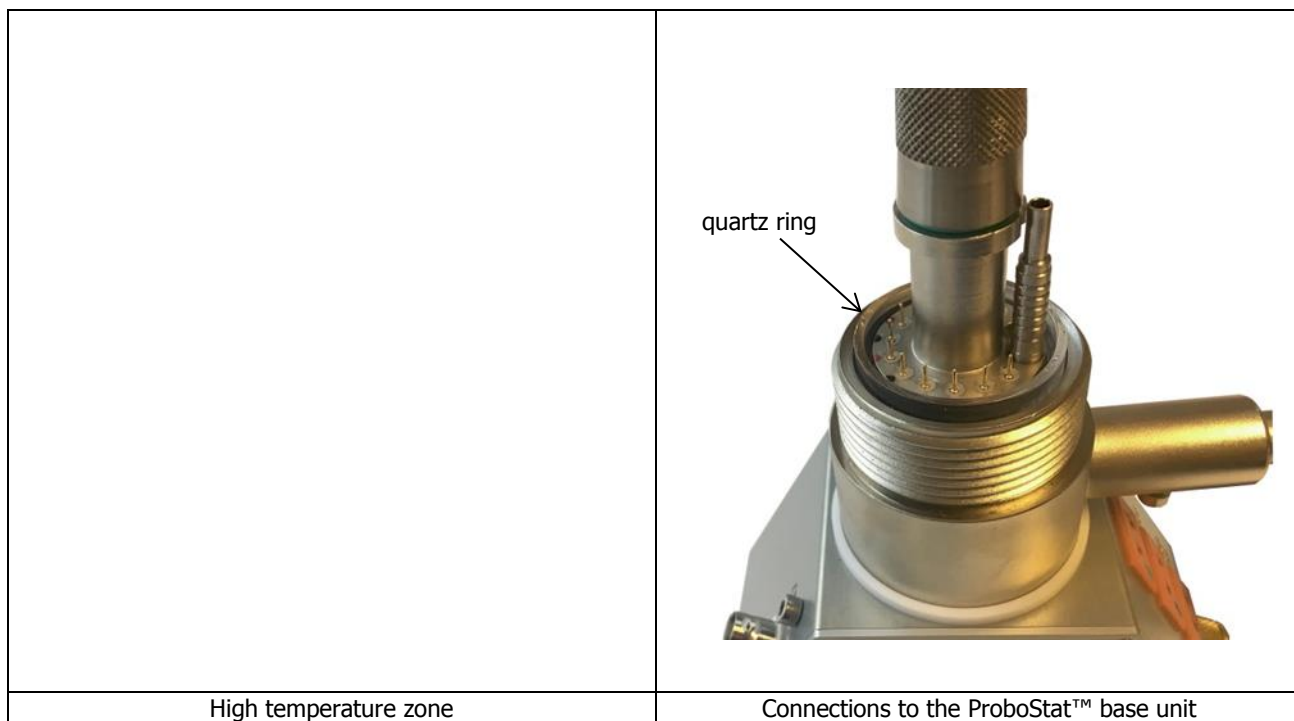
Step 2: Mount the inner “hand” electrode contact onto ILC and ILV feedthroughs, no. 3 and 4 accordingly. Carefully insert the top part of the electrode contact down into the multi-bore tube.



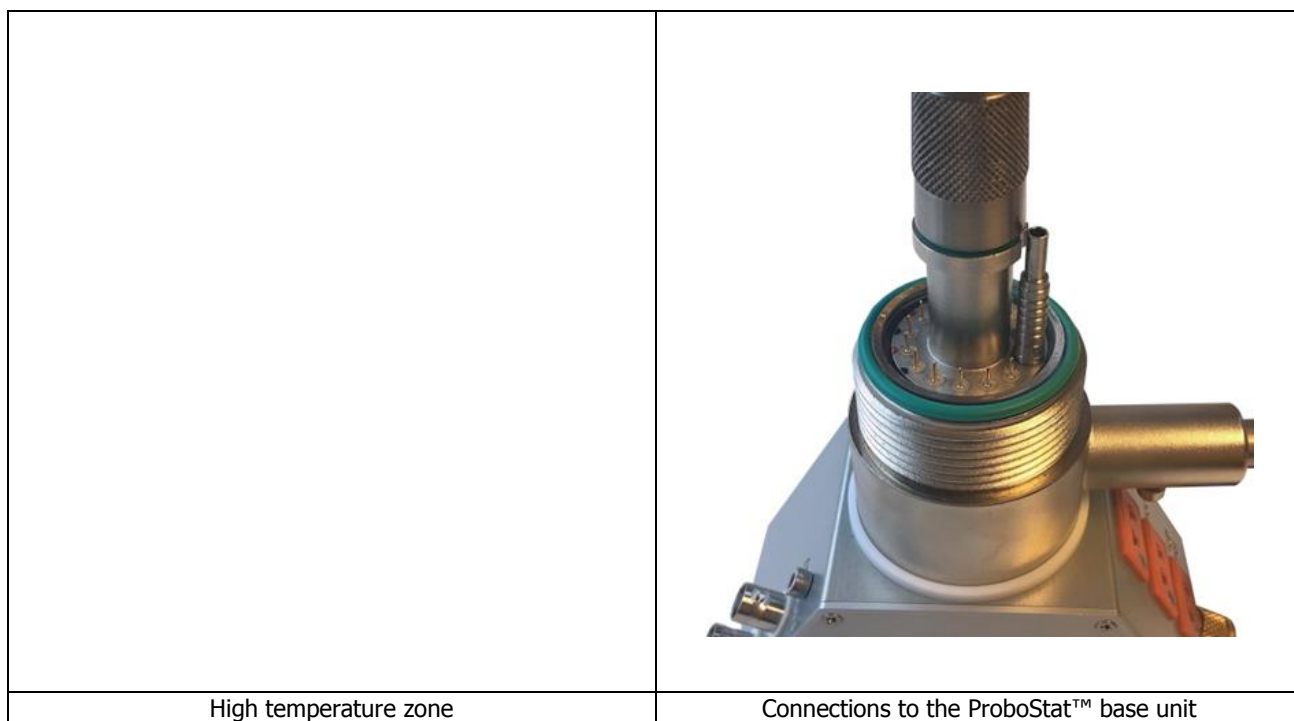
Step 3: Mount the sample support tube. Adjust the height of the gas tube so that the electrode contact touches sample when the support plate / support tube adapter is mounted (see Step 7). Take the adapter off.



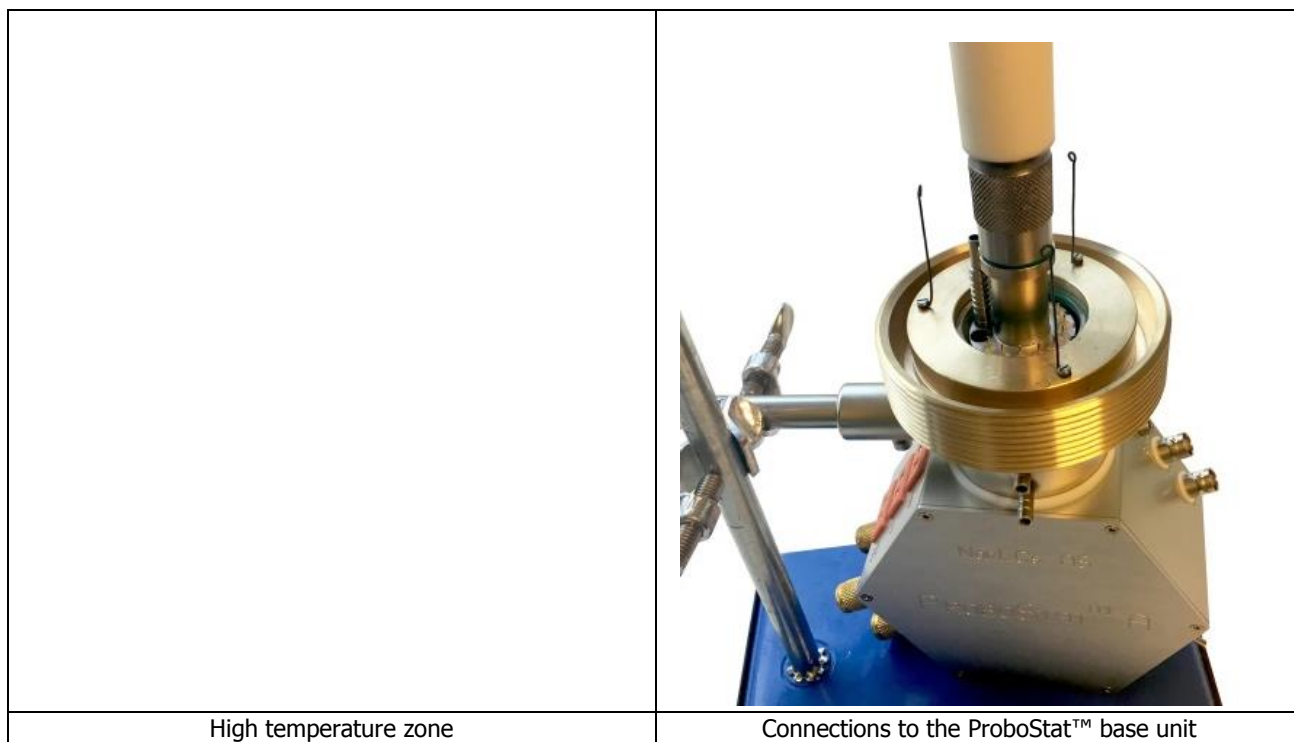
Step 4: Insert the enclosing quartz/metal ring in the base unit groove for the enclosing tube.



Step 5: Place the standard Viton O-ring (40×3 mm) outside the quartz ring.



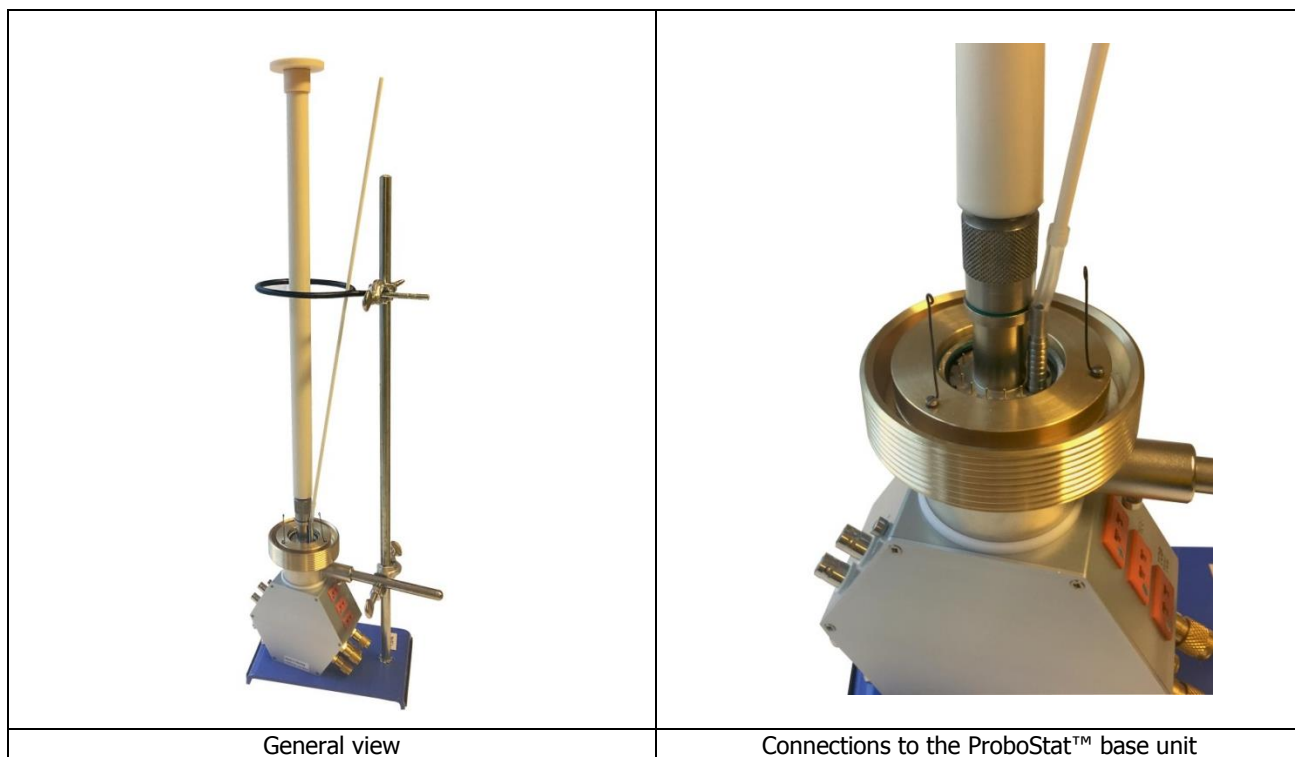
Step 6: Mount the neck adapter.



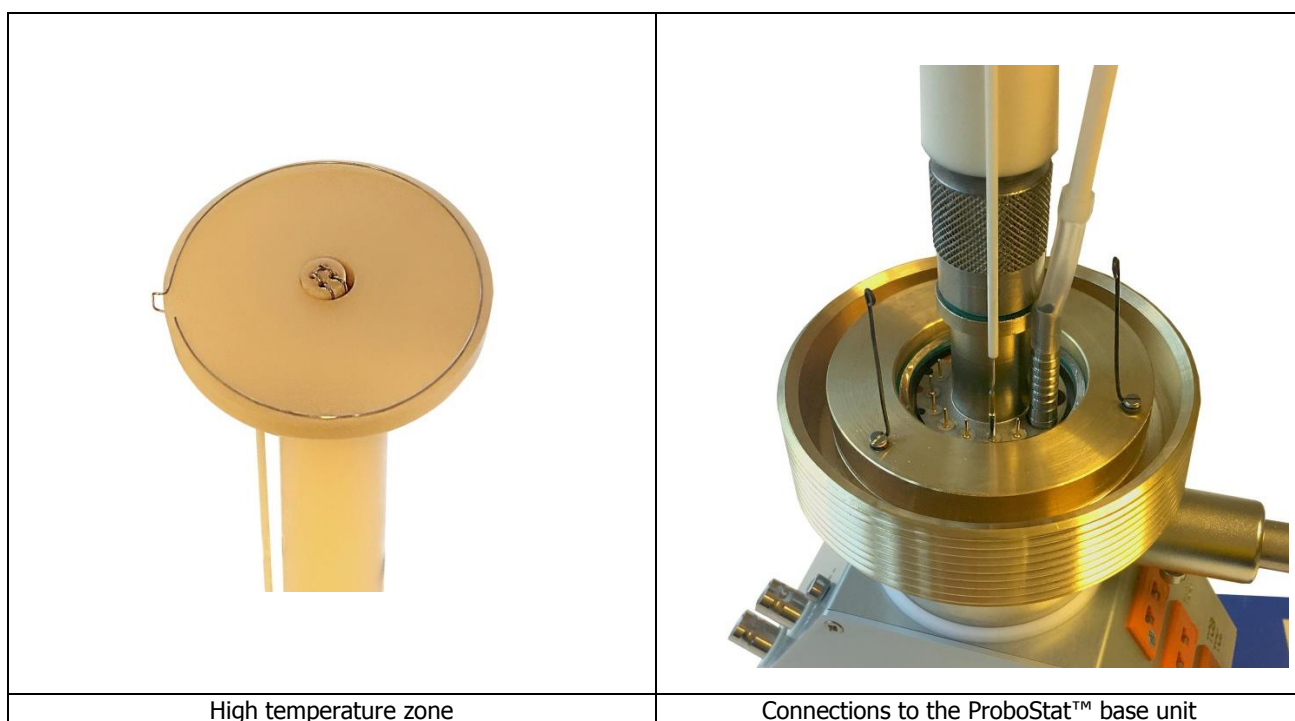
Step 7: Place the support plate / sample support tube adapter on top of the support tube. The gas supply tube should be approximately 1 mm above the adapter.



Step 8: Mount the outer gas supply tube onto the outer steel gas socket. Be careful when mounting. We recommend using the stand ring for support during mounting.



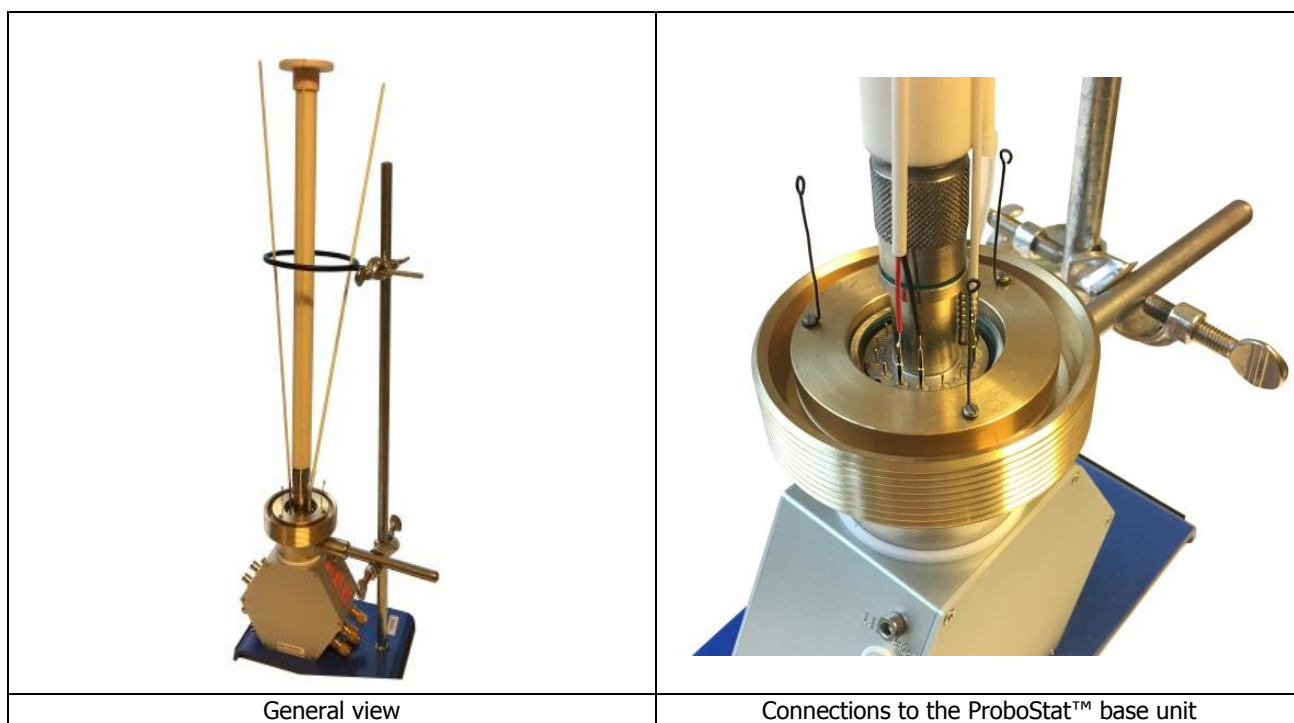
Step 9: Insert the ring electrode contact onto LC feedthrough (no. 6). Carefully bend the top part of the electrode contact so that it covers the rim of the alumina plate.



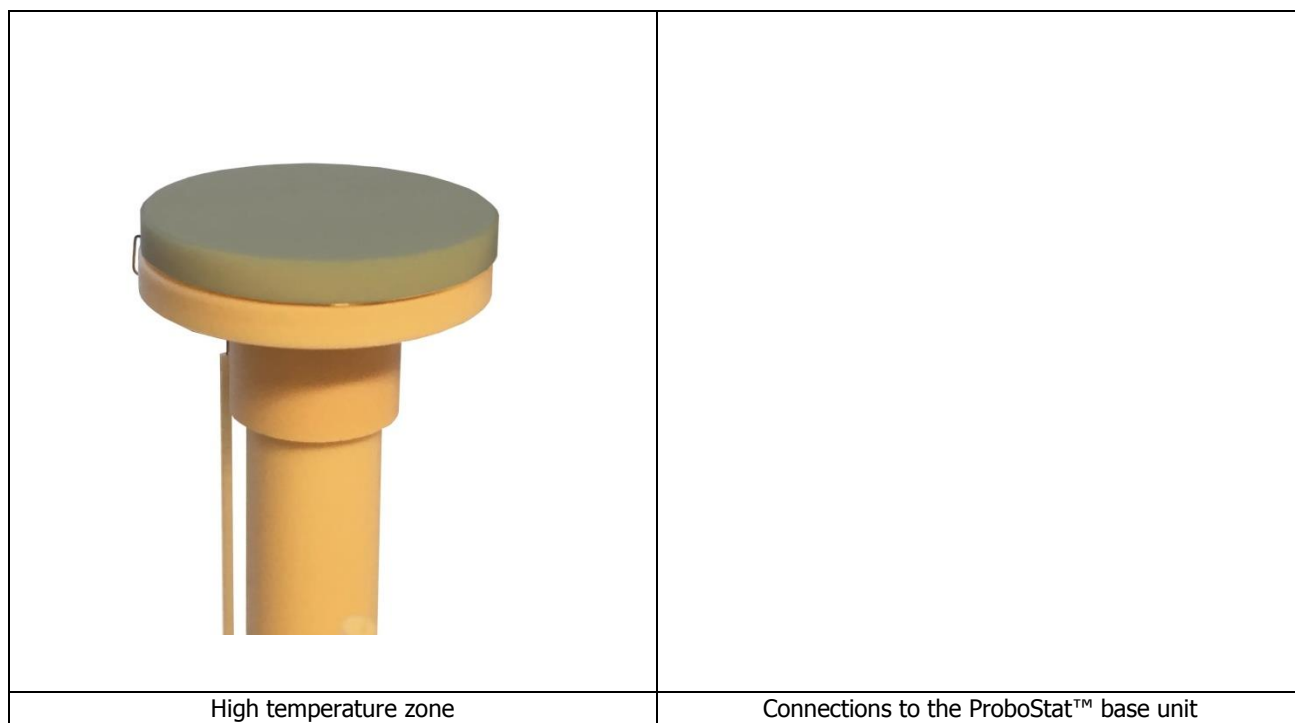
Step 10: Place the 20 mm Ø platinum net on top of the inner “hand” electrode contact.



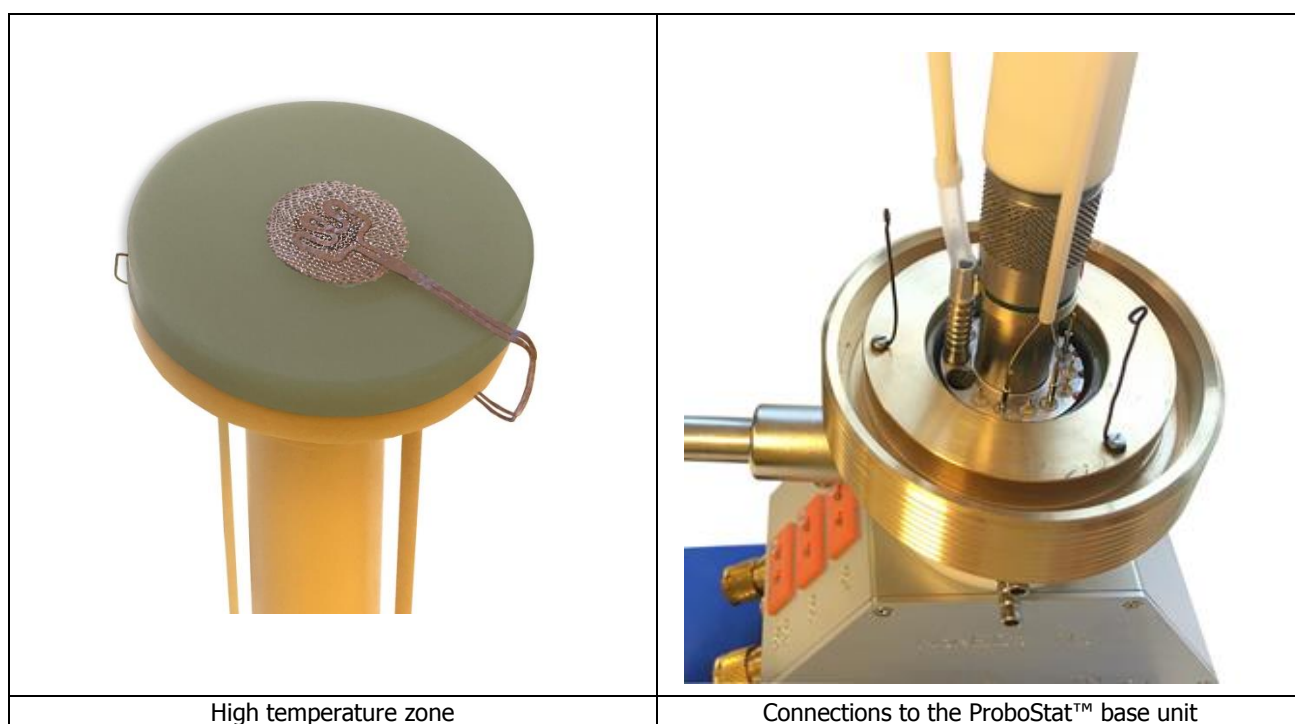
Step 11: Insert the control thermocouple onto feedthroughs no. 9 and 10, or no. 11 and 12. Mind the polarity!



Step 12: Place the sample so that it rests on the support plate. Make sure that it slightly presses down the the inner "hand" electrode contact, and the ring electrode contact is in the correct position.



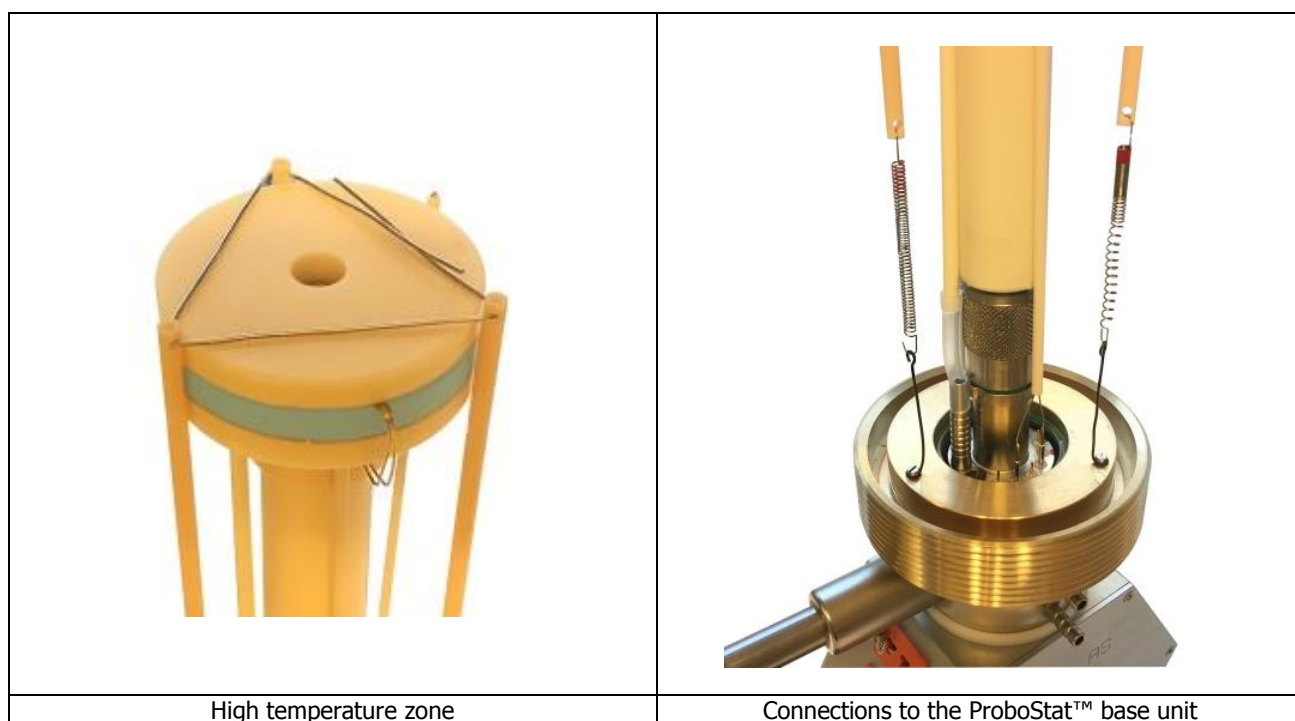
Step 13: Insert the outer "hand" electrode contact onto HV and HC feedthroughs, no. 13 and 15 accordingly. Carefully bend the top part of the electrode contact.



Step 14: Place the support plate with central hole on the outer electrode contact.



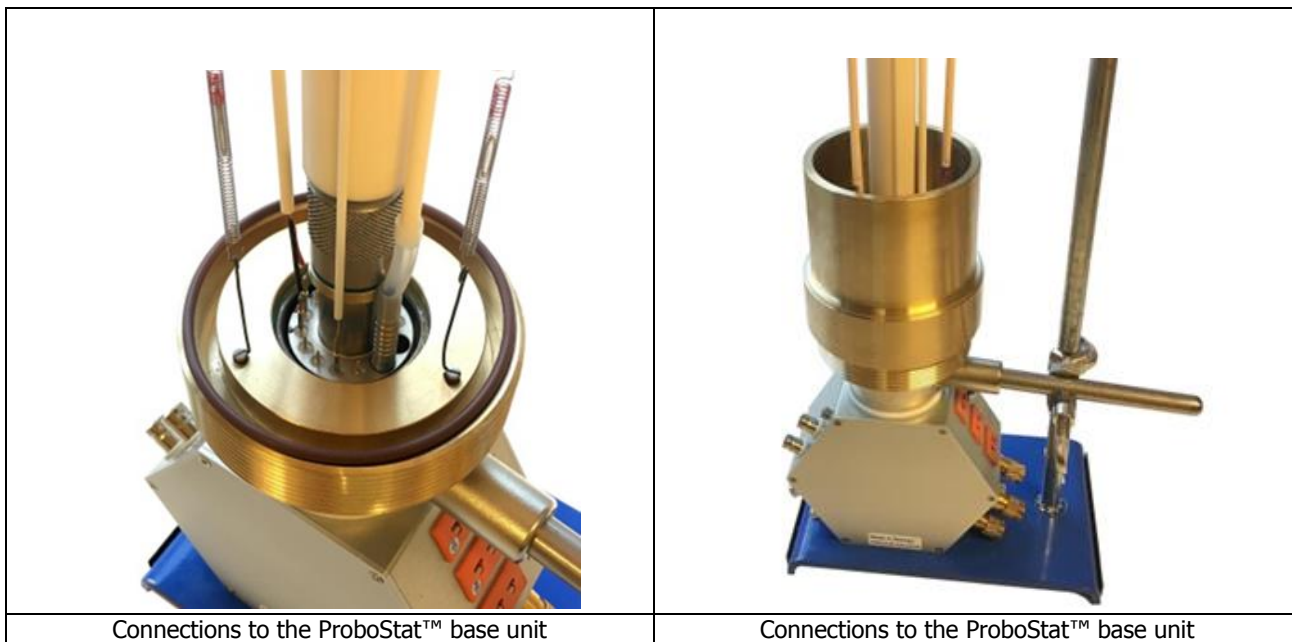
Step 15: Position the pre-mounted spring load tubes into the triangle vertexes. Place the assembly on top of the support plate. Hook the springs onto the lower part of the spring load tubes. Hook the springs over the neck adapter. Hold firmly the top part in place while connecting.



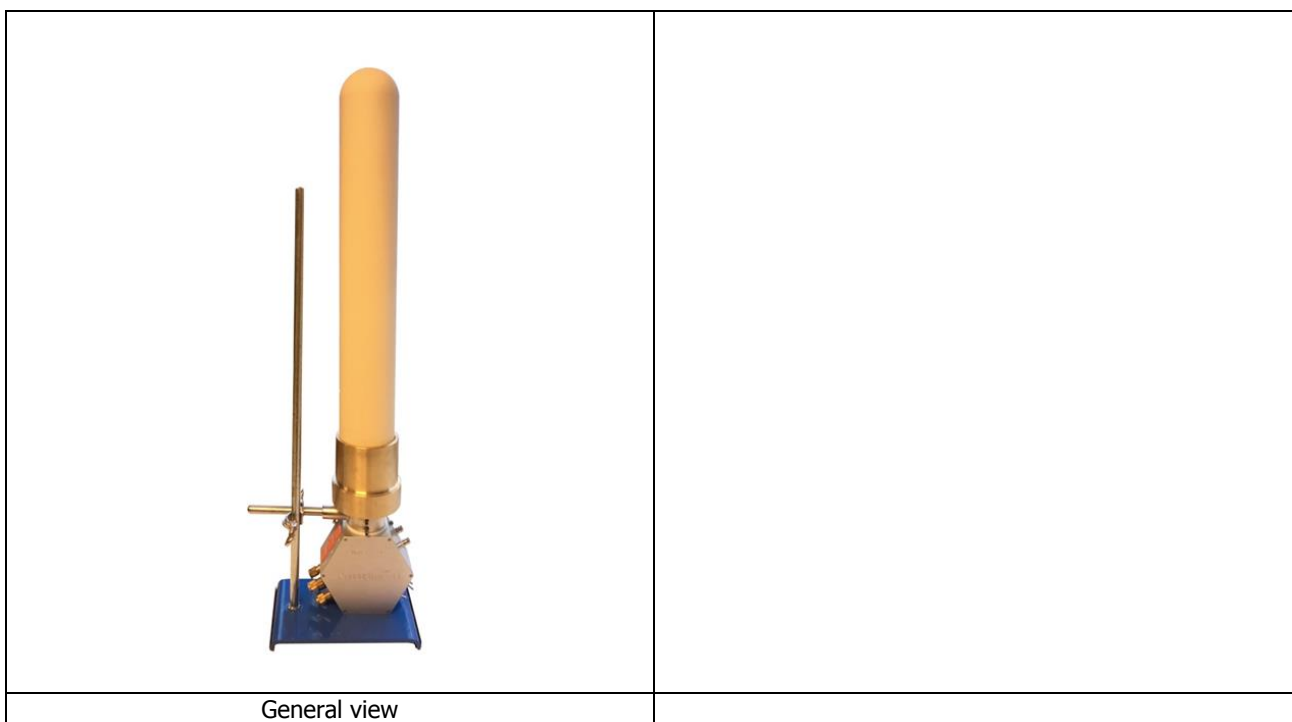
At this point the setup is assembled. There are two methods to close it with the enclosing tube.

Alternative one:

Step 16: Place the O-ring on the neck adapter and screw on the flange all way down. Do not tighten.

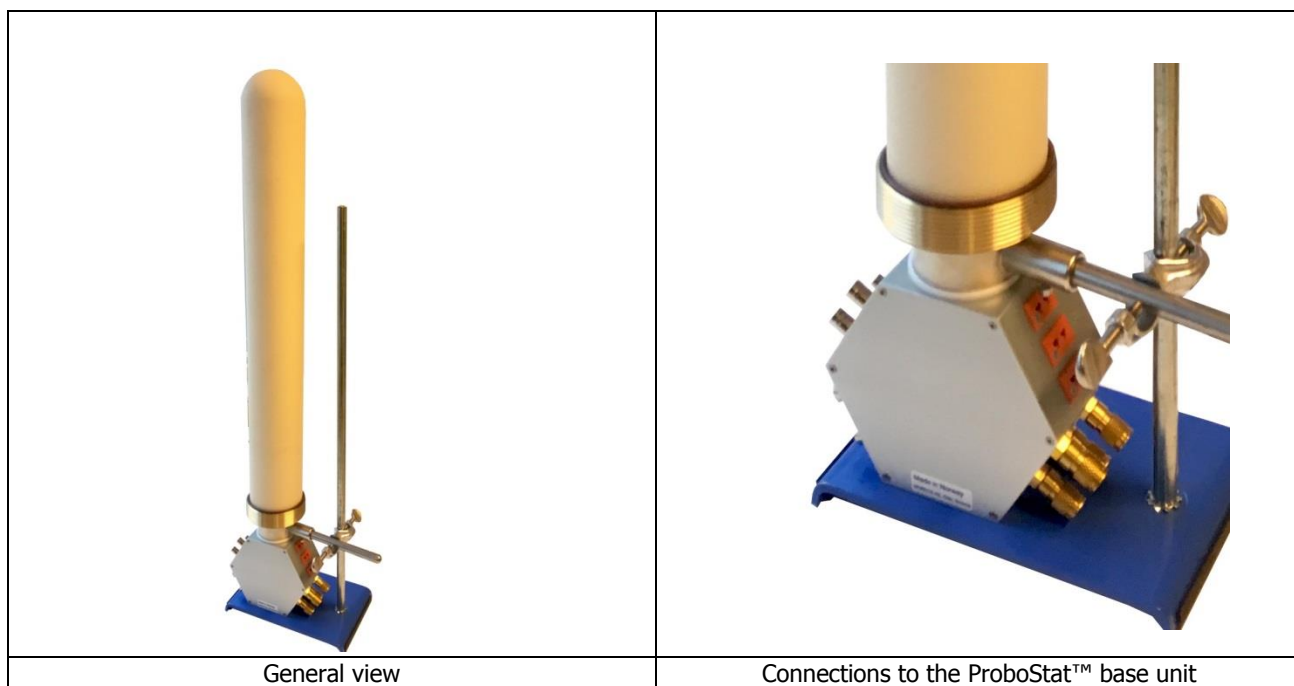


Step 17: Insert the enclosing tube into the flange. You should be sure that the tube reaches the bottom of the base unit groove. Do not drop it! Afterwards tighten the flange.

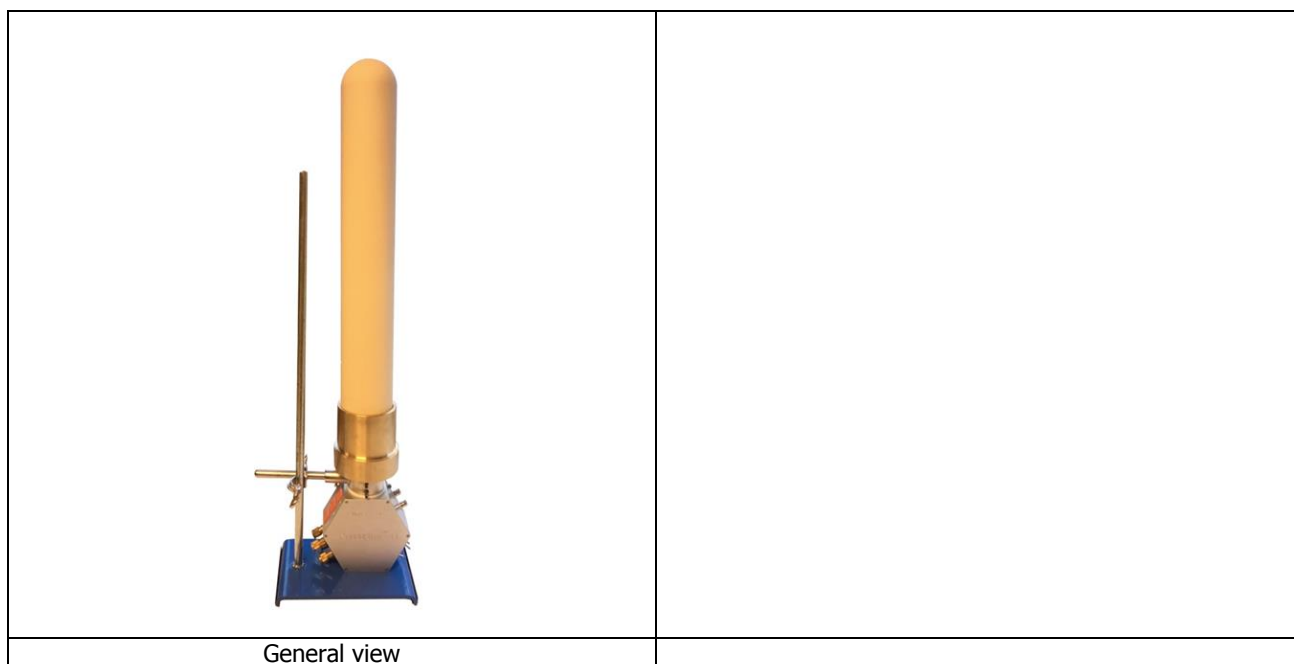


Alternative two:

Steps 16: Mount the O-ring onto the open end of the enclosing tube. Insert the tube into the base unit groove. Move the O-ring down.



Step 17: Carefully move the flange down the enclosing tube. Do not tilt it as it can become jammed! Tighten the flange.



Step 18: Make electrical and gas connections (please see the ProboStat™ Manual, Chapter 8).

The ProboStat™ manual can be downloaded from the NORECS website www.norecs.com.

For more information please contact us:

NORECS AS
Gaustadalléen 21
NO-0349 Oslo, Norway

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