Operator Quick Guide

ORBISPHERE 29981

HACH ULTRA™

EXCELLENCE IN PROCESS ANALYTICS

Revision D - 16/09/2008

Operating Information

About this Guide

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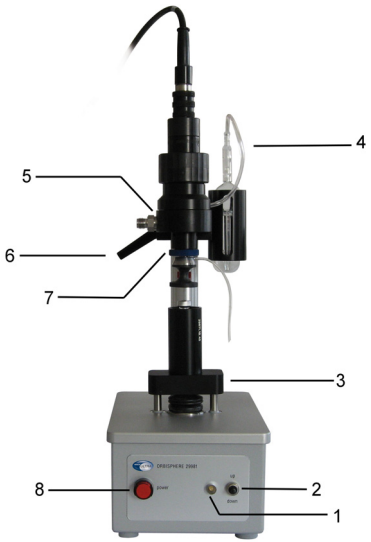
Sampler Description



The PharmaPack can analyze volumes as small as one milliliter, in ampoules and vials. The oxygen sensor makes headspace gas measurements and sends them to the analyzer for display. The package sampler is operator controlled. Position of the sample platform controls the flow of purge gas.

When the package moves up, the piercing needle assembly is pressed up against the bottom of the flow chamber, thus blocking purge gas flow by compressing the inner O-ring. Note the difference in the space and the compression of the O-rings between the flow chamber and the needle assembly.

Subsequently, when deaerated water is dispensed into the package, the headspace gas is forced up the inner needle, and into the flow chamber.

The arm (**5**) supports the oxygen sensor, the flow chamber, and the piercing needle assembly. A black handle (**6**), located at the rear of this arm, allows for height adjustment to fit the package size.

The motorized sample holding platform (**3**) moves up and down, controlled by the toggle switch (**2**) on the front panel. When the toggle switch is lifted, the platform stops automatically at a preset level. This is set by the adjustable red lever (**7**), located behind the piercing needle. When the toggle switch is lowered, the platform stops automatically at a factory preset level.

Two indicator lights are located on the front panel. A red light (**8**) illuminates when the power is turned on (switch on the back panel). The light (**1**), at the side of the toggle switch, is orange as the sample platform is raised, turns to green when the platform is lowered, and is off when the platform stops.

The oxygen sensor is mounted into the top of the flow chamber and the piercing needle assembly is attached to the bottom of the flow chamber.

A purge gas connection is located on the flow chamber side. An outlet tube (**4**) is connected to a small glass bubbler secured in a plastic holder, and which allows a visual check of the purge gas flow.

A deaerated water dispenser (not shown) is used for forcing the headspace volume into the flow chamber for measurement. This dispenser is connected by flexible tubing to the piercing needle assembly.

Preliminary Checks and Set Up

Check Deaerated Water Dispenser

Check that sufficient deaerated water is available in the dispenser before starting a measurement session.

For better results, make sure there are no air bubbles trapped in the tube attached to the PharmaPack.

Check the amount of water to be delivered with each shot is set correctly (the recommended quantity is 1mL).

If you purchased the model 28300 Orbisphere dispenser with your PharmaPack, lift the plunger to ensure it dispenses water correctly (no air bubbles) and returns automatically to the closed position. You may need to do this a few times until the plunger returns smoothly to the start position.

Adjust Package Support Ampoule Height

To adjust the ampoule package support to the height of the ampoule, place an ampoule inside the package support, with the flat end of the ampoule in the smaller (top) part of the support. Screw support together, finger-tight.

To adjust height, turn the Allen screw on the bottom of the package support (top picture) until the flat end of the glass ampoule is even with the top surface of the support (bottom picture).

Needle Assembly Check

Check the correct needle assembly is used. The package sampler uses two different piercing needles, one for ampoules and one for vials. If you use both, you must use the correct piercing needle assembly each time.

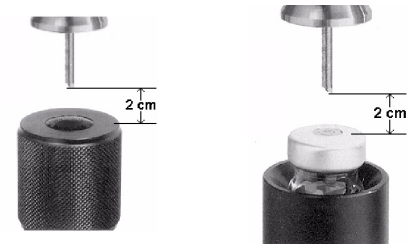
The piercing needle assembly for ampoules has a black knurled collar, while the piercing needle assembly for vials has a grey knurled collar.

Adjust the Arm Height

Lower the sample platform all the way and remove the rubber seal from the piercing needle.

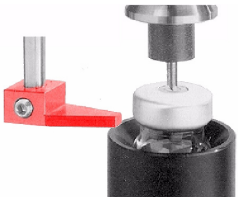
Place a package support on the sample platform.

- **Ampoules:** Leave the ampoule support empty, with both parts screwed tightly together.
- **Vials:** Place a sample vial into the package support.



Loosen the black handle and adjust height of arm until the needle is 2 cm above the top of the ampoule package support (left photo) or the vial septum (right photo). Then, tighten the handle.

Adjust Piercing Stop Height



When the toggle switch is raised to the **up** position, the platform is lifted and stops automatically at a preset height. This level is set by the position of the red lever, located behind the piercing needle.

Move the toggle switch to **up**.

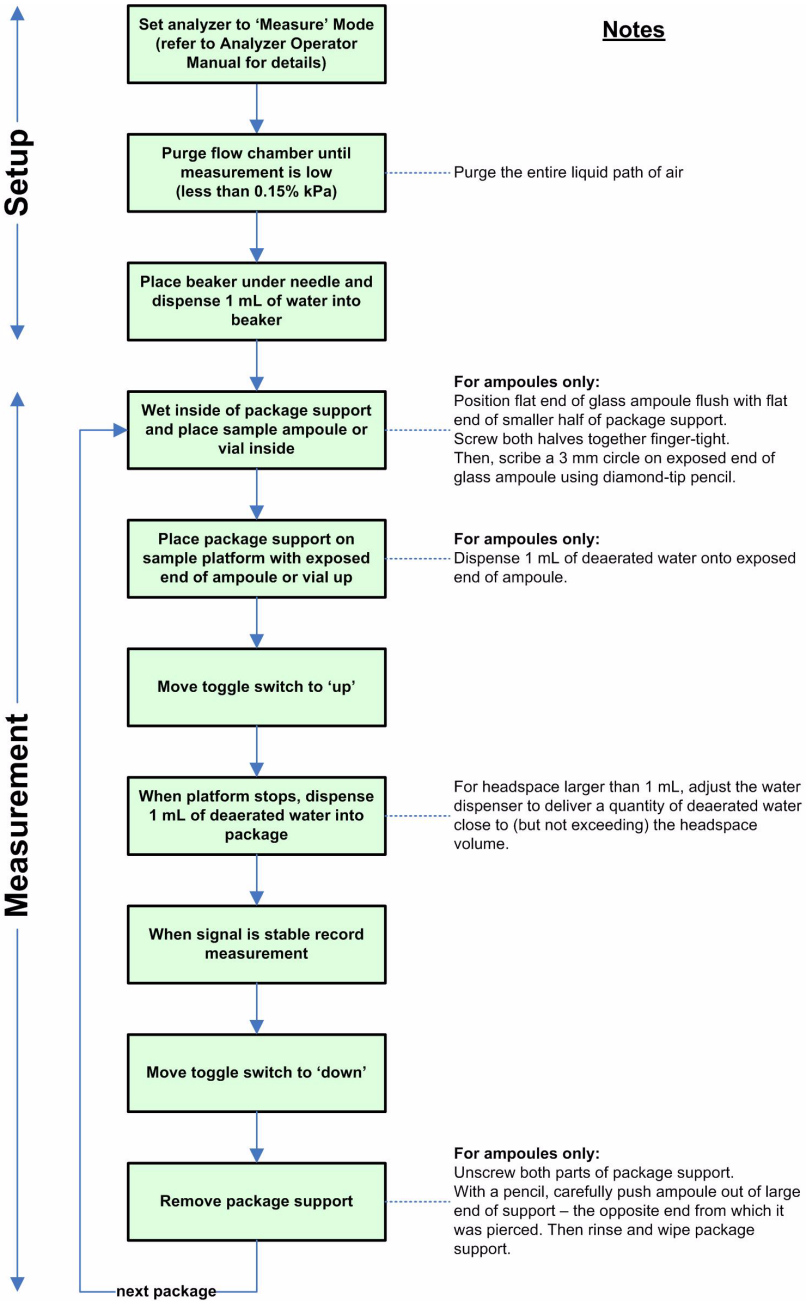
- **Ampoules:** When the top of the cutting edge of the needle is level with the top of the empty package support, stop the platform move by switching to **off**.
- **Vials:** When the needle start to pierce the septum of the vial, stop the platform move by switching to **off**.

Loosen the Allen screw on the side of the red lever (located on vertical post behind needle, see photo). Adjust the red lever until it just touches the top of the package support.

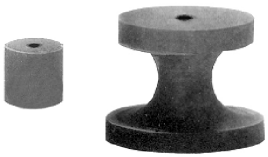
Tighten the Allen screw and reinstall the needle rubber seal.

If proper sealing cannot be achieved with the adjustment above, replace the needle rubber gasket. Lower the sample platform, remove and install a new rubber gasket around the needle.

Operating Sequence



Sampling Procedure



The needle assembly for vials has a grey knurled collar, while the needle assembly for ampoules has a black collar.

The rubber gasket used for vials is a cylindrical gasket (above left), while the H-shaped rubber gasket is used for glass ampoules (above right).

Procedure for Glass Ampoules

Setup

1) Switch the analyzer into **Measure** mode
2) Start the flow of purge gas from a cylinder of pure nitrogen. Check the flow rate in the glass bubbler, and verify that 2-3 bubbles appear each second.
3) Purge the flow chamber until the analyzer reading is very low (<0.10%)
4) Check that no air is present in the water tube or in the piercing needle assembly. To do this, place a beaker under the piercing needle assembly to collect water from the needle. Then, from the deaerated water dispenser, send 1 ml (one shot) through the water tube and piercing needle into the beaker

Measurement

Wet the inside of the two-part package support and place the sample ampoule inside.

The flat end of the ampoule must be flush with the flat end of the smaller piece of the support. To insure proper placement, put the support on a table and insert the ampoule carefully. Screw the support together, finger-tight.

Scribe a 3 mm circle on the exposed flat end of the ampoule with the diamond tip pencil provided. If you do not do this, the mechanical shock of piercing the ampoule could introduce sufficient air to generate a false reading.

1) Place the package support on the sample platform with the exposed end of the ampoule up
2) Send 1 ml of deaerated water through the piercing needle assembly, onto the exposed end of the ampoule in the package support. This water removes any air trapped between the ampoule surface and the sampling system
3) Move the toggle switch on the front of the sampler base to the **up** position. The indicator light turns orange
4) The sampling platform moves up and stops automatically at the correct level after piercing the ampoule. The purge gas shuts off automatically just before the needle pierces the ampoule (no gas bubbles should appear from the purge gas exit)
5) From the dispenser, send 1 ml of deaerated water through the piercing needle and into the ampoule. Watch for bubbles at the purge gas exit tubing
6) Once the signal on the analyzer display peaks, record the value. The signal will increase at first, then stabilize. You should record the value at the end of the increase slope, right at the beginning of stabilization
7) Move the toggle switch down. The indicator light turns green. The sampling platform is lowered and stops automatically
8) Remove the package support and unscrew the two parts
9) With a pencil, carefully push the ampoule out of the support and rinse and wipe the support with a paper tissue

Procedure for Vials with Rubber Septum

The package supports for vials are one piece. Place the vial, with septum up, into the package support. There is no setup needed, just let the needle pierce the rubber septum of the vial. The measurement process is then the same as for glass ampoules.

Troubleshooting

Using the Test Unit

The test unit (illustrated left) allows for a variety of tests without having to pierce actual vials or ampoules.

A purge gas inlet is situated at the bottom of the unit, with the outlet at the top.

Check for Purge Gas Leaks

To test for purge gas leaks, place a beaker of water on the sample platform. Then raise the platform so that the needle is immersed in the water (but the beaker is not pressing on the needle or gasket).

Start the purge gas and regulate the purge gas pressure to slightly more than atmospheric pressure.

When the purge gas is on, bubbles should be visible in the glass bubbler, and from the needle in the beaker. However, when the purge gas is off, the bubbling should stop.

The analyzer display should show a change of no more than 0.01 kPa per minute, otherwise a leak may be present and you should check all fittings.

Unable to Purge the System

If you observe no bubbles when the purge gas is on during the preceding test, check to see if either the piercing needle assembly is too tight or the sensor is improperly mounted on the flow chamber.

Sudden Jet of Water

A sudden jet of water can come out of the purge gas exit tubing if the purge gas pressure is too high. Excessive purge gas pressure can cause gas to leak into the flow chamber during the measuring phase, thus distorting readings. Adjust purge gas pressure and flow rate.

Check for Air Leaks

If you suspect an air leak somewhere in the flow, first make sure that your purge gas supply is running, and that your sensor is at baseline, that is, showing a typically low oxygen value.

Fill the test unit with deaerated water, full enough to form a protruding meniscus at the test unit's opening. Place the test unit on the sample platform and raise the test unit. When the rubber gasket forms a seal with the test unit, stop the platform.

After five minutes, check the instrument measurement. The oxygen value should remain constant, rising no more than 0.05% (kPa) during that delay. If the value increases significantly, then you may assume that the system is leaking. Check all appropriate fittings and try this test again.

Alternate Check for Purge Gas Leaks

The display value could also decrease in the preceding test if there is leakage of purge gas into the flow chamber. First, verify the presence of bubbles in the glass bubbler.

Then, place the test unit on the sample platform and raise the platform until the rubber gasket forms a seal with the test unit. Check to see that the bubbling stops.

If the bubbling does not stop, the leak is continuous. Disassemble the piercing needle assembly, and replace the small O-ring. (Do not forget to place a thin film of silicone grease around the O-rings before reassembling the piercing needle assembly.)

If the purge gas tubing "burps", the leak probably is due to purge gas pressure that is too high. Adjust purge gas pressure and flow rate.

Troubleshooting (cont)

Check for Water Leaks

Connect a source of high purity N_2 with a known concentration of O_2 to a needle to pierce the test units rubber septum. (Use N_2 with a concentration of O_2 close to the concentration that you will want to measure).

Insert the needle into the rubber septum, which is located near the bottom of the test unit.

Place the test unit on the sample platform and raise the platform until the rubber gasket forms a seal with the test unit. Check if the instrument measurement corresponds with the known concentration of O_2 in the gas.

Then, remove the needle from the test unit and check the measurement display. If the reading decreases with the removal of the needle, water is probably trapped in the flow chamber.

Purge the system with more N_2 and repeat the reading. If the decreasing reading persists, indicating that the flow chamber has not dried out, remove the sensor from the flow chamber and carefully dry the sensor face with a clean cloth.

Check Oxygen Values

Connect an oxygen supply to a regulator and a needle to pierce the test unit's rubber septum. Use a supply of known O_2 content, preferably of a value that closely matches your expected sample oxygen level, for example, 97% N_2, 3% O_2, to match a 3% expected sample value. You also may wish to install a three way valve ahead of the regulator to allow for alternate gas sources.

Insert the needle into the rubber septum located near the bottom of the test unit. Place the test unit on the sample platform, and run enough gas to purge the test unit cavity.

Then, raise the sample platform. As the test unit approaches the rubber gasket, drop the flow of gas to a trickle (0.01 to 0.03 bar) just before it makes contact. After the rubber gasket forms a seal with the test unit, the glass bubbler should show a few bubbles per second. Check if the analyzer measurement value matches the gas sample.

Check for Clogged Needle

Fill a small container with soapy water and place on top of the test unit.

With the purge gas running, place the test unit (with the container on top) on the sample platform.

Raise the test unit so that the needle is immersed in the soapy water but ensuring that the container does not come into contact with the needle assembly. Check for bubbles at the needle (as illustrated above). If bubbles show in the glass bubbler but not from the needle in the container, then the needle may be clogged.

Unable to Obtain Low Residual Value

If you have performed all the checks above and there are no air or water leaks, and you are still having problems in obtaining a low residual value, your sensor may need servicing.

Check Purge Gas Shutoff for Vials

Vial have different piercing characteristics, because of the septum shape and its material. These differences may require adjustment to the rubber gasket surrounding the piercing needle. When a sample vial is raised by the platform, two actions occur:

1) The vial is applied against the rubber gasket that surrounds the needle. This action pushes up the piercing needle assembly against the flow chamber. This stops the flow of purge gas to the flow chamber by compressing the inner O-ring. Purge gas flow is indicated by bubbles flowing from the exit tubing into a beaker of water. When the purge gas stops, there are no more bubbles.
2) The needle starts piercing the rubber septum of the vial.

For proper headspace gas measurements, the purge gas must stop flowing before the needle pierces the septum of the vial. You may need to adjust the height of the rubber gasket (shorten or lengthen it) to ensure that the purge gas flow stops right before the septum is pierced.

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