

ComPrep User Manual



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rhd instruments GmbH & Co. KG

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1 Product description

The ComPrep is a device that allows the user to manually apply a mechanical force to an electrochemical test cell.

The mechanical force is applied by operating a spindle. The test cell is placed between a lower platform and a contact plate at the bottom of the spindle, such that the hard metal pistons or flat contact surfaces of the test cell in contact with the sample get compressed. The geometry of the frame and test cells assure that the force is applied uniaxially to the sample.

The force applied in the ComPrep is adjusted using a torque wrench and a given force-torque correlation, see 7.5.

Please read this manual carefully in order to learn how to use the ComPrep successfully, safely, and efficiently.

Thank you for choosing the ComPrep. We wish you joy and success working with it.

2 General information

The instructions in this manual were checked carefully for correctness. However, liability for any mistakes in form and content will not be assumed. Additionally, rhd instruments GmbH & Co. KG (in the following declared as rhd instruments) reserves the right to change the setup and design of the products presented and described within this manual. Such changes are necessary to guarantee the continuous development of the products and, thus, the improvement of product quality and reliability.

The ComPrep is used to prepare a CompreCell with a solid sample and to remove it. It is also used to manually apply a mechanical force to a solid sample in a CompreCell before or during an electrochemical measurement.

During operation the measuring cell is located between a lower platform and a contact plate attached to a moving trapezoidal thread spindle. Once the measuring cell's upper piston is in contact with those two surfaces, direct mechanical contact between the spindle and the frame is established. Hence, further movement of the spindle in the lower direction will increase the mechanical force applied to the measuring cell while movements to upper positions will decrease the force.

Note: In this manual the mechanical unit of the ComPrep system will often be referred to as "ComPrep".

The applied force can be estimated by the use of a torque wrench and the attached force-torque correlation table, see 7.5.

Markings in this manual

Marking	Meaning	
WARNING	Indicates a hazardous situation which, if not avoided, could result in serious injury or death.	
ADVICE	Indicates potential physical damages and other important information associated with your device.	
IMPORTANT	Important pieces of information are emphasized in special boxes. Please take special note of these as they may contain safety-critical information.	

3 Essential features at a glance

- » High quality cell stand for manual pressure application on CompreCells.
- » As airtight measuring cells, CompreCells enable the handling of sensitive samples outside a glovebox.
- » Stand-alone operation possible.
- » Universal compatibility with all potentiostats/galvanostats.
- » Support for preparation and dismantling of measuring cells.

Note:

If you have any questions, for example with regard to the compatibility of your measurement devices, do not hesitate to contact us via email (info@rhd-instruments.de) or phone (+49-6151-8707187).

4 Important general safety notes

- » To avoid physical injuries and damages, please read this instruction manual carefully before using the device for the first time.
- » Please pay attention to all safety notes in this instruction manual.
- Please keep this manual safe. In case of selling or leaving the device to third parties, please do not forget to hand this manual over as well.
- The operation of the ComPrep system should only be performed by properly trained and experienced members of staff.
- » The setup has been developed for electrochemical measurements of solid samples under force-, and potential-controlled conditions as well as for the preparation of CompreCells and the removal of samples from them. It must not be used for any other purpose.
- » To avoid unstable operating conditions and injury, the ComPrep setup as well as the individual components should not be used if
 - they show noticeable damage,
 - they were stored or operated under unapproved conditions (see operational condition, storage and rated values),
 - they were exposed to high mechanical stress, exceeding normal usage,
 - they were altered by members of staff not authorized by rhd instruments.
- » Do not operate the device with wet hands.
- » Operate the device only in dry rooms.
- » Do not operate the device outdoors.
- » Please follow only the instructions in the hardware manual for cleaning your device.
- » Please follow only the instructions in the hardware manual for maintaining your device.
- Prevent your device from mechanical impact. In case the device fell down, please contact rhd instruments or a technician authorized by rhd instruments before using it again.

- » If your device shows any visible damage or defect: Never operate your device in a damaged state. Never repair the device on your own. The device should only be repaired by either rhd instruments or by a technician authorized by rhd instruments.
- » Do not loosen nuts or bolts unless this manual specifically tells you otherwise.



WARNING

Under no circumstances loosen any nuts or bolts while a force is applied!

» Only use original spare parts delivered and approved by rhd instruments.



ADVICE: Connect and disconnect any cable connection carefully.



ADVICE: Handle chemicals with care.

When handling chemicals during preparation and execution of measurements with the ComPrep, the usual safety advice in accordance with the H, EUH, and P statements (in the European Union: rating principles according to the CLP regulation) and appropriate safety measures have to be observed. This applies to subsequent cleaning and decontamination as well.



ADVICE: Sufficient cleaning increases the lifetime of your system.

» After using the ComPrep for electrochemical measurements all components in contact with chemicals need to be thoroughly cleaned. Insufficient cleaning, decontamination, and drying of the components may result in damage due to corrosion and, thus, may affect the quality of your measurement results.



ADVICE: Be careful when bringing chemicals in contact with parts of your system.

» When operating your ComPrep system with compatible measuring cells please be advised to only use samples that are chemically inert towards the main materials of the measuring cell (e.g. alumina, polyether ether ketone (PEEK), tungsten carbide, polyethylenimine (PEI)).

Caution when using metallic Lithium in combination with a measuring cell with an insulation sleeve made from any oxide ceramic. The sample temperature should not be set to temperatures high enough that metallic Lithium starts to react with the ceramic material. In general, the ComPrep system is only allowed to be operated under conditions that correspond to the specifications described in this manual and under which the main components of the measuring cell used are stable.





Always wear protective gloves and eye protection when working with the system under pressure or with chemicals.

5 Installation

5.1 Unpacking your ComPrep

- » Open the cardboard box.
- » Take off the upper foam cover.
- » Take the torque wrench, the nut, the two rods and the handwheel out and put them aside.
- » Take the ComPrep out of the box by lifting it on the two front rods.
- » Place the ComPrep so that the spindle points upwards.
- » Take out the three preparation and dismantling tools and put them aside.

5.2 Components of the ComPrep

» Please check if the delivery is complete:

ComPrep as delivered as a tool together with CompreDrive or CompreFrame:

- 1 x ComPrep
- 1x handwheel
- 1 x torque wrench
- 1 x nut wrench size 18 mm
- 2 x Piston removing aid for CompreCell 12
- Adaptor 4 mm male to 2 mm female
- Adaptor 2mm male to 4 mm female
- Extension plug 4 mm
- 2 ml of grease

Additionally, if ComPrep was ordered separately as item 840104141:

- 100 Ohm test resistor
- Cell holder for cell preparation
- Sample removing aid for CompreCell 12/6

- Sample removing aid for CompreCell DP
- » Please check if the delivered items are undamaged



ADVICE: If the delivered items are incomplete or damaged please contact rhd instruments via e-mail (info@rhd-instruments.de) or via phone (+49-6151-8707187).

rhd instruments will reject any claims for warranty or responsibility in case damaged equipment is used.

In case accessories of other manufacturers are used, rhd instruments will accept no liability.

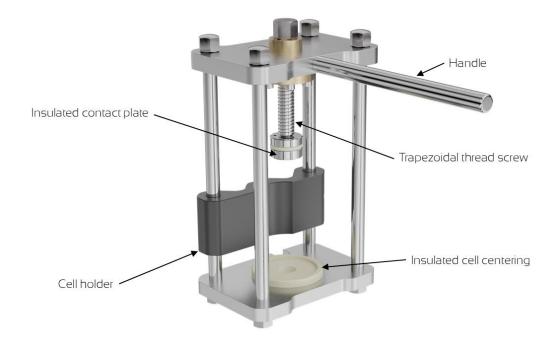


Figure 1: Components oft he ComPrep

6 Operation conditions, storage and rated values

» Temperature range during operation $T_{operation} = -30 \,^{\circ}\text{C}$ to $+100 \,^{\circ}\text{C}$

(ComPrep)

» Temperature range during long-term T storage = +10 °C to +40 °C

storage:

» Relative humidity (RH) for working and (non-condensing) 0 to 80% RH

storage area:

» Atmosphere during storage Non-corrosive

» Maximum electrode potential $U = \pm 30 \text{ V}$

» Maximum measurement current 2A

» Rated dimensions 96x140x300 mm

with mounted lever. 270x140x300

» Typical weight: approx. 4,4 kg

» Maximum force 23 kN

» Maximum force (long-term reproduci- 18 kN

bility of the force-torque table)

» Maximum torque (for 23 kN)
45 Nm

Maximum torque (for 18 kN) 35 Nm

» Force at lever (for 23 kN)

Materials:

Part	Material	
Spindle, rods and platforms	Stainless steel	
Spindle nut	Bronze	
Insulation	PEEK (polyether ether ketone)	
Screws, nuts and bolts	Stainless steel	
Cell holder	POM (polyoxymethylene)	

7 General Operation

7.1 Moving the spindle

The central contact plate of the ComPrep is moved by turning the spindle at the top of the ComPrep.

When looked at from above the directions of travel are:

- » Clockwise: Move the platform down / increase mechanical force
- » Counterclockwise: Move the platform up / decrease mechanical force

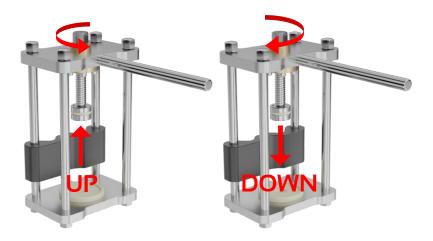


Figure 2: Directions of travel

The spindle can be turned by hand. The handwheel can be attached to the spindle to make turning easier.

In order to apply higher forces more easily, the ComPrep includes a lever. It has a threaded end and can be screwed into the upper platform of the frame. When not in use, the lever can be screwed into the lower platform at the rear of the frame and stowed away.

The ComPrep includes a torque wrench with an 18 mm nut, that can be placed on the bolt head and used as a second lever.

Hold the fixed lever with one hand and move the torque wrench to change the force on the test cell.



Never exceed the force ratings of the ComPrep to avoid permanent damage!

7.2 Analyzer connection to the ComPrep

The ComPrep is designed to facilitate connection of an electrochemical analyzer to the test cell. It has a built-in connection point for the top piston, typically used as working-or counter-electrode.

The top electrode is connected via a 2 mm banana receptacle on the insulated contact plate at the bottom of the spindle. The receptacle is connected to the upper piston in the test cell, once a force is applied to the cell in the frame.

The bottom electrode can be connected via the two 4mm holes in the bottom part of the CompreCell.



Figure 3: Upper and lower cell contacts detail



Figure 4: Lower cell contact in bottom part of CompreCell

To standardize the sizes of the banana connections, two adapters are included with the ComPrep. These can be combined in such a way that the cell can be connected uniformly with 2 mm or 4 mm banana plugs.

The 4mm extension plug must be used at the bottom as otherwise the housing of normal banana plugs would rest on the centering and tilt the cell. This must be avoided at all costs, as it can damage the cell.

7.3 Loading the test cell into the ComPrep

Consult the CompreCell manual for instructions for preparation of the test cell.

Turn the spindle of the ComPrep counterclockwise to make enough vertical space to insert the CompreCell into the frame.



Make sure that the test cell is sitting centered inside the PEEK ring and not on top of it.

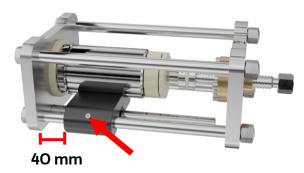
The test cell must not be angled away from vertical.

Misplacing the test cell in the frame can cause damage to the device.



Figure 5: CompreCell in ComPrep

7.4 Adjustment of the cell holder



If necessary, the height of the cell holder can be adjusted. To do this, the setscrew on the cell holder must be loosened using a 3 mm Allen key. The cell holder can now be moved. To ensure that all cells are supported, a distance of 40 mm from the lower platform is recommended. Once the cell holder is at the correct height, the setscrew can be carefully tightened again.



The cell holder allows the ComPrep to be placed on its back without the cell falling out.

7.5 Usage of the ComPrep for measurements

With a cell in place, turn the spindle clockwise, until the contact plate at the spindle contacts the cell. After the first contact, set the torque wrench to the desired torque and continue turning the spindle slowly with the torque wrench. The desired torque can be read from the force-torque table at the top of the ComPrep. When the torque wrench reaches the preset torque, it makes a clicking sound. At this point, the desired force has been applied.

The force-torque correlation given at the top of the ComPrep was determined on the basis of a large test series. Nevertheless, it will not be possible to achieve a high degree of accuracy with this. This correlation only applies as long as the torque of 35 Nm is not exceeded. After a higher load, the correlation changes permanently.

7.6 Usage of the ComPrep for preparation and extraction

The ComPrep can be used to pre-compact a sample that has been filled into a CompreCell (see the manuals for the CompreCells). To do this, place the CompreCell filled with a sample into the ComPrep and turn down the spindle. To make turning easier, place the handwheel on top of the spindle.

The ComPrep can also be used to extract stuck pistons. To do this, unscrew all parts of the CompreCell until only the jacket with the stuck piston remains. Now screw the sample removing aid suitable for the cell into place of the base. Put the CompreCell into the ComPrep. In this application the ComPrep has to be used horizontally, as the piston pushed through the opening in the lower platform. Insert one piston removing aid between the contact plate and the stuck piston. Push the piston as far in as possible, then insert the second piston removing aid and repeat that. The stuck piston should be loose. Make sure the hard metal pistons do not hit hard surfaces.

7.7 Usage of the ComPrep in a temperature chamber

The ComPrep is suitable for use in a climate chamber (temperature limits see chapter 6).

7.8 Usage of the ComPrep in a glovebox

The ComPrep is designed to work in a glovebox without any limitations to its main functions.

8 Maintenance

8.1 Cleaning

Clean the lower PEEK plate thoroughly before each use. Impurities, powder or dust could be pressed into the plate during use and damage it.

Clean the two spherical contact surfaces of the contact plate and the upper cell plunger with a swab and isopropanol or acetone before every measurement. This ensures the electrical contact as well as the validity of the force-torque-correlation, see 7.5.

Occasionally clean dust from the other surfaces of the ComPrep with a simple cloth and deionized water or isopropanol. Do not use any other solvents.

8.2 Lubrication

The spindle should always be lightly greased with clear all-purpose grease. The spindle should be cleaned and re-lubricated at least every 50 force cycles or as soon as the grease on the spindle turns black due to metal abrasion. This ensures that the force-torque-correlation remains valid, see 7.5.

The spindle can be cleaned with a cleaning cloth or cleaning paper and acetone or pentane.

Carry out the cleaning steps:

- Screw the spindle all the way down.
- Place the solvent-soaked cloth at the top of the spindle in a thread valley and turn the spindle upwards while continuing to press the cloth against the spindle.
- Screw the spindle up until the contact plate touches your fingers.
- Set the cloth down, screw the spindle down a little, reapply the cloth and clean the last threads in the same way.
- Then repeat the procedure on the spindle above the upper platform while turning the spindle down.
- When there are no more large black grease stains, cleaning is complete.

Carry out the re-lubrication steps:

• Screw the spindle all the way down.

- Apply a small amount of clear all-purpose grease to a cloth and spread it evenly over the exposed spindle.
- Screw the spindle all the way up and down several times to distribute the grease evenly on the thread. The contact plate should never hit the upper platform.
- Wipe the threads with a fresh cloth once as described in the cleaning steps to get rid of excess grease.

8.3 High contact resistances of the test cell

Thoroughly clean the spherical indentation of the contact plate, as well as the holes used for contact with a clean tissue and isopropanol. Make sure that no contaminant, especially particles are present on any part.

The piston surfaces can be cleaned by polishing with diamond paste 250 nm particle size.

Try assembling the cleaned and polished test cell without any sample but the 100 Ohm test resistor, apply a small force (max. 0,3 kN) with your fingers on the spindle head, and measure the contact again. You can also measure the contact between the individual parts, i.e. upper contact and upper plunger, cell bottom and upper plunger, etc., to narrow down the problematic contact.

If the issue can't be found or fixed, please contact rhd instruments for further instructions

8.4 ComPrep is making unusual noise when turning the spindle

Make sure the spindle is clean. Make sure there is no dust or particles between spindle and spindle nut. The issue can be resolved by cleaning and relubricating the parts.

9 Settlement

Warranty will be granted for a period of 2 years starting at the date of delivery.

Explicitly left out from warranty are parts that are subject to premature wear and tear due to use or other natural wear and tear. For example, insulation materials, nut or spindle damages due to high torque or damages on CompreCells as a result of unintended use. These components are regarded as consumables.

rhd instruments has to be notified of apparent defects and damages which occurred during production or delivery within 14 days after receiving the delivery. If a notification of apparent defects and damages does not occur within this period of time, the goods shall be deemed to have been accepted; as a result, the order will be assumed to be completed and approved.

Please note: Only workshops authorized by rhd instruments are allowed to perform repairs on the devices. If any mechanical or electronic components of the products are altered by customers themselves or by unauthorized workshops, a claim for warranty against rhd instruments is forfeited.

In case of a claim or sending back goods for repairs to be performed, please ask for the decontamination form beforehand. In general, rhd instruments must be contacted via e-mail or phone prior to any shipping of damaged goods.

10 Contact and Technical Support

For any questions with regard to our products, orders, or request for repairs please contact rhd instruments:

info@rhd-instruments.de

Phone: +49 6151 8707187

Fax: +49 6151 8707189

Web: https://www.rhd-instruments.com

rhd instruments GmbH & Co. KG

Otto-Hesse-Straße 19 / T3

64293 Darmstadt

Germany

Sitz der Gesellschaft: Darmstadt

Amtsgericht Darmstadt HRA 85824

WEEE-Reg.-Nr. DE 54715752

Haftende Gesellschafterin: rhd instruments Verwaltungs GmbH

(Sitz: Darmstadt, Amtsgericht Darmstadt HRB 96374)

Geschäftsführer. Dr. Benedikt Huber und Dr. Marcel Drüschler