Oxygen in the Brewery

Why, Where and How to Measure Oxygen

Beverage

orbisphere
www.hachultra.com

Introduction

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Oxygen Measurement and Control

This brochure is intended to enhance the brewer's knowledge of how dissolved oxygen levels can be monitored and controlled throughout the entire brewing process - from wort to final package - in order to help produce beer of a consistent quality and flavor.

We are witnessing, now more than ever, a heightened consumer awareness that the taste of beer noticeably degrades over time.

We know that improper amounts of oxygen present in various stages of the brewing process will greatly affect the final taste. Accurate oxygen analysis allows the brewer to control the process so that the shelf life of beer can be greatly extended.

Orbisphere is the world's largest manufacturer of dissolved oxygen, nitrogen and carbon dioxide analyzers for the brewing industry.

Illustrated is an **Orbisphere** electrochemical (EC) oxygen sensor.

Why Measure Oxygen

Beer contains many substances that react on exposure to oxygen. These "oxidation reactions" are greatly accelerated by warm storage, boiling, and/or pasteurization, since oxidation is more rapid at higher temperatures.

Neglect of proper oxygen levels can cause noticeable changes in taste and clarity of the final beer.

Care must be taken throughout the brewing process to minimize oxygen (air) pick-up. The last, and perhaps the most critical step, is the elimination of oxygen addition in the packaging operation.

All steps, from the fermenter to the package, must be taken into account since this oxidation is an additive process, resulting in the reduction of your product's shelf life expectancy and taste.

Why Measure Oxygen

In wort

In fermentation, oxygen is essential for adequate propagation of yeast cells. During the boiling operation, large amounts of oxygen are driven out of the wort so, once it has been cooled to the appropriate temperature, oxygen must be added by injection or aeration.

In traditional brewing operations, the wort is saturated to a desired dissolved oxygen level of about 6-9 ppm - although some modern strains of yeast can require even more, up to about 14 ppm.

Precise control is required when adding oxygen or air to the wort. Too much oxygen results in an undesired rapid and over-vigorous fermentation. This affects flavor and results in excessive yeast growth.

Overproduction of yeast is costly to the brewer since excessive beer losses in spent yeast are obviously undesirable.

Conversely, a lack of oxygen in the initial stages results in poor fermentation and could lead to an increased level of acetyl coenzyme A in the yeast cells. This in turn can produce higher levels of esters in beer and other undesirable off-flavors.

In brewing liquor

Modern deaeration equipment can produce blending water containing only 1 ppb dissolved oxygen.

In high gravity brewing, this capability significantly reduces the overall dissolved oxygen value that will be found in the beer after blending.

In beer

After one day of fermentation, dissolved oxygen levels will have fallen to less than 30 parts per billion (ppb), and now the yeast breaks down the sugars in solution, producing alcohol and other by-products.

Some of these by-products are extremely susceptible to oxidation, and so one objective for the brewer is to ensure that oxygen (air) does not contact the beer in its journey from fermenter to final package.

If beer containing unsatisfactory levels of oxygen is packaged, irreversible damage will be done to the flavor profile. Any beer that is filtered before packaging must be protected from oxygen since it no longer contains oxygen-consuming yeast.

British cask ale is commonly regarded as not requiring this protection since it still contains yeast, and traditionally ale has been served by allowing air to enter the cask as it is dispensed.

In practice, significant oxidation does occur and this, together with the effects of air-borne microbial contamination, leads to a very short shelf-life.

The most detectable off-flavor that arises from oxidation is a "cardboard" or "wet paper" taste produced by the reaction of polyphenols.

Careful beer handling in the brewery can result in packaged dissolved oxygen values of less than 100 ppb. At this level the shelf life will be greatly extended.

Where to Measure Oxygen

In wort

Once in the brew kettle, boiling removes dissolved gases. This deaerated wort is then normally cooled to around 10° C to 15° C before transfer to the fermenter. Yeast is then added together with oxygen to allow yeast growth.

Air or oxygen?

Air contains roughly 4/5th nitrogen and 1/5th oxygen.

If air is injected into the wort in an unpressurized fermenter, the wort will at most contain 6-10 ppm oxygen, depending on the temperature.

If pure oxygen is injected and allowed to saturate the wort, then dissolved oxygen levels could rise to 30-50 ppm.

Modern brewery practice has seen a large changeover to pure oxygen addition.

The tables that follow give the basis for this rationale.

Air injection - Advantages	Air injection - Disadvantages
• Compressed air is inexpensive. • It will saturate to approximately the level required by the yeast, although dissolved oxygen should still be measured to ensure consistent fermentations.	• Air must be sterilized. • The large volume of N_2 introduced with the air is very difficult to fully dissolve and will pass through the fermenter, causing thick top foams. • Aromatic flavor compounds can be sparged from the wort by these bubbles.

O_2 injection - Advantages	O_2 injection - Disadvantages
• Cylinder oxygen is free from microbes. • Only the quantity of oxygen required for the fermentation needs to be injected, to reduce energy costs. • No large "nitrogen foams" will be created in the fermenter. • Concentration levels are adjusted easily and accurately. • Since oxygen is very insoluble, usage costs are very low.	• Extremely high levels of dissolved oxygen are possible unless a feedback control system from a dissolved oxygen analyzer is used.

Where to Measure Oxygen

CO_2 from fermenter

CO_2 leaving the fermenter to be collected should be checked with an oxygen analyzer to ensure all the air is vented off.

The graph illustrated below is a real example, measured in-line before the filler buffer tank, showing the effect of poor bright beer tank changeovers where air had not been flushed from the line connecting to the new tank.

The oxygen spike only lasted for 10 seconds, but was enough to cause the dissolved oxygen in the buffer tank to increase from 10 to 65 ppb.

Purge empty vessels before beer transfer

A major source of air contamination in bright beer occurs when it is transferred between vessels.

All pipes and filters should be filled thoroughly with water and all vessels purged with pure N_2 or CO_2 before receiving beer.

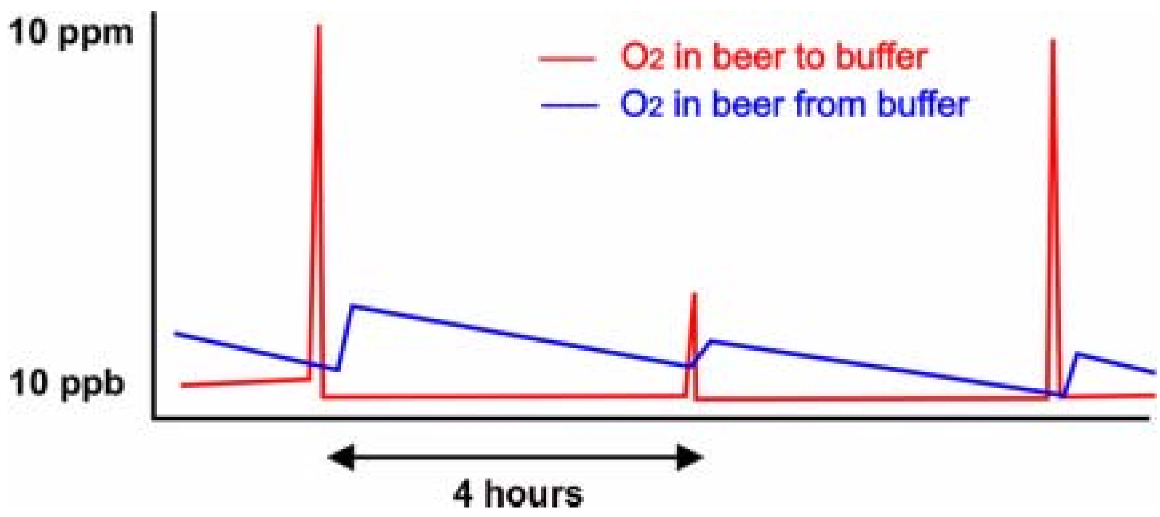
In small breweries it is possible to water pack the vessels and drain them under N_2 or CO_2, but this is not feasible in large breweries due to the volume of water needed.

Bright beer

After every tank transfer, or operation such as filtration, the beer should be checked to ensure it is not picking up dissolved oxygen.

The major source of air contamination is inadequately purged vessels. Leaking pump glands, leaking valves, and filter aid dosing pumps can also allow air ingress.

By measuring throughout the process it is possible to identify the source of any air contamination.



Where to Measure Oxygen

In package

The packaging process can be another major source of air ingress, and care needs to be taken at this time to make sure air is excluded.

Kegging can usually be carried out successfully without air ingress, but of course the keg must be evacuated to remove air before being filled with beer and the keg line counter pressured with oxygen-free CO_2 or N_2.

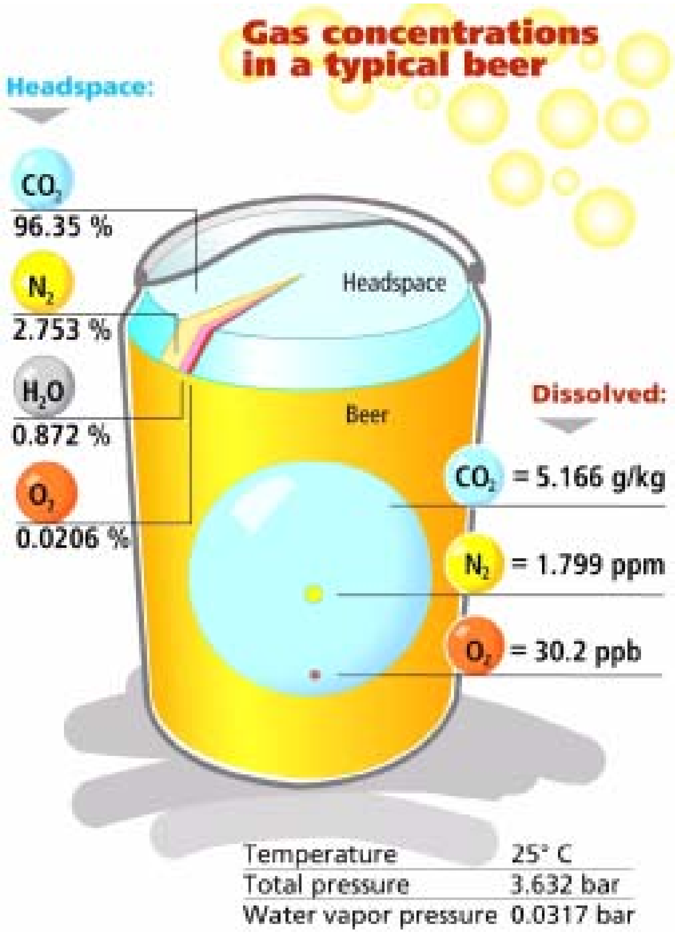
Canning requires the filler bowl to be counter-pressured with CO_2 or N_2. As beer goes into the can, the filler must be of a design where the air being displaced does not return to the filler bowl.

As the can leaves the filler, a drop of liquid nitrogen can be added to drive off the headspace air or, alternatively, an inert gas can be used to purge the headspace before the lid is fitted at the seamer.

Bottles provide a similar challenge, but since they are a rigid package they can be pre-evacuated before filling and a fine needle jet of water can be injected into the filled bottle to promote fobbing. As the beer fobs on its way to the crowner, air in the headspace will be driven out.

Illustrated below is a schematic of the measurable gases found in a typical can of beer.

Modern sampling techniques make it possible to measure total package oxygen, nitrogen, and carbon dioxide in a single package.



Where to Measure Oxygen

Tank purging

When purging tanks to remove oxygen, it is best to use a low pressure gas supply and to control the gas flow with valves on the inlet and outlet. This allows the purge gas to gently fill the vessel, and minimizes gas wastage.

When using CO_2 as the purge, always fill from the bottom and vent at the top of the tank since CO_2 is considerably denser than air.

If the purge gas enters too rapidly, usage will be very high and turbulence in the tank may even extend the purging time.

Typical acceptable oxygen levels after purging are in the range of 0.2% - 0.5% or less.

A slow, controlled purge gives the best results.

CO_2 recovery from fermenters

The gas leaving the fermenter initially will be the headspace air driven out as the fermentation starts to produce bubbles of CO_2.

Since fermentation times often vary, it is necessary to measure the oxygen concentration before collecting the CO_2.

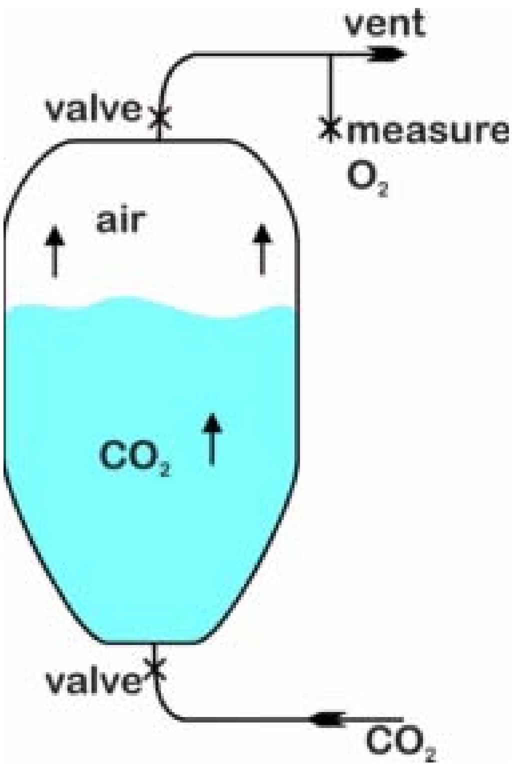
A widely held belief is that subsequent compression (and liquefaction) of the collected CO_2 will give an oxygen-free gas.

Liquefaction will certainly greatly reduce air contamination but unfortunately oxygen is very soluble in liquid CO_2, and thus it can still be carried through into the final CO_2 storage vessel.

Ideally, start collecting CO_2 from the fermenter when the oxygen content falls to 0.5% - 1%. Once this CO_2 has been compressed, use it only for tank flushing.

Some 12 hours after beginning to collect CO_2, the gas will be very pure.

After compression it can be used for injection into beer, or as the blanket gas in the filling operation.



How to Measure Oxygen

In wort

Hot wort is normally cooled in a plate and frame heat exchanger.

Formerly, air or oxygen was injected into the hot wort to ensure dissolution within the highly turbulent plate spaces.

This is rarely done now because it can lead to severe oxidation off-flavors in the wort. Oxygen can be injected into the bulkhead, which separates the regeneration section from the final trim section, since this will force the oxygen into solution in the final few plates after the wort has been cooled (option 2 below).

Air injection is less common here because the volume of gas is too large.

If gas is injected after the heat exchanger, it is necessary to use a very fine sinter (option 3) to assist in dissolution, but even this is often not totally successful.

Air injection can be done in this way, but as discussed earlier, it often leads to excessive head foams in the fermenter.

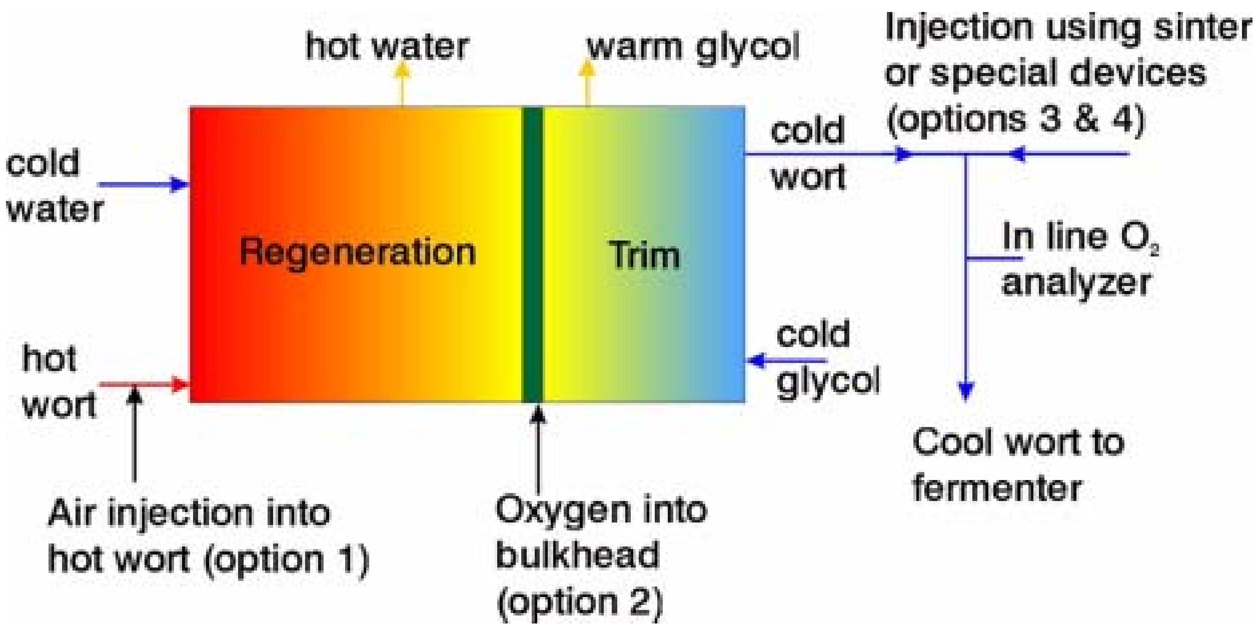
Modern devices (option 4) have now been developed to ensure total gas dissolution. They are based on proprietary designs with extremely high internal pressures and turbulence.

Oxygen is best used in these new devices to avoid excessive gas volumes. The practice in a modern brewery is to very tightly control dissolved oxygen (±0.1 ppm) and to use only just enough.

This ensures consistent fermentations and minimum beer loss in excess yeast.

Because of particulates and the need for feedback control, it is preferable to use an in-line oxygen analyzer for measurements in wort.

Orbisphere offers a specially designed wort flow chamber (Model 32007W) for use with its portable oxygen analyzers.



How to Measure Oxygen

Bright beer

Typical dissolved oxygen values vary widely between breweries, but values should be less than 0.2 ppm.

Whether measuring in bright beer or wort, it is imperative that all gases are in solution before oxygen is measured by the analyzer.

Insoluble gases such as oxygen are extracted by bubbles, which would artificially reduce the dissolved concentration value.

Portable measurements

When sampling beer, always fully open the pipeline sample tap and regulate the beer flow using the flow control valve on the outlet side of the **Orbisphere** flow chamber.

In this way the sensor will always be at the beer line pressure, and although it does not matter if the beer leaving the flow chamber is degassing, this will ensure that beer on its way to the sensor is clear and bubble-free.

Flow rate through the flow chamber is not critical but should be above a minimum value of 300 ml/min.

Note that changing the flow rate will cause a temporary change in the displayed value, due to a change in pressure on the sensor membrane. If left for a further minute, the original value will return (unless there is a leak in the sample tubing or if gases are coming out of solution).

Always switch on the analyzer a few minutes prior to sampling, since allowing the sensor to polarize for a few minutes before taking the measurement will improve the response time.

Be aware that the first measurement of the day will have a slower response, since the sensor must rid itself of any air accumulated under the membrane and must also adjust to the beer temperature.

To track down the source of oxygen contamination, the portable **Orbisphere** Micro O_2 Logger can be used for spot checks but also has the facility to be left on the sample point for several hours in its "logging" mode.

At the end of the day these results can be downloaded to a computer and viewed graphically to see if any oxygen pick-up has occurred.

When particular points in the process are identified as frequent sources of oxygen ingress, then in-line analyzers can be installed downstream from that point to provide permanent monitoring.

How to Measure Oxygen

Orbisphere Model 3650 Micro O_2 Logger

Expected oxygen levels throughout the brewery	
In wort	6 - 14+ ppm
Fermentation	< 10 ppb
Filtration	5 - 150 ppb
Bright beer after filtration	10 - 200 ppb
Beer at the filler	20 - 200 ppb
Package dissolved O_2 (bottle)	30 - 250 ppb
Package dissolved O_2 (can)	20 - 120 ppb
Total package dissolved O_2	50 - 450 ppb

How to Measure Oxygen

In-line analysis

Bright beer is a very expensive product; if it is damaged by oxidation, those effects cannot be reversed. Because of this, it is vital to continually monitor the process in order to give an immediate warning should oxygen pick-up occur.

Sensor installation can be made into most parts of the beer line, including near bends and valves, but must be sited as far as possible away from pumps (particularly avoid siting just downstream of them).

Install sensors into horizontal or rising mains, never falling mains.

Always install the sensor so that it lies horizontally. It is particularly important never to put any sensor, for oxygen or otherwise, vertically into the top of a pipeline since an air pocket could be trapped here and effective CIP would be impossible.

Sensors are fitted into pipelines, not tanks, since the minimum beer flow required across the sensor is 0.5 meters/second.

All **Orbisphere** systems are designed to withstand hot (99°C) or chemical cleaning-in-place without damage or performance loss.

Illustrated below is the ProAcc insertion device, which allows sensors to be removed for maintenance while the beer flow continues.

The latest **Orbisphere** analyzers allow the customer to select fast membranes (for rapid response to oxygen "spikes") or standard membranes (for extended time between maintenance).

These new systems also allow a selectable thermal cut-off, and by setting this value to a low temperature, just above that of the beer, the sensor will automatically switch off if the line is empty or is being cleaned. Use of this feature will greatly extend the routine maintenance cycle.

How to Measure Oxygen

Package analysis

Target dissolved oxygen values vary, but generally, values should be less than 0.2 ppm. Many breweries now achieve in-package oxygen levels of less than 0.1 ppm.

Samples are obtained from kegs by applying a gas top pressure of CO_2 or N_2 to the keg to drive the beer out through the normal spear fitting, to the **Orbisphere** flow chamber.

Samples are obtained from bottles or cans with the model 29972 package piercer (illustrated below), once again applying top gas to drive out the beer.

Systems are also available to measure total liquid and headspace O_2, CO_2, and N_2 in a single package.

Important points:

1) The applied CO_2 or N_2 pressure must be higher than the pressure of the total dissolved gases in the beer, to prevent bubble formation.
2) Always measure packages immediately after filling and before pasteurization, because the heating process allows dissolved oxygen to react rapidly with the beer.
3) Always shake packages vigorously before piercing. This allows formation of smaller bubbles and provides for a more rapid equilibration.

Note :
If measuring glass bottles, always make sure that a protective screen or other device is put in place before top gas pressure is applied, in case the bottle is flawed.

How to Measure Oxygen

Oxygen in the package vs. shelf life

Oxygen trapped in the package contributes to flavor degradation in beer. The greater the amount of oxygen, the greater the flavor degradation.

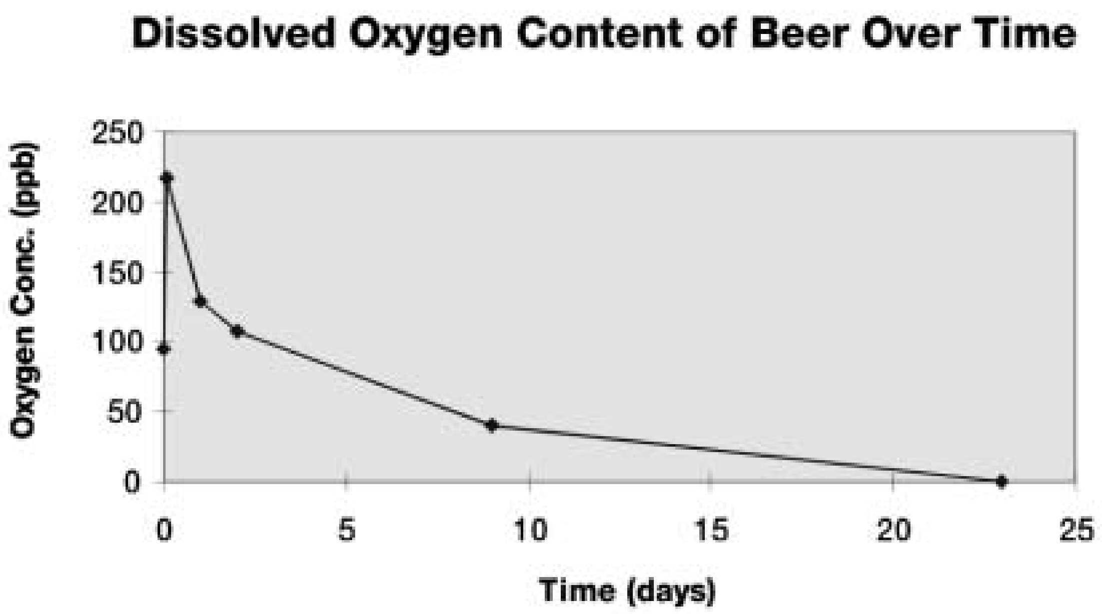
The oxygen content of a package can be measured to determine if the origin of the oxygen is from the headspace, or from the filling operation.

The most common flavor change attributed to oxygen exposure is a "wet cardboard" taste caused by the formation of the compound, trans-2-nonenal.

Beer consumes the oxygen in the package over time. In most beer, the oxygen is consumed over the period of one to six weeks, but the flavor doesn't change for two to three months.

Below is a graph of the oxygen concentration of a package for 4 weeks after packaging. In the case of the first measurement, the unshaken dissolved oxygen level was 95 ppb.

When the container was shaken, the dissolved content increased to 220 ppb. This indicated that the major contribution of oxygen was in the headspace. The bottles were then stored at room temperature and shaken before subsequent measurements were taken.



How to Measure Oxygen

Oxygen in the package

Finding the source

Oxygen is introduced into the package in two places: at the filler, or in the headspace.

Filler oxygen can come from oxygen already in the beer or from air trapped in the bottle or filler tubes during filling.

Headspace oxygen comes from air trapped in the headspace between the filler and the crowner or lid seamer.

Because the partial pressures of gases in the headspace and the liquid are not at equilibrium immediately after packaging, all packages must be shaken before measuring dissolved O_2 (or N_2 and CO_2).

Typically, the largest concentration of oxygen is found dissolved in the beer.

The following steps can be used to determine whether the filler or the jetter is the major contributor of oxygen.

All measurements should be taken on unpasteurized beer. As much as 60% of the oxygen can be consumed during pasteurization.

1) Pull a dozen packages off of a moving filler that is free from starts and stops.
2) Warm the packages to room temperature.
3) Measure six of the packages after shaking them for two minutes.
4) Measure six of the packages that have not been shaken.
5) Compare the dissolved O_2 concentration of the average measurement from each group.

If the value goes up after shaking, the major source is in the headspace. If the value goes down, as is typical, the major contribution is from the liquid.

From this it can be determined if the jetter or the filler is the source of the overall oxygen content.

How to Measure Oxygen

Oxygen exposure after packaging

Crown closures and O_2 ingress

After packaging, beer sold in bottles is still susceptible to oxygen contamination due to the properties of crown closures. Crown closures provide a semipermeable barrier between the beer in a bottle and the air outside the bottle.

The oxygen content in the bottle is significantly lower than the oxygen content of the air outside the bottle. Through passive diffusion, both oxygen and nitrogen leak into the package.

With the exception of newer oxygen scavenging and barrier crown closures, little can be done to eliminate the closure leakage.

The oxygen then reacts with the beer in the bottle and effects the flavor of the beer. This leakage or ingress tends to equilibrate the partial pressures of the gases both inside and outside the bottle.

Because the oxygen in the bottle is constantly reacting with the beer, the oxygen content of the bottle remains very low.

Typical crown closures "leak" 1 to 2 ppb of oxygen into the package per day. Over a three month period, the cumulative amount can be as much as 180 ppb of oxygen ingress. This ingress can be greater than the total oxygen exposure of the beer before bottling.

Considering the great strides that have been made over the last few years to reduce the total oxygen content of the beer during packaging, some breweries now have a total oxygen package content of less than 25 ppb.

The **Orbisphere** 3625 Package Sampler, or the 3650 Micro O_2 Logger with a 29972 Package Sampler, can quickly measure the dissolved gases in the package.

This can help you better understand the total oxygen exposure of your beer during the packaging process.

How to Measure Oxygen

Gas phase analysis

Because the **Orbisphere** system uses a membrane-covered sensor, it can be used just as easily to measure oxygen in gases or liquids.

Many **Orbisphere** oxygen analyzers offer "dual phase" capability, allowing easy switching between dissolved and gas phase display.

Others are dedicated to displaying gas phase results in a variety of choices of units.

Most include the possibility of measuring and compensating for sample pressure.

Since gases are compressible, the sampling technique is different from the procedure for liquids.

To measure volume %O_2 you must ensure the gas is at atmospheric pressure by using the following method:

- Reverse the flow, so that the flow valve is on the inlet side.
- Remove the check valve.
- Use a gas sample flow of 100 ml/min or less.

At the carbonator

When adding CO_2 to beer, the CO_2 added must contain virtually no oxygen since large weights of CO_2 are being added under high pressure and could rapidly lead to a build up of dissolved oxygen.

Adding carbon dioxide at high pressure

Try to ensure that the injection apparatus forces all the gas into solution immediately. Otherwise, the true gas concentration will be difficult to measure and volatiles in the beer could be lost into the receiving tank headspace.

Amount of added CO_2	Concentration of O_2 impurity in CO_2		
	0.001%	0.005%	0.02%
1 g / liter (0.5 vol.)	7 ppb	35 ppb	142 ppb
2 g / liter (1 vol.)	14 ppb	71 ppb	284 ppb
4 g / liter (1 vol.)	28 ppb	142 ppb	567 ppb
	Dissolved oxygen added to the beer		

Orbisphere Analyzers for the Brewing Industry

In-line process analyzers

Model **510/410** series analyzers offer a precise and powerful process monitoring capability.

Designed to complement Orbisphere's high quality oxygen sensors, these instruments provide accurate, repeatable trace level measurements and an impressive level of data management.

Model **3610** series single channel carbon dioxide and nitrogen analyzers' unique membrane-covered dynamic thermal conductivity method ensures accurate measurement with no effect from other gases. And it's virtually maintenance free.

Model **3620** series two channel analyzer. Measure two gases simultaneously: Choose any combination of CO_2, N_2, and O_2. All the benefits of the single channel analyzers in a cost-effective package.

Model **3660** series oxygen analyzers combine economy with accuracy, with internal data acquisition, real-time clock, user selectable membranes, and measurement down to 0.1 ppb.

Orbisphere Analyzers for the Brewing Industry

In-line process analyzers

Model **3624** ProBrix Plus. For process CO_2 °Brix (and %Diet), and O_2 measurement. Fast in-line beverage analysis saves time and reduces product waste.

Portable & package analyzers

Model **3650** Micro O_2 Logger. Combines accuracy and reliability with on-board data logging. Fast first response, with no additional "warm up" times, for heavy duty daily use.

Orbisphere Analyzers for the Brewing Industry

Portable & package analyzers

Model **3654** Micro CO_2 Logger. Ideal for quality control and for confirming process instrumentation measurements. Outstanding performance and simple operation.

Model **3625** Package Analyzer. For the ultimate in quality control: measure dissolved and headspace O_2, N_2 and CO_2 in a single pierced package.

A PC interface supplies on-screen operator prompts and logs all data for statistical analysis.

Model **3640** Spectrac Analyzer. Measures a wide range of gases and vapors, including alcohol and CO_2, using an exclusive photo-acoustic spectroscopy method.

Useful Hints and Conversions

Oxygen

- At 20°C dry air contains 20.94% O_2 = 209,400 ppm by volume.
- 100% humid air contains 20.45% O_2 = 204,500 ppm by volume.
- In solution, 1 mg/kg O_2 is often called 1 ppm (by weight).
- All the following solubilities assume a pressure of 1 atmosphere.

  When water is saturated with air it will contain:

  9.10 ppm O_2 at 20°C (68°F) : 14.64 ppm O_2 at 0°C (32°F).

  Therefore, considering oxygen measurements at 20°C:

  204,500 ppm by volume is equivalent to 9.10 ppm by weight.

  When water is saturated with pure oxygen it will contain:

  43.45 ppm O_2 at 20°C (68°F) : 69.90 ppm O_2 at 0°C (32°F).

Carbon Dioxide

- Dry air contains approximately 0.03% CO_2.

  1 volume CO_2 per volume of beer = 1.98 grams / kg at 20°C.
- Carbon Dioxide is far more soluble in water than oxygen.
- When water is saturated with CO_2 at 1 atm pressure it will contain:

  1.72 g/kg CO_2 at 20°C (68°F) : 3.37 g/kg CO_2 at 0°C (32°F).

Nitrogen

- Dry air contains approximately 78% N_2.
- In solution, 1 mg/kg N_2 is often called 1 ppm.
- Nitrogen is less soluble in water than oxygen.
- When water is saturated with air it will contain:

  15.3 ppm N_2 at 20°C (68°F) : 23.2 ppm N_2 at 0°C (32°F).
- When water is saturated with nitrogen it will contain:

  19.7 ppm N_2 at 20°C (68°F) : 29.8 ppm N_2 at 0°C (32°F).

Pressure

- 1 atm absolute = 1013.25 mbar = 1.013 bar = 760 torr = 0 atm gauge.
- All pressures for solubility data above are in absolute units.

Packages

- In a typical small package, the same weight of oxygen will be found in 15 ml of headspace as in 440 ml of beer. Thus, shaking is required to ensure equilibration before analyzing.
- Always shake very vigorously, as this will cause the formation of very small bubbles, which equilibrate much faster than large bubbles.

Notes

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