Application Note:

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Making OTR Measurements in Bottles using the

BottlePerm Fixture

Introduction

Measurement of oxygen transmission rate (OTR) of bottles can be complicated and time consuming. To make accurate OTR measurements, the bottle needs to be flushed with nitrogen so that there is little to no oxygen in the bottle. The bottle then needs to be sealed without any oxygen entering the bottle during the sealing process and through the seal.

In order to obtain the OTR of a bottle, the bottle must be sealed in such a way that that the only pathway for oxygen to enter in to the bottle is via permeation though the wall of the bottle itself. In order to measure the ingress of oxygen in to a bottle, it is best that the bottle be devoid of oxygen prior to the beginning of a test. The BottlePerm fixture is designed to facilitate the determination of the OTR of a bottle by providing a platform on which to seal and flush a bottle via gas-tight valves and fittings that make it possible to purge all oxygen from the package with a pure, inert gas.

The BottlePerm fixture is designed for use with an OxySense 5250i instrument. The 5250i instrument features the OxySense Gen III software which includes a module for package and bottle permeation testing providing OTR results reported in cubic-centimeters per package per day (cc/package/day). The BottlePerm fixture is equipped with an OxyDot® oxygen sensitive optical sensor. The OxyDot® resides behind a reading window and, after adhering and sealing the bottle to the BottlePerm fixture, effectively resides inside the bottle. Hence, the oxygen concentration inside the bottle can be determined by using the OxySense 5250i instrument to non-invasively read the OxyDot® through the reading window.

The flushing and sealing of the bottle can be easily achieved by using the BottlePerm fixture which is designed to be universal and can be used with a large range of bottle neck sizes regardless of the thread size. The BottlePerm fixture is shipped with an application kit that includes everything needed to adhere and seal the package to the fixture as well as a scraper to clean the residual adhesive from the fixture after the test is completed. Once cleaned, the BottlePerm fixture can be reused countless times. The BottlePerm fixture consists of a top plate that has an inlet and an outlet valve for flushing, a glass window with an OxyDot® and an adjustable bottom plate that attaches to the top plate and the bottle just below the threaded part of the neck. A gas tight seal is created by applying

Epoxy to the junction between the top plate and the top of the bottle. Once the epoxy has cured, the bottle can be flushed with nitrogen and the flushing process can be monitored by continually measuring the oxygen via the OxyDot® and the OxySense 5250i instrument.

Oxygen Permeation Measurements

Equipment required:

- OxySense 5250i instrument
- 4mm fiber optic extension (part 300350-502)
- BottlePerm fixture with OxyDot®
- BottlePerm fixture application kit
  - 5 minute epoxy with mixing spatula
  - Epoxy application tool
  - Epoxy scrapper
- Ultra High Pure (UHP) Nitrogen

Method:

- Attach the BottlePerm fixture to the bottle to be tested
- Turn the top plate so that it is resting on the valves
- Then place the bottle so that the opening of the bottle is centered on the OxyDot® and the inlet tube is inside the bottle (see picture below)

- Place the bottom adjustable plate as shown below with “V” just below the threads
- Move the screws on the top plate to the appropriate location for the size of the bottle

- Attach the wing nut to the screws as shown below so that the bottle is held firmly in the fixture (the wing nut should be finger tight only)

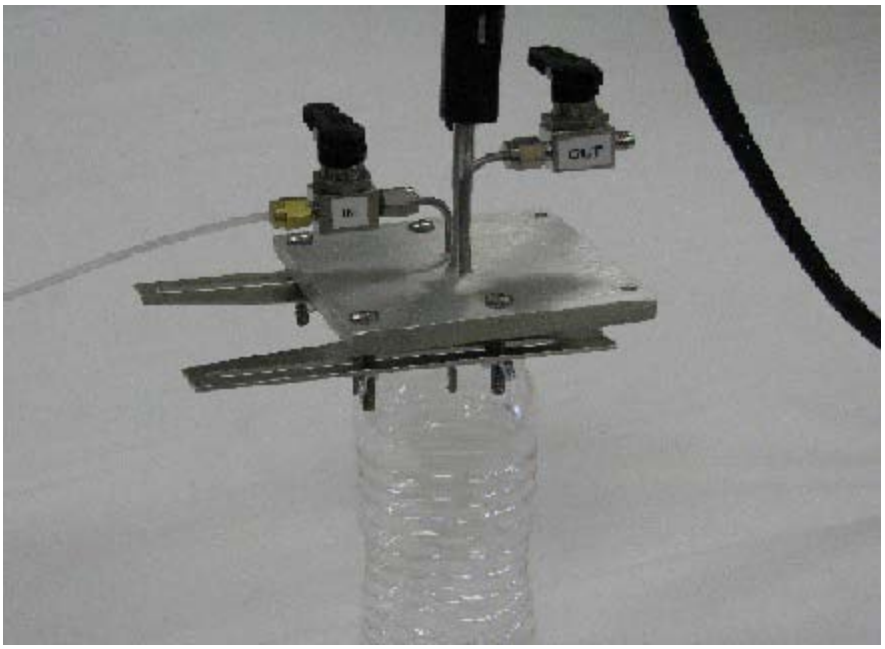
- Mix the epoxy in a suitable container

- Apply the epoxy to the top of the bottle touching the top plate as shown below (making sure to apply the epoxy all the way around the bottle)

- Allow the epoxy to dry for a minimum of 5 minutes (for best results allow 1 hour)

- Attach the fiber optic extension to the fiber bundle

- Attach a hose from the UHP nitrogen supply to the inlet valve of the fixture
- Place the fiber bundle with the adaptor on the BottlePerm fixture
- The bottle is now ready to be purged and used for OTR measurements.



Calibrating the OxyDot®

Although the OxyDot® on the BottlePerm fixture has been calibrated at the factory for the most accurate results, it is always a good idea to calibrate it again before using it for OTR measurements. The calibration process is simple. The atmosphere in the bottle after attaching the BottlePerm fixture is room air where the oxygen concentration is 20.8%. This can be used as the "high" value in the calibration process.

- Click on the calibration tab on the OxySense software
- Click on the "capture" button several time to make sure that oxygen concentration readings are stable (the reading should be close to 20.8%)
- Click on the "Get High" button
- Purge the bottle with UHP nitrogen
- Keep clicking the "capture" button during the purging and observe the oxygen concentration (this should be dropping)
- Once the observed oxygen concentration has reached its lowest point and is stable (it will fluctuate slightly) securely close the inlet valve first then securely close the outlet valve
- click on the "Get Low" button
- Click on the "Calculate" button (the new dA and dB numbers will appear in the "New" column)
- Click on the "Save" button (the new calibration will be entered in the software)
- Write the new calibration numbers down in a note book
- Check the calibration by clicking on the "Capture" the oxygen reading should be 0.00% +/- 0.03%
- The calibration is complete and the bottle is ready for OTR measurements

Once the OxyDot® has been calibrated and the bottle is purged with nitrogen, OTR measurements can begin. OTR measurements can be performed simultaneously on multiple bottles using the OxySense 5250i and multiple BottlePerm fixtures.

Making OTR Measurements

The OxySense package permeation software uses the technique of dynamic accumulation to determine the OTR of a bottle. The dynamic accumulation technique is one where oxygen that permeates into the bottle is allowed to accumulate within the bottle overtime and the OTR is calculated from the rate of increase of the oxygen concentration within the bottle. The OTR of multiple bottle can be determined using a single 5250i instrument by setting up unique tests in the OxySense "package Permeation" software for each individual bottle under test (detailed instructions can be found in the OxySense 5250i and BottlePerm manuals).

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