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CLOSURES: Random facts

Those in the wine industry speak of random oxidisation, but often without a full understanding of its nature and causes. *Sally Easton MW* guides us through the science to date

Random, or sporadic post-bottling, oxidation is a misunderstood term. We speak of it confidently, yet not always with full knowledge of its possible causes. Is random oxidation a convenient "cork-bashing" tool now that those cork manufacturers who adopt best practice are finding ways to minimise TCA? Or are there wider issues with implications for both bottle and closure and the process that joins them together? And what about the variability of natural cork?

It may seem obvious, but "random oxidation is bottle-to-bottle variation" says Dean Banister, sales director of Diam. "A lot of people misunderstand and confuse oxidation with random oxidation." If a bottle is being tasted in isolation, without comparison to the rest of the batch, it's impossible to say if it's random oxidation, ie, bottle variation, or a more widespread quality control issue.

There are three causes to consider: the closure, the bottle and bottling.

The closure
Everyone agrees that random oxidation can occur under any closure.

An inconsistent performance of the closure will increase the likelihood of random oxidation. Technical closures made on an industrial scale, such as screwcaps, synthetics and technical corks, such as Diam and Twintop, have much greater consistency than natural cork. Jim Peck, senior research scientist with G3 Enterprises, says: "Dense technical corks, such as the Diam and Neutrocork, have very uniform exteriors and press against the bottle bore quite tightly, reducing the possibility of oxygen ingress no matter what the position of storage."

Natural cork is a potential culprit here; it's a product of nature and its sealing capability is influenced by nine years of natural growing conditions.

The bottle
For driven closures, the neck bore of the bottle is key, for screwcaps, the top of the bottle. Geoff Taylor, managing director of UK technical lab Corkwise, says: "One bottle mould could give a slightly defective bore [though still within specification]. Even with a perfect closure, this could cause random oxidation." This is complicated by the fact that different neck bore sizes are used in different parts of the world.

Bottling
Joining closure and bottle together is a risky process for wine integrity. Bottling is, essentially, an exercise in oxygen avoidance. The opportunities for oxygen pick-up are manifold and if something goes wrong with the equipment even the best risk-management protocols can be breached.

Even with a perfect natural cork, says Taylor, "The cork may be OK, but if something as simple as the corker jaws are not set properly, the cork will be over compressed. The cork is blamed for random oxidation when the jaw is at fault. Synthetics are more of a problem with over-compression because they spring back less." And if a crease develops along the length of the closure, then the opportunity exists for rampant and random oxidation.

Olav Aagaard, chief technology officer for Nomacorc, says: "Bottling and closures are not yet integrated into winemaking. If something is wrong at bottling, people say the closure has to be guilty, but it is not always the case." If one or two heads in a 24-head filler are badly tuned, this can lead to random oxidation down the road. And this is with any closure.

"Differences in tooling and maintenance can create differences" says Jacques Granger, consultant to the manufacturers of Stelvin. "The shape of the filling pipe in the bottle, jogging or shocking bottles as they move between filling and capping can allow in oxygen." Banister adds: "If the labeller breaks, filled bottles wait, then they're sent to the capper." If the vacuum doesn't pull on one bottle, to evacuate the headspace, some bottles start out with a little more oxygen.

Sulphur dioxide management
"No matter how good the closure is" says Banister, "if there's not enough SO2 in use to begin with, then the wine will die."

The first Australian Wine Research Institute (AWRI) closure trials identified oxidative characters developing in white wine at about 10mg/l free SO2. This is a potential risk for producers operating a minimum SO2 regime. "There's no leeway" according to Taylor, so "if organoleptically there is no difference between 15mg and 10mg free SO2, why not bottle at 15? We need to take more notice of SO2 because it is related to oxidation."

Control of sulphur levels is not a random issue. But if there is no leeway, then sporadic incidences of oxidation may occur as a result of other causes.

Ascorbic acid
Ascorbic acid is often used in conjunction with SO2 to keep wines fresher and more youthful than by SO2 alone, and there have been suggestions that ascorbic acid might be implicated in random oxidation events. Dr Geoffrey Scollary, now consulting to the wine industry, has done much research on ascorbic acid and says: "This is the critical thing. Ascorbic acid as ascorbic acid is fine and safe;
as soon as it breaks down, when more than 95% is consumed, that's when colour reactions [signifying oxidation] occur."

But, he says, "Ascorbic acid does not decay rapidly unless a large amount of oxygen is present." Indeed, AWRI research showed wines were less oxidised if ascorbic acid was added at bottling than if it was not added. Industry consultant Richard Gibson, of Scorpex, backs this up, saying: "Ascorbic acid is not the culprit – it cannot degrade SO2 unless it has been exposed to oxygen. And it is just not possible to get enough oxygen into the bottle at bottling to account for the extent of change that has been seen."

Storage and transport
Incidences of driven closures moving within the neck of the bottle are well recorded, especially as wine travels across the equator. Mai Nygaard, Nomacorc's business development manager, said she had observed situations where "there's no temperature control in the container, there is more variation on the outside boxes than in the ones in the middle of the container", which suggests such an occurrence may lead to random incidences rather than the whole container being compromised.

In a shop environment, display bottles in the window or under bright lights will have a different evolution than those kept in their cartons in the storage area.

Back to natural cork
In addition to the risk factors that cross closure type, natural cork is created by a process of nature which give it unique attributes, both positive and negative. Does its natural variability affect random oxidation events?

Studies using Mocon Oxtran measurements 10 years ago found a 1,000-fold variation in oxygen transmission rates (OTR) across a sample of natural corks. Yet other studies, using free and total SO2, and colour change measurements, show only a two- to three-fold variability in the OTR of natural cork.

Latest developments
So where is the science at now? Gibson, who was involved with the original Mocon research during his time at Southcorp, says: "The 1,000-fold variation reflects the risk of random oxidation when upright storage with some batches of cork is used. Variation is less when laid down storage is used, but can still be considerable." He emphasises the variation across cork batches, saying some batches are fine, but "I've continually said some cork batches are worse than others. I've no idea why".

One of the analytical issues with Mocon is that it only uses dry cork, that is, cork not in contact with wine. At G3 Enterprises in California, Peck has developed a "wet" OTR cork method for the Mocon Oxtran, which is designed to more closely resemble horizontally stored wine bottles. He says: "In an upright situation it is likely the lack of a good seal between the cork and glass that allows in oxygen through what I call micro-channels between cork and glass. As the bore of the glass bottle is decreased, a better seal is created by increased compression between the glass and the cork, sealing off these channels, lowering the OTR. In a laid down or inverted position, wine will help seal these and also soften the cork to conform to the glass more completely, resulting in low OTR." The study is ongoing so he doesn't reveal the actual OTR value.

A study by Elizabeth Waters, a biochemistry research manager at the AWRI, also 10 years ago, looked at different natural corks, bottling a wine with the corks, also bottling the same wine with screwcap and bottling the wine with cork plus screwcap, one on top of the other on the same bottle. The idea being any variability under cork-stopper should not be replicated with the cork-plus-screwcap stopper.

The study found with some of the natural cork there was a big spread in the data for free SO2, total SO2 and colour, yet Waters says: "It is very hard to calculate an OTR variation rate from the spread of SO2 data, but with some assumptions, we can attribute a two- to three-fold variation in OTR to the largest spread of SO2 we saw in this study."

Of another study, using non-destructive spectrophotometry to monitor browning in white wine as a measure of oxidation, she says: "The spread of data was more like two fold. These values are a long way from 1,000 fold."

Scollary's observations of random browning also concur with the two- to three-fold variation.

Miguel Cabral, heading up research and development at Amorim, the world's biggest cork producer, says: "After the publications of Paulo Lopes et al [at the University of Bordeaux] in the Journal of Agricultural and Food Chemistry in 2005, 2006 and 2007, it became clear that the theories defending OTR values with a 1,000-fold variation in natural cork stoppers cannot survive a scientific analysis.

"This variability obtained by Hart et al [the Southcorp work] was probably due to a methodology error, or something else. But one thing is clear, this information was not published in a scientific [peer reviewed] paper, as Lopes' study has been. Lopes' data was clear: technical corks have less variability in permeability than natural corks but nothing even remotely comparable to a 1,000-fold variation."

He adds: "We are looking into reasons for the possible variability in natural cork stoppers, but it's too soon to present any definitive conclusions."

What's clear is the science does not agree and partisan proponents are able to pick the parameter that best promotes their cause (whichever cause it is). Speculation among scientists includes a rogue result in the 1,000-fold study, for example, by a crease in one of the cork samples, or some unknown aspect of Mocon Oxtran's measurement that is not yet understood. Gibson emphasises that he sees samples with oxidation rates that cannot be explained by oxygen ingress at bottling, and emphasises certain cork batches are worse than others for reasons we don't yet know.

It's clear the science is at odds on this issue, which is not necessarily unusual, and industry can draw no definitive conclusion until the science catches up to explain experience. It's only in recent years that miniscule oxygen ingress has been widely accepted as a normal part of bottle age.

Work to be done
The reality is there's not been much work done looking at the OTR variability of natural cork, apart from the studies mentioned. Another reality, perhaps related to the first, is the measurement of dissolved oxygen in wine is still a major technical and analytical challenge, whatever the methods used. But science is on the cusp of a new frontier of oxygen measurement and, therefore, management. This will undoubtedly help our understanding of non-random and random oxidation events. Peck even suggests emphasis might shift to the bottle, saying: "I suspect that bottle bore may have been the culprit as much or more than the cork. We now do much of our OTR testing in precision bore glass sleeves to eliminate problems with bottle bore variation."

We await results from Peck's "wet" Mocon measurements. And at the AWRI, Waters is beginning research using two new methods. Oxysense uses "oxydots, a fluorescent method, using scanning technology to measure oxygen through bottles", and the other new method has been developed at the AWRI, which uses a specific oxygen trap.

What is clear is that random oxidation can occur across closure types. Natural cork is more variable than industrially produced closures. More than this we cannot really say until science provides industry with consistent, and commercially useful, information. The spotlight, or maybe the oxydot, is on.

What are the causes of random oxidation in wine?
What role does closure type have in the problem?
We quizzed a handful of people in the closures industry

Simon Waller, vice president of global sales and marketing, Supreme Corq
Random oxidation can occur with any type of closure and in some cases it is found to be the result of closure variability. Our extensive research, both internal and external – Chambre d' Agriculture, (France), Geisenheim (Germany) and Vinquiry (USA) – on the SupremeCorq X2 demonstrates an extremely consistent level of oxygen permeability and wine preservation over time.

Published research in winemaking journals has proven the wide range of oxygen levels that can be present in a bottle of wine immediately post-bottling, before the permeability of the closure has come into play, suggesting that this is an area that should be focused on when trying to explain random oxidation. Wine faults, whether oxidation, taint or reduction, continue to be an issue, as seen in international wine competitions.

As evidenced by the 2007 International Wine and Spirits Competition (IWSC), fault percentages were highest for natural cork, which is inherently variable, followed by screwcap, where control of headspace oxygen levels is most challenging. Synthetic closures had the lowest fault percentages, a result of being more consistent and easier to manage with regard to headspace oxygen.

Carlos De Jesus, director of marketing & communications, Amorim
There is a combination of causes for random oxidation – it can be the closure, it can be the bottle, or both. There is much more than one simple cause and therefore no simple solution. Essentially, anything that is random is difficult to explain and you can't pin randomness to one usual suspect.

In fact, the very essence of the concept of randomness is that there is no predictability – so how do you find the culprit? However, perhaps the ingress of oxygen is not random, there is a pattern, but just a very low incidence of the problem. And it will take cooperation between a lot of different people to find the solution, but already, the problem of oxygenation is decreasing.

Anne Seznec, marketing manager, Guala Closures
Guala Closures is really committed in the research of a liner that is able to manage the oxygen transmission rate (OTR). Today the work has been carried out for the two kinds of closures we currently supply to market, the standard Divinum 30H60 ROPP and the new WAK.

The main difference between the two caps is their application and their liner. Our study is based on the main division between the two types of leakage: one occurs between liner and glass, and the other, the oxygen permeability that occurs through the liner.

Only managing, at the best, this separated phenomena, we are getting the best results. The current status of the testing is concentrated on the OTR of the single liner.

Tim Croxon, operations manager, Croxons
Looking from an overall packaging point of view, random oxidation is caused by the inability to provide a closure barrier that guarantees levels of oxidation. Many winemakers will agree that some oxidation is a required factor to produce good wine, however it is not an option when considering the standard closures currently available.

Screwcaps provide an absolute barrier, leading to reductive qualities in the wine, and even the best natural cork cannot give a narrow standard deviation for oxygen migration. It is only now that we are seeing a closure, in Korked, which provides a guaranteed level of oxygen migration. It looks like a screwcap (or cork depending on your preference), therefore providing that ease of convenience which many consumers have grown to appreciate, while still offering that migration of oxygen for the winemaker.

Utilising a research-led project, an organic polymer membrane was developed that would provide a barrier that was only permeable by oxygen at a predictable rate. It is available in both cork, but perhaps more interestingly, a screwcap variety.

Leal Ferreira, R&D director, Alvaro Coelho & Irmãos SA
Random oxidation can have several origins: firstly, bottle-neck diameter versus cork diameter. If this relation is not appropriate and when associated to the irregularities of the bottle necks, there is a higher probability of oxygen ingress between the cork and the bottle neck at different levels from bottle to bottle. These differences will lead to different densities of the cork on the inferior end and lead to different micro oxygenation kinetics. These different kinetics will in turn lead to different evolutions of the wine from bottle to bottle.

Secondly, if the type of cork stopper is not appropriate to the reductive potential of the wine. All wines have different reductive potentials. Some can withstand micro oxygenation for longer periods than others and with higher intensities. The type of wine and its reductive potential will determine the type of cork to use. The cork product range today, from micro granulates to straight natural corks covers all types of wines and all reductive potentials.

Thirdly, inappropriate surface treatments. The compounds used in surface treatments sometimes have hydrophilic properties that can lead to different wine absorption capabilities. These differences in absorption can generate a higher or lower contact of the wine with the cork in turn leading to micro oxygenation variations.

Fourthly, cork diameter. Cork diameter on its own can also determine the different micro oxygenation of wine. The higher the diameter of the cork, the more compressed it will be in the bottle. This compression will in its turn lead to an increased density, thus making gas exchange between the cork and wine more difficult.

Dr Olav Aagaard, director of global research, Nomacorc
The closure should seal the bottle properly and deliver the right amount of oxygen to the wine without bottle-to-bottle variation. That we are talking about random oxidation means that the exposure to oxygen is

not consistent bottle-to-bottle. So, where does the closure inconsistency contribution come from? Well, there are several factors at play:

External factors at bottling, like vacuum variation, inerting system not working properly or jaw damage to the closure; and internal factors, such as leakage at the interface between glass and closure, and closure OTR inconsistency.

It is interesting to note a couple of scientific reports on closure leakage done by AWRI, University of Bordeaux and G3 enterprises. In the studies they describe the oxygen ingress of a natural cork and a Nomacorc closure. They investigated the pathway of oxygen ingress and determined that with natural cork the majority of the ingress occurred via the interface of glass and closure. So, as it were, OTR is more leakage. In the case of Nomacorc they didn't find any leakage which proved that OTR is happening through the foam of the closure. This is exactly what you would expect from a co-extruded closure where the skin layer is specially designed to seal the glass and the foam is designed to transfer the oxygen.

Now the link with random oxidation can be easily made if you take into account that there are bottleneck bore differences within a lot of bottles.

In the case of the tested natural cork this will lead to different amounts of leakage, and hence there is a real chance that some bottles will be overexposed to oxygen, which would explain the random oxidation.

Mark Coleman, business director, Neocork
If we're speaking specifically of "random" oxidation I think one has to consider a variety of potential causes, such as O2 management at bottling, bottle type, closure type, shipping and storage conditions, as all of these are role players.

Closure type is obviously important because a compromised seal between the bottle and closure can be a leading cause of random oxidation within a specific batch of wines. Further, since the industry accepts that bottleneck diameters will always be inconsistent, it requires the closure to compensate for the bottleneck's inconsistencies. Some closures are better at this than others. I think this is why we hear so much about random oxidation in screwcapped wines. It's not really the screwcaps' fault, it's the bottles! You are trying to ensure a perfect long-term seal between a screwcap, which has a very tight application tolerance to a bottle, which has a very wide dimensional tolerance. It's asking a lot, especially once you subject those capped bottles to uncontrolled shipping and storage environments where the caps are either knocked together in transit or exposed to excess weights when pallets are stacked, both of which can compromise the seal.

Most quality wood bark corks have the elastic properties necessary to compensate for inconsistent bottlenecks and seal properly. However, since it is a natural product you tend to have inconsistent densities and weights from cork to cork and while you may not have extreme cases of random oxidation you will find that 12 wines in a case will evolve inconsistently to each other.

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