

# HOW TO MEASURE DISSOLVED OXYGEN IN THE BREWERY

## In Wort

Techniques 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 ( $\pm 0.5$  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 analyser for measurements in wort.



## Bright Beer

Typical dissolved oxygen values vary between breweries, but values should be less than 0.05 ppm. Whether measuring in bright beer or wort, it is imperative that all gases are in solution before oxygen is measured by the analyser. Soluble gases such as oxygen are displaced by bubbles, which artificially reduce the dissolved concentration value.



## Portable Measurements

When sampling beer, always fully open the sample valve and regulate the beer flow using the flow control on the outlet side of the instrument. In this way the sensor will always be at the beer pressure, and this will ensure that beer on its way to the sensor is clear and bubble-free.

Flow rate through the instrument is not critical, but it should be low enough that there is no degassing at the oxygen sensor.

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

To track down the source of oxygen contamination, the portable 3100 Dissolved Oxygen Analyser can be used for spot checks but also has the facility to be left on the sample point for several hours as a portable data logger.

### ORBISPHERE 3100 Portable Dissolved Oxygen Analyser



#### Industry standard oxygen levels throughout the brewery

In wort	8 – 17+ ppm
Fermentation	< 10 ppb
Filtration	5 – 50 ppb
Bright beer after filtration	10 – 50 ppb
Beer at the filler	10 – 30 ppb
Package dissolved O <sub>2</sub> (bottle)	20 – 50 ppb
Package dissolved O <sub>2</sub> (can)	30 – 60 ppb
Total package dissolved O <sub>2</sub>	40 – 150 ppb

## In-line Analysis



Finished beer is a very expensive product; if it is damaged by oxidation, these effects cannot be reversed. It is recommended to continually monitor the process in order to give an immediate warning should oxygen pick-up occur.

Oxygen sensors can be installed in most parts of the beer line, but must be placed as far away as possible from pumps, CO<sub>2</sub> injection or wort aeration sites.

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.

Hach instrumentation allows selectable thermal cut-offs. 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.

## Package Analysis

The Target dissolved oxygen values vary, but ideal concentration should be less than 0.5 ppm.

Samples are obtained from kegs by applying a gas top pressure of CO<sub>2</sub> or N<sub>2</sub> to the keg to drive the beer out.

Samples are obtained from bottles or cans with a package piercer, by pressurising the head space to push the beer past the oxygen sensor. (Systems are also available to measure total liquid and headspace O<sub>2</sub>, CO<sub>2</sub>, and N<sub>2</sub> in a single package.)

### Important points:

- The applied CO<sub>2</sub> or N<sub>2</sub> pressure must be higher than the pressure of the total dissolved gases in the beer, to prevent bubble formation.
- Always measure packages immediately after filling and before pasteurisation, as the heating process allows dissolved oxygen to react rapidly with the beer.
- Always shake packages vigorously before piercing to equilibrate the head space and dissolved gases.



## Shelf Life

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

In most beer, the majority of the oxygen is consumed over the period of one week, but the flavour doesn't change for two to three months. Oxygen consumption of packaged beer can vary greatly, depending upon the storage temperature, type of beer and beer yeast content.



## Oxygen in the Package

Oxygen is introduced into the package in two places: during filling or in the headspace through incomplete fobbing. Filler oxygen can come from air 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 after the closure is applied. Because the partial pressures of gases in the headspace and the liquid are not at equilibrium immediately after packaging, all packages should be shaken before measuring dissolved O<sub>2</sub> (or N<sub>2</sub> and CO<sub>2</sub>).

The following steps can be used to determine whether the filler or the jettler is the major contributor of oxygen. All measurements should be taken on unpasteurised beer.

1. Pull six packages off of a moving filler that is free from starts and stops.
2. Measure three of the packages after shaking them for five minutes.
3. Measure three of the packages that have not been shaken.
4. Compare the dissolved O<sub>2</sub> 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, the major contribution is from the liquid. From this it can be determined if the jettler or the filler is the source of greatest oxygen content.

### Oxygen exposure after packaging

After packaging, beer sold in bottles is still susceptible to increased oxygen exposure from the oxygen ingress through crown closures. Crowns provide a semi-permeable barrier between the beer in a bottle and the air outside the bottle.

The normal 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 flavour of the beer. This leakage or ingress tries 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. In many instances, this ingress is 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, many breweries now have a total oxygen package content of less than 40 ppb.

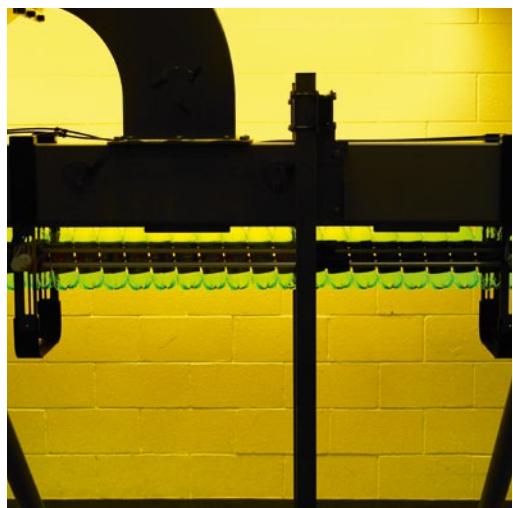
## Gas Phase Analysis

The Hach system allows you to measure both the dissolved oxygen concentrations of beer or carbon dioxide used for tank purging.

Analysers offer a dual phase capability, which allows the user to switch back and forth between liquid and gas phase measurements.

To measure volume % O<sub>2</sub>, ensure the gas is at atmospheric pressure by using the following method:

- Control the flow through the instrument before the analyser where the sample originates.
- Open the flow on the analyser to minimise back pressure through the instrument.
- Use a gas sample flow of 100 ml/min or less.



## At the Carbonator

When injecting CO<sub>2</sub> to beer, the CO<sub>2</sub> added must contain virtually no oxygen since large concentrations of CO<sub>2</sub> are being added under high pressure, which could rapidly lead to a build-up of dissolved oxygen.

**Adding carbon dioxide at high pressure:**

Amount of added CO <sub>2</sub> CO <sub>2</sub>	Concentration of O <sub>2</sub> impurity in CO <sub>2</sub>		
	0.001%	0.005%	0.02%
0.5 V/V	7 ppb	35 ppb	142 ppb
1.0 V/V	14 ppb	71 ppb	284 ppb
2.0 V/V	28 ppb	142 ppb	567 ppb
<b>Dissolved oxygen added to the beer</b>			

### Useful Units and Conversions

#### Oxygen

At 20°C dry air contains 20.94% O<sub>2</sub> = 209,400 ppm by volume.  
100% humid air contains 20.45% O<sub>2</sub> = 204,500 ppm by volume.  
In solution, 1 mg/kg O<sub>2</sub> is often called 1 ppm (by weight).

All the following solubility statements assume a pressure of 1 atmosphere.

When water is saturated with air it will contain:  
9.10 ppm O<sub>2</sub> at 20°C (68°F): 14.64 ppm O<sub>2</sub> 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<sub>2</sub> at 20°C (68°F): 69.90 ppm O<sub>2</sub> at 0°C (32°F).

#### Carbon Dioxide

Dry air contains approximately 0.03% CO<sub>2</sub>.  
1 volume CO<sub>2</sub> 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<sub>2</sub> at 1 atm pressure it will contain:  
1.72 g/kg CO<sub>2</sub> at 20°C (68°F): 3.37 g/kg CO<sub>2</sub> at 0°C (32°F).

#### Nitrogen

Dry air contains approximately 78% N<sub>2</sub>.  
In solution, 1 mg/kg N<sub>2</sub> 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<sub>2</sub> at 20°C (68°F): 23.2 ppm N<sub>2</sub> at 0°C (32°F).  
When water is saturated with nitrogen it will contain:  
19.7 ppm N<sub>2</sub> at 20°C (68°F): 29.8 ppm N<sub>2</sub> 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 analysing.