TPO and TPAir determination in beverages

Introduction

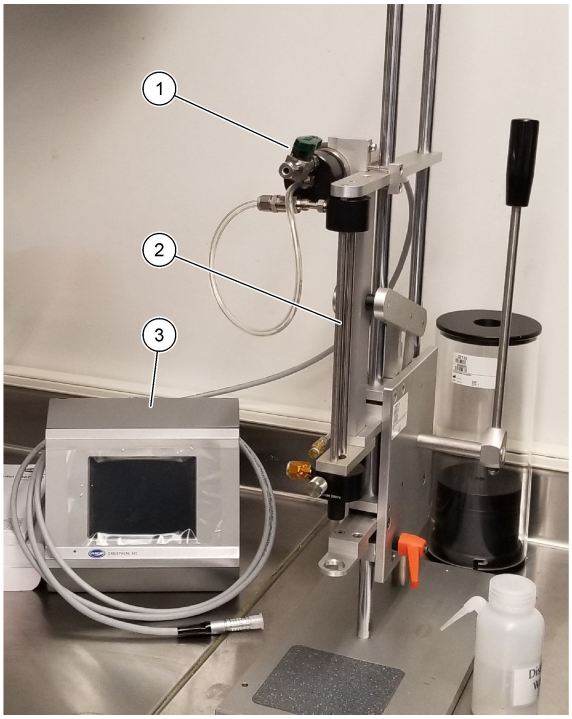
Use this procedure to determine the Total Package Oxygen (TPO) and Total Package Air (TPA) in beverages.

TPO is the sum of the dissolved and headspace oxygen (the mass of oxygen in the liquid plus the oxygen in the headspace, divided by the volume of liquid in the package). The TPO value is the oxygen in a package that can react with the beverage in the package.

TPA is the same as TPO but is given in air content. The TPA value comes from initial wet lab procedures that absorbed the CO_2 of the package. The volume of gas remaining was called “air”, which may not be correct because of the oxygen consumption by sensitive components or nitrogen being added to the product or nitrogen used in the production process.

Refer to Figure 1 for a system overview.

Figure 1 System overview



1 Flow cell with oxygen sensor	**3** Orbisphere Model 410 analyzer
2 Package sampler	

Test preparation

Instrument-specific information

Table 1 shows all of the items included in the DKM1100-TPO Beverage oxygen analyser kit.

Table 1 Instrument-specific information

Description	Item no.
Orbisphere 410 Controller O_2 (LDO), Panel Mount, 100-240 VAC, 0/4-20mA	410M/P1C00000
Power supply cable for 85 to 264 VAC wall and panel 410/51x instruments, 2 m, Europe	33031
Power supply cable for 85 to 264 VAC wall and panel 410/ 51x instruments (2 m), US	33032

Table 1 Instrument-specific information (continued)

Description	Item no.
Orbisphere M1100 online Luminescent dissolved oxygen analyzer for in-line applications, 0-2 ppm, with 28 mm Orbisphere fitting	M1100-S00
Handle for Orbisphere 510 Table	33107
Lower frame with 2 legs for Orbisphere 510 table	33108
Sensor cable to connect M/K-type sensor with Orbisphere 410/51x instruments, 3 m	32510.03
Beverage Package Sampler for M1100, grey	29972.M
Flow Chamber in Delrin for TPO solution	32001.M
Piercing seal tube gasket for 29972	28067
Bottom block sample seal tube, 3 pcs	28069
Bottle protection screen	32115

Items to collect

Description	Quantity
Analytical balance	1
Timer	1
Shaker (optional)	1

Before starting

⚠ DANGER

Multiple hazards. Only qualified personnel must conduct the tasks described in this section of the document.

Install the system. Refer to the documentation supplied with the sensor and controller (DOC024.98.93023), and the piercing device (DOC024.98.93010).

1. Connect the piercer to the forcing gas (CO_2, N_2) supply and install the liquid circuit.
2. Install the sensor in the flow chamber of the piercer and connect it to the controller.

Configure the controller on page 3

Calculate the overflow and net content volumes on page 3

Adjust the piercer on page 3

Flow rate on page 4

Configure the controller

Configure the controller with the settings that follow:

1. Go to MAIN MENU > MEASUREMENT > CONFIG. INSTRUMENT and select SAMPLE MODE.
2. Go to MAIN MENU > MEASUREMENT > CONFIGURE CHANNEL > SAMPLE MODEL and select TPO ENABLE and TPA ENABLE as necessary.
3. If TPA is selected, enter the K coefficient. The K coefficient is the O_2 / N_2 ratio in air. Default: 5

   Note: *The default value for the K coefficient is only applicable if the procedure does not add N_2 to the sample. Make sure to adjust the K coefficient as necessary. Determine the correct K coefficient with a comparative study with alternative procedures.*
4. Select STOP CRITERIA and configure the settings that follow:

   Note: *The TPO / TPA determination only completes when the measurement stops because one of the stop criteria occurs.*

   - **Type**: Stability
   - **Variation**: recommended between 2.0 and 5.0 ppb (test and trial)
   - **Depth**: 5 points
   - **Maximum time**: 300 s

Calculate the overflow and net content volumes

Calculate the overflow and net content volumes to determine the TPO and TPA. The overflow volume (Brimfull) is the volume of the package. The net content volume is the volume of liquid in the package. Calculate the volume of three packages (recommended).

1. Use a balance to weigh each package (a).
2. Remove all of the liquid in the package. Weigh the empty package (b).

   Note: *If the package is a bottle, make sure to weigh the cap with the bottle.*
3. Fully fill the package with water. Weigh the package (c).

   Note: *If the package is a bottle, make sure to weigh the cap with the bottle.*
4. Calculate the volumes as follows:

   a. Overflow volume = c – b
   b. Net content volume = (a – b) ÷ sample density
5. If the package is a bottle, the bottle weight can change. Calculate the average of the weight of replicates and record the value. Use the calculated value to determine the volumes.

Adjust the piercer

⚠WARNING

Pinch hazard. Parts that move can pinch and cause injury. Do not touch moving parts.

⚠CAUTION

Personal injury hazard. Glass components can break. Handle with care to prevent cuts.

Wear appropriate protective equipment and obey all safety protocols. Adjust the position of the piercer body to the type of package. Refer to the steps that follow and Figure 2.

There are two types of piercing knifes for the piercing head. Refer to the documentation supplied with the piercing device (DOC024.98.93010) to change the piercing knife.

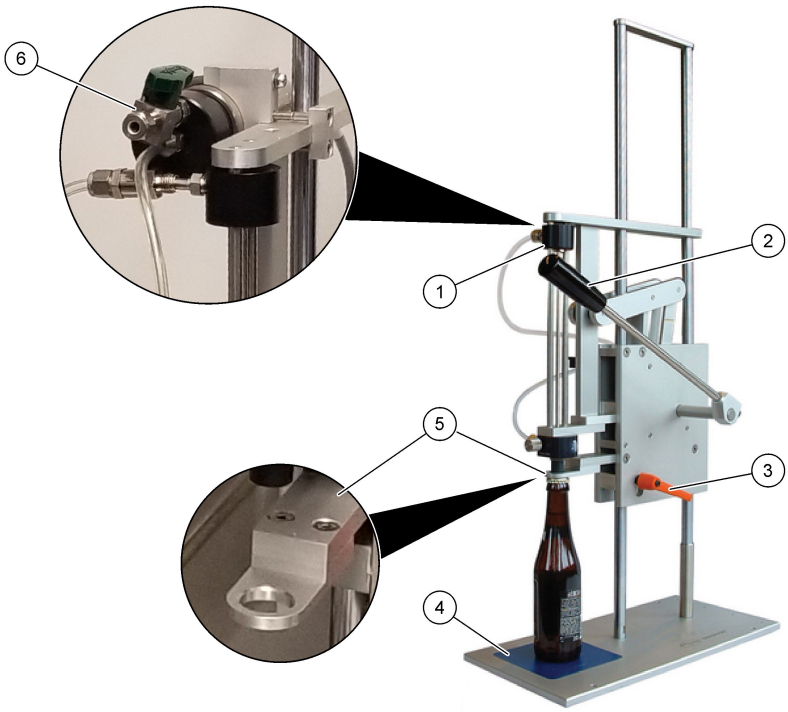
- 28072: Piercing knife for metal caps
- 29979: Piercing knife for PET bottles or plastic caps

1. Push up the black block to the highest position (1) to lift the piercing and sampling assembly.
2. Make sure that the piercing lever (2) is in a vertical position.

3. Hold the piercing unit sliding assembly with your hand and loosen the orange securing handle (3).
4. Move up the piercing unit sliding assembly to the highest position through the two metal columns.
5. Put the beverage package on the mat of the base plate (4). By default, the factory installed piercer head support (5) is used for plastic bottles. If the package is a plastic bottle, continue to step 6. For cans or metal caps, do the steps that follow:
   a. Use an allen wrench to remove the two bolts that attach the piercing head support (5) to the piercer body.
   b. Turn the piercer head support so that the down side is on top. Attach the piercing head suport to the piercer body with the same bolts.
      Note: *The bolts don't fully screw on the can configuration of the piercing head support.*
   c. For small package sizes, it is possible to put the can upside-down on the base plate.
6. Carefully lower the piercing unit until the piercing head support plate with its circular hole touches the top of the beverage sample (5). The top of the beverage sample must hold the piercing head support and prevent accidentally movement of the piercing head.
7. Lift the orange securing handle to the up position to tighten the handle.

The piercer is now adjusted to the type of package and prepared for measurement.
Note: *If the type of package changes (a bottle or can with different dimensions), do the adjust procedure again with the new package.*

Figure 2 Package adjustment



1 Black bock	**4** Base plate
2 Piercing lever	**5** Piercing head support
3 Securing handle	**6** Flow cell. Connect the inlet in the middle (center) and outlet on top (12 o'clock).

Flow rate

For a stable flow rate, add the recommended flow chamber (item no. 29971.M) and set the flow rate for dissolved oxygen measurements to 100–150 mL/min.

Procedure

TPO and TPA measurement

⚠WARNING

Pinch hazard. Parts that move can pinch and cause injury. Do not touch moving parts.

⚠CAUTION

Personal injury hazard. Glass components can break. Handle with care to prevent cuts.

1. Use a shaker to shake the package (can or bottle) to equilibrate the oxygen content between liquid and gas phases. Shake the package a minimum of 5 minutes.
2. Put the package on the piercer. Adjust the position of the piercer body to the type of package. Refer to Adjust the piercer on page 3.
3. Move the lever forwards so that the rubber and the knife of the piercer head touch the package.
4. Pierce the package and move the needle down. If the package is not pierced, adjust the piercer and do steps 2 to 4 again.
5. Open the forcing gas inlet. Make sure to use the correct piercing and back pressure. The typical piercing pressure for cans may be 25 psi and for bottles 30 to 35 psi. Use the correct pressure based on the type of sample and package (e.g., the package size and sample temperature). Make sure to always use the supplied bottle protector.

   Note: *Degassing can cause artificial low and noisy measurements. Carbonated or nitrogenated liquids must have sufficient back pressure to make sure that no degassing occurs in the flow chamber. Examine the tubing between the piercer and the flow chamber. Make sure the tube is clear and there is no active gas going out of the solution. If gas is seen coming out of the solution, adjust the piercing pressure or the back pressure with the flow valve.*
6. Start the measurement on the transmitter. The measurement stops when a stop criteria occurs.

   Note: *If OFF is pushed on the controller or the measurement does not get a stop criteria before the maximum time set in the controller configuration expires, "ABORTED" shows on the screen. A TPO / TPA determination is not possible for the sample. No data is saved in the controller.*
7. Enter the Overflow Volume and Net Content Volume in the controller. Refer to Calculate the overflow and net content volumes on page 3.
8. Push COMPUTE TPO / TPA. The result of TPO / TPA shows on the display. If necessary, do the calculation again with other volumes. Push OK to record the result.

   Note: *If CANCEL is pushed, the result is not saved.*
9. When the procedure is complete, close the forcing gas entry. Wait until the pressure in the package decreases.
10. Move the needle up and lift the lever to the vertical position.
11. Remove the package.

Maintenance

⚠WARNING

Multiple hazards. Only qualified personnel must conduct the tasks described in this section of the document.

Refer to the documentation supplied with the sensor and controller (DOC024.98.93023) and the piercing device (DOC024.98.93010) for additional maintenance tasks.

Clean the system

NOTICE

Do not use oxidizers (e.g., potable water) to clean the system while the controller is set to on. Drift, more frequent calibration and shorter cap life will occur.

Clean the system after a series of measurements is complete or before the system shutdown. Use dechlorinated water or water with no active oxidizers (e.g., ozone) to clean the system. If biologically active liquids (e.g., beer or kombucha) are measured, clean the system weekly with ethanol or isopropyl alcohol.

1. Fill a pierced package (1 L bottle or can) with non-chlorinated and warm water (40 ºC, 104 ºF). Keep the crown on the package.
2. Put the package on the piercing device.
3. Open the forcing gas inlet. Move the piercer head down.
4. Let the water flow in the system.
5. When the package is empty, move the piercer head up and close the forcing gas inlet.
6. Remove the package.

Note: *Contact your Hach representative or technical support for additional information about this procedure.*

HACH COMPANY World Headquarters
P.O. Box 389, Loveland, CO 80539-0389 U.S.A.
Tel. (970) 669-3050
(800) 227-4224 (U.S.A. only)
Fax (970) 669-2932
orders@hach.com
www.hach.com

HACH LANGE GMBH
Willstätterstraße 11
D-40549 Düsseldorf, Germany
Tel. +49 (0) 2 11 52 88-320
Fax +49 (0) 2 11 52 88-210
info-de@hach.com
www.de.hach.com

HACH LANGE Sàrl
6, route de Compois
1222 Vésenaz
SWITZERLAND
Tel. +41 22 594 6400
Fax +41 22 594 6499

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