

**TRANSCRIPT**  
**AMORIM TECHNICAL SEMINAR, ADELAIDE, 29 OCTOBER 2003**  
**THE ROSA PROCESS**

**PART B: PANEL DISCUSSION**

**Tony Telfer**

Ladies and Gentlemen, welcome back. I'd now like to introduce to you the members of our panel. First of all, the chairman, Len Evans.

Len, as you know, has had 45 good years in the wine industry that includes holding positions such as chairman of Petaluma, Rothbury and Tower Estate. He has also had the distinction of being one of the only two Australians, I think this is correct Len, to hold the title of Decanter magazine Man of the Year.

Joining Len on the panel will be Antonio de Barros and Miguel Cabral and three of the researchers who have validated the ROSA process.

Martin Hall is Director of Food Science at the Campden and Chorleywood Food Research Association. Martin is a Fellow of the Institute of Food Science and Technology and a Chartered Chemist and also a member of the Royal Society of Chemists. Before becoming a director he was the Head of the Chemistry and Biochemistry Department. Martin has over 25 years of experience and is the author of many scientific-papers and reports. He is very experienced in flavour and taint research, having coordinated several national and European research projects, including the Quercus project. Nick Byrd is the senior analytical chemist at Campden and manages the chromatography section within the Chemistry and Biochemistry Department. He also has many years experience in chemical analytical techniques, and particularly the identification of and determination of organic contaminants in food and drinks.

Mark Sefton will also be known to many of you. Mark has a PhD from Western Australia in organic chemistry and he is a principal research chemist with the Australian Wine Research Institute where he has been for 16 years. Like Campden, the Australian Wine Research Institute has played a leading role in the research to understand mustiness and cork related taint in wine, focusing on cork in the bottle. Mark has published several scientific papers on the subject. Ladies and gentlemen, Len Evans.

**Len Evans**

Well thank you very much indeed, Tony. Ladies and gentlemen, it's a pleasure for me to be here. I'd like just before we start, to give you a brief history of my involvement. I was asked some years ago to be part of this—if you like, the institution of cork and I said there's no way I was going to be part of it as an apologist. Because probably drinking more than any of you, and certainly better bottles than most of you, which is... someone has to do it, I was absolutely peed off with the cork situation, the TCA problem, as much as anybody. I had the pleasure then to go to Portugal and had talks with some of the senior people

including one glorious conversation when I turned around and said, I'm very much against the sort of smokescreen thing you're talking about, screw cap and what it does and synthetic cork and what it does. What we should do is concentrate on the reality, the reality being that TCA is the killer and the cork industry, if this goes on, we are not going to have a cork industry. Who was the chap that authorised these other campaigns? He said "I did" and from that moment on we got on terribly well and it became a matter of, in fact, looking at the problem, the great problem of TCA and how this one company in particular has handled it. And I think it's remarkable what they've done. In fact it's lovely to be here surrounded by so much quality control. A chap has to have it.

We're going to ask very briefly from commentaries from Martin and Mark before we ask questions and then I'll take over again.

### **Martin Hall**

Thank you very much indeed, Len. It's a pleasure to be here. Our involvement, as has been said, was in relation to validation of the effect of the ROSA treatment on the levels of TCA in wine. If I may I would like to ask for your forbearance for a moment. I've been involved in the area of mustiness in wines and other products for some time and I was involved in the European research project called Quercus which some of you may have heard about. It was very obvious from that trial the significance and the seriousness of the level of TCA issues, and arising out of that project there was quite a lot of scientific data that was produced. I think one of the things that has impressed me most, since that project, is the way that the scientific data has been used and developed further by key players in the industry. In particular its application to bring about changes in the way that corks are produced. I'm not just referring to ROSA but the whole strategy of cork improvement. But back to ROSA, which is really what I'm meant to be talking about.

TCA as many of you know is a particularly difficult compound to deal with. It's difficult to analyse and difficult to deal with because it binds very strongly to many matrices and not least cork material. So when we're talking about methods of removing TCA from cork we've got quite a challenge there. What I think is quite appealing about this approach is that it is based on very simple scientific principles. I don't doubt that there is very elegant engineering behind it, but the scientific principle is very straightforward: steam distillation. And what we're seeing here from the data that we've produced, and I'm delighted as an analyst to see how consistent it is across the other laboratories that validated it, is a reduction in the region of 70 to 80 percent. I think that is very significant. What we've obviously got here, as you would expect from the distillation process, is a partitioning effect. We're getting rid of 80 per cent of what was there in the first place; that's very significant and I think fair to extrapolate further. TCA is obviously the major culprit but Miguel presented results for some other compounds. We can assume ROSA will provide the same sort of reductions in other compounds that may or may not give rise to other problems in the cork.

**Mark Sefton**

I'll just make a few comments. I must say first of all as a part-time analytical chemist it's a relief as much as a pleasure to get similar results to what everyone else is getting. Perhaps something Miguel didn't point out was that the samples that we analysed were also analysed by himself and his colleagues. The analyses were not done in the same way; theirs were analysed instantly, ours were flown half way across the world in a depressurized cabin. The soaking solutions were different, the analytical methods were different and yet the percentage reduction that we saw in six out of the eight units was almost identical. In one case the results that we obtained were rosier, if you like, than those obtained by Miguel and his colleagues. We had better reduction and the other one was slightly worse and he actually showed that here. So when you can get similar results obtained in really, in some respects, quite different ways, that's very encouraging and suggests that the results are indeed highly robust.

Another observation which I think is encouraging is that the samples were analysed by looking not at the total TCA in the granules but what's extractable through the wine, what's extracted over a fairly short period of time. So the bottling trials Miguel has shown are also very important. We know that TCA moves only extremely slowly, if at all, through the cork but it's nevertheless possible that what is removed by the ROSA process is only what is very close to the surface. But these bottling trials indicate that what you see in the short term extraction is more than likely what you will see in corks made from these granules in the bottle over a long period of time. There are probably many other points I could make but I'll leave it there for now.

**Len Evans**

Well, it's time for questions. We have a travelling mike. Tony is going to rush around with it. Would you be good enough to state your name clearly and your company and then any of the particular members you'd like to address or if it's an open question, I'll appoint someone.

**Richard Gibson**

*I'm Richard Gibson, Scorpex Wine Services. Hi Miguel, how are you? A question for you. Just go back one step before the ROSA process if you could, the slide that you showed on the purchased cork and the cork processed at Ponte de Sor and Coruche, the percentage differences there broken up by TCA level, would you just explain the basis of that TCA level that you had in those graphs. It was nanograms per litre. Was that releasable TCA and was that per closure, per litre of wine or on what basis were those measurements made.*

**Miguel Cabral**

Those results were obtained always after analyzing 50 corks in a composite soak and analyzing with GCMS coupled with SPME. And so we are talking about corks after punching, not after washing, after punching, and the punched corks we bought on the market were compared to corks in the same condition, that is punched corks, that we got from Ponte de Sor and Coruche. And so the differences between less than 2, 2 to 5 and over 5: we set those ranges because less than 2 is our quantification limit; 2 to 5 was because we know that after

drying, after washing and drying, the TCA level came down a bit more; and with the upper limit, more than 5, it's a little bit difficult to use those corks before ROSA.

[Indistinct comment from floor].

**Greg Oke**

*Greg Oke from Southcorp Wines. Question to Miguel. A lot of the data you have pre-ROSA, TCA levels around the 15, 20 nanograms level. You get a 70 to 80 per cent reduction. What results do you get if you start at 5 nanograms or 4 nanograms?*

**Miguel Cabral**

Well, we start always with high contaminated batches because we want to see the difference that ROSA is able to make. So that is the reason why we made all these analyses in highly contaminated batches. However in the slide I showed of our internal validations, there are different levels of contamination. Some of them are over 15 nanograms, others 10 nanograms, others a little bit less. And not in a very consistent way. There was one case I think that was not exactly like that but with all the others, when we have higher levels of contamination we have less percentage reductions. So the reductions were 76, 73, 74 per cent when we have 15 nanograms or more and when we have 10 and 15, in that slide, we got higher reductions, around 86 sometimes or 83 per cent. So that is what we have in the results and that can indicate to us that probably we would get even better reductions when we have much lower TCA levels than when we have lots of highly contaminated granules.

[indistinct question]

At this stage we are not doing this kind of analysis because as you all understand we need to be quick with this thing. And so what we want is to keep our contaminated batches aside, so we have data to confirm what we think. But I agree it would be interesting to see the reductions with lower levels of TCA than with these ones; fortunately they are going to be, if not similar, probably better.

**Mark Sefton**

Perhaps I could comment on that. What you say is logical when you think about the low levels going into the process to begin with. [Indistinct ] A lot of the methods that are being used are not very accurate when you get down to the likely ROSA level. ROSA produces something from 3 nanograms to half a nanogram. Chances are you won't be able to tell the difference between nothing and 1 nanogram so you can have anything from 100 per cent reductions to something much less than that. With modern instruments and modern techniques it will become easier to see those lower levels and it should be possible to look at what happens with relatively uncontaminated granules, which of course is something that ultimately we will want to do. But that's the reason for it. It's purely for practical reasons so we can get after-process numbers that are accurate enough to get some idea of what's going on in the process. So they're really in a sense an experimental sample.

**Martin Hall**

I was just wanting to make one small point on that. I mentioned at the start that we were talking about what is in many respects a very simple scientific principle, steam distillation, and the distributional equilibrium if you like between the gaseous phase of steam and the solid phase. And that equilibrium should be relatively consistent. So we would expect the same sort of efficiency of extraction at high levels or low levels and so I would expect, as Miguel said, at least the same levels of extraction at those low levels.

**Geoff Linton**

*Geoff Linton, Yalumba. I guess I've got a deep abiding concern about composite soaks. I was wondering...I didn't quite understand, did you do individual cork soaks or were they all composite samples?*

**Mark Sefton**

They're granules, so they are automatically composites. The results for individual replicates were very, very similar. In the first instance what we are soaking is many hundreds of granules. And each batch of granules behaves after the ROSA process in an identical way to the other batch. So they were composite soaks. The question arises of course as to what extent this process of soaking something for 24 hours represents what happens when you put something made of these granules into a bottle and that's not necessarily going to be same thing. And that's why the bottling trials are ultimately so important.

*Peter Godden, AWRI. Your answer to Greg's question, does that imply if you were to treat some granules a second time you'd remove another 75 per cent of the TCA that's still there.*

**Martin Hall**

This was actually a question that I'd already asked Miguel. I think in terms of scientific principles I see no reason why with a second extraction you wouldn't get further reduction but I think there are other reasons why a second extraction is not feasible but I will leave that to Miguel to explain.

**Miguel**

Well we did that a couple of times and actually we didn't get similar reductions. We got further reduction but not as much as the first time. But the big problem with doing that is that after the steam distillation the granules are more dense. And so if we repeat with another steam distillation then the density is so high that we cannot use them to produce cork stoppers. So it's more mechanical than chemical. Of course if we do another steam distillation for a group of cork granules that has something like 5 nanograms per litre for instance, probably we would get something like 3 nanograms per litre afterwards, but then these corks would be not so good.

**Mark Sefton**

Another thing too is that you put fresh granules into the process, and the TCA in the granules is not always bound necessarily in the same way. Some may be loosely bound, and that's easily removed by the process. Some of it may be very

tightly bound in the interior of the granules and that's never removed at all by either wine or the ROSA process. And you can have everything in between, in theory, at least. So it may well be that the first ROSA process removes all the easily exchangeable TCA and some of the not-so-easily exchangeable TCA. And when you put it through the second time, perhaps what you can't remove is some of that TCA that may still be extracted by the wine but is not so easily blown off with the steam, because it's deep within the cork granules or for whatever reason. You wouldn't necessarily expect the same result. That seems to be what you get.

*[indistinct remark from floor ]*

Well that's exactly right. In this context extractable TCA means TCA extracted over a 24-hour period by a wine made in certain way, carried out under certain conditions. And that's why I say, that's one form of extractable TCA. You can think of extractable TCA as what you can extract in 3 months, 12 months or 5 years. And so the critical thing here, which still has to be determined, is what is the relationship between extractable TCA at the time of these experiments and what you see in the bottle. And that's why I say again and again why the bottling trial is so important, so we can see whether there is a relationship or not.

### **Paul Bowyer**

*Paul Bowyer, University of Adelaide: Miguel I'm always happy to see the application of old technologies in a new way and that's pretty much what's happened here. Steam distillation, as has been commented, has been around for a long time and it's effective in this manner. I just have more of a question on the economic side. The effectiveness of this procedure obviously relates to the surface area of whatever you are treating so the granules respond well and the natural corks probably not so well. What proportion of your market share is consumed by granulated products as compared with the whole natural stoppers and is this going to increase in future?*

### **Miguel Cabral**

I am not going to answer that question because I don't know. Mr. de Barros will answer you.

### **Antonio de Barros**

Well still undoubtedly the natural cork stopper is the leading closure for wine bottles. The proportion is approximately two-thirds natural to one-third technical. This is evolving rapidly. In our case, in the case of Amorim & Irmãos, I'd say that it's nearly two-thirds/one-third, natural and technical.

### **Peter Leske**

*Peter Leske, from Nepenthe. Probably for Miguel. The question is about the other impacts of the ROSA process on the cork. You had data there on MIB and guaiacol, but I'm interested to know if you've done anything on general phenolic compounds, tannins and the sorts of things that people talk about being responsible for woody cork characters, which some people view positively. And secondly on the physical property of the cork, we know the INOS process was considered a couple of years ago, I think when you spoke, [and it was thought] to be problematic with natural corks because of distortion and need*

*to abrade it down, all those sorts of things. They worked for discs but not the stoppers. Have you done anything yet on the physical effect of the ROSA process on natural cork?*

**Miguel Cabral**

Yes we have done lots of things on physical properties. I'll start with the second question and then you will have to repeat the first because I didn't understand it very well. Yes we have done a lot of tests on mechanical parameters for natural cork stoppers and there's no problem in terms of mechanical performance. However, when we pass natural cork stoppers through ROSA we have deformation. And after this deformation the corks swell and so we need to rectify them and take them back to the previous dimension. Theoretically we could have problems in terms of density which could create problems in terms of compression, but this is not the case. So we have the same values with a batch of cork stoppers analysed before ROSA and after ROSA; no problem in mechanical terms. This is for natural.

For cork discs that is one of the reasons why we are a bit delayed, with cork discs compared to cork stoppers. Actually there are another two reasons. First of all, we have INOS and INOS is already well proven in the market. Secondly, after ROSA we have some corks that swell, and we have to polish them in diameter. At this stage we don't have a machine to do that and so we have to separate the ones that are bigger than the others. I would say that it's probably not very difficult... Of course, it's not for me to do it, but it's not very difficult to construct a machine to rectify them in diameter. We have to deal with that. You will have to repeat the first question.

**Peter Leske**

*The first part of the question was about tannins and phenolics that may be extracted from cork—where more and more people are talking about that generally, probably not really the science, but around the industry it's about the corkwood character. Whether or not that's positive or negative is another story, but presumably the steam extracts dust, phenolics, all sorts of things. Do you have any data on that?*

I'm a bit concerned that it was a very, very rough test, so I cannot answer you in a deep way. But what we did was this. We put cork stoppers from the same lot before and after ROSA in a water soak and in a 10 per cent ethanol soak and it showed the difference in colour coming out of the two soaks. Much, much lighter in the second case than the first one. But probably that is not enough to use their corks for instance in spirits, where the colour as you know is a big problem.

**Mark Sefton**

I think if I can add further. Certainly when we are talking about tannins and phenolics and all of that, they are not volatile and therefore not likely to be blown off by steam [indistinct]. Woody characters of course are more likely to be from other volatile compounds which if they are volatile, depending on how volatile they are, may well be removable by steam, although without knowing what those compounds are [indistinct] and the levels at which it happens [indistinct]. I know for what it's worth that the granules before they went into the process had a strong TCA odour and the ones that came out of the dryer which

were pretty hot, and you have a better chance of smelling things when they are hot than when they are cold, had very little odour. [Indistinct]

**Greg Oke**

*Greg Oke again. Just to add to that. I've smelt the granules pre and post ROSA treatment and the ones post ROSA certainly seem a lot cleaner. The treatment does appear to improve the quality of the technical cork based on your results. I'm wondering Miguel what is Amorim doing about the other aspect of the technical cork which is the glue that binds the granules and also the glue that binds the discs to the agglomerate body? What effort is going into that, to remove those characters that can be picked up from the glue?*

**Miguel Cabral**

As you know we are not experts in glues so all we can do is apply to international chemical companies to develop glues, which was exactly what we did a year ago or a little bit more. And we challenged them to develop different glues in order to avoid some of those characters [with the current glue] that are not yet clearly defined, but there are some problems with these glues. However we challenged several different companies, from Australia, from France too, and we tried to do that kind of project, in collaboration of course with those companies. And we have started very recently, two months ago, a bottling trial with a new glue that is polyurethane-based, produced by a French multinational, which performed mechanically very well. So now we are testing migration in bottle. We are doing that bottling trial in France. It started two months ago at the beginning of September. So that concerns agglomeration.

But there is another glue that we use to glue the cork discs. Actually we were not thinking to do anything about that but we were approached by two different chemical companies that asked us if we were interested or not in testing the glues they have. These glues are also polyurethane-based but different types of glues than before. We have already done mechanical tests on the first one and it is, I would say, completely comparable to the old glue. We just received the sample for the second one so we haven't tested it yet. The main idea for the glue for discs is to try to change the casein glue that we still have to use in champagne corks for France, because they just won't act. They were waiting for a new glue that can be approved by the CIVC, which is Comité Interprofessionnel du Vin de Champagne. One of the glues that we are now checking has already been approved for that. The other one has not been approved yet but has been submitted for approval, so we will see the result.

[Indistinct question]

It is difficult to say. About this new glue for agglomeration: we started the bottling experiment for this new glue in September, so as a minimum we are going to do six months, and then we have to have some time for the analysis. About the glue for discs, that will take a little bit longer because we have not even started the bottling experiment.

**Bill Seppelt**

*Bill Seppelt, Southcorp. Miguel, with the development and improvement now of analytical techniques and the finesse of those techniques, has it been established firstly if there is TCA in all cork and secondly is the intent to treat just those batches of cork or that cork which is indicative of high TCA or do you intend to treat all cork?*

**Miguel Cabral**

I'm sorry again I didn't understand the first question. About the second question, first of all concerning cork granules: we are going to treat all cork granules for agglomerates, for technical corks, for Twin Top corks. All cork granules are going to be treated in the same way by ROSA. Concerning cork stoppers and cork discs: the idea, the plan, is to treat all corks but we are at a stage where we have just started doing tests on ROSA for natural cork stoppers in an industrial routine. Not yet for cork discs. So after we have those results, not just the TCA results but also the mechanical results, we have to make the decision of whether to treat all corks or not. But the idea is of course to do all corks.

**Bill Seppelt**

*The first part of the question really is with the improvement of the technology and analytical processes prove the research you've done so far. Has it been confirmed or discovered that TCA is in fact in every cork? Or is it isolated to certain batches or forests or regions?*

**Miguel Cabral**

It is not in every cork, at all. There are lots and lots of corks that have no TCA. We have less than 2 nanograms per litre with our quantification limit. And when we have our machines prepared to increase the time of incubation, we have no detectable TCA in the corks. But we have some lots which are really problematic.

**Mark Sefton**

Can I elaborate a bit on that and say that if you grind up corks fine enough and extract them thoroughly enough, it's a rare cork that doesn't have TCA in it. But if you take those corks and before you grind them up you soak them in the wine, it's only a very small proportion that release their TCA into the wine. And that... we interpret then in certain circumstances, and there is other evidence for this, it's when TCA is on or near the surface of the cork that you get a problem. And so Miguel is right. If you got your last batch of corks, if you soak them, you won't get TCA at all but that doesn't mean there isn't TCA deep in the cork. And I suppose when you produce granules, where in effect you are grinding up the cork, the chances are you're going to expose more of that TCA. But at the same time, if you can remove it by appropriate washing or by a steam distillation process, as long as you clean the surface properly, you can get rid of most of the problem.

**Chris Taylor**

*Chris Taylor from Henschke Wines. Just following on from that comment from Mark Sefton, with respect to treating cork stoppers with the ROSA process, would the reshaping that occurs post ROSA then run the risk of exposing TCA. Has that been looked at?*

**Miguel Cabral**

We have done an analysis on reshaped corks because no doubt we have to reshape them because they've been deformed. And we have the results on the reshaped corks and the reduction of TCA is clear.

*Indistinct comment*

We did once and actually I presented that slide here in Australia. We did one interesting experiment. It was from a highly contaminated batch of cork stoppers. We took 50 corks and we soaked them and then we reduced, shaved them to a smaller dimension, I can't remember what it was, and we did that into the interior, we shaved the corks four or five times. Before they were shaved they were treated with ROSA and some of them, one or two or three of them... from 50, I do not know the exact number, there was the same result. So our conclusion was clear. ROSA was not able to take out TCA from the interior of the cork stoppers. How deep it goes exactly I don't know, but from the interior, completely, I don't think so.

**Richard Gibson**

*Richard Gibson from Scorpex again. Miguel, where will the ROSA process be applied? Will it be applied to punched cork or will it be applied to rectified cork or to washed cork?*

**Miguel Cabral**

The idea at this stage is to apply it to punched corks. After punching, before rectification, in order to reduce the amount of rectification of the cork that we need to do. So the idea at this stage is to do it after punching.

**Martin Lightfoot**

*Martin Lightfoot, Hassle & Lightfoot, a small wine company. My question is, has Amorim a date in mind when corks could be bought confident there is no TCA identifiable in the end product?*

**Miguel Cabral**

That question has two answers. If you are referring just to the ROSA process, we hope that in 2004 we'll be able to do that, to supply ROSA-treated corks, natural corks. But, it's important to say, because it's not negligible at all, that after the application of these quality controls by GC-MS and GC-ECD, we are making an enormous effort in terms of quality control. Clearly we have detected contaminated lots. I agree with everybody that with this system we are not able to detect sporadic corks contaminated in a lot, but we are able to detect contaminated lots. And we have a case study that I was telling you about in the presentation, and I can go a little bit deeper into that now. We have one company that sells to America. It was in 2000 and ETS at that time already had the GC-MS installed. And in that company we had problems because the corks were analysed sensorially and so the great amount of them were passed by that quality control and they were sent to America. When they arrived in America, the company made soaks and they sent those soaks to ETS and they were able to do quality control for TCA by this procedure, and they realised that there were a

lot of contaminated corks at that time. That led us to suspect that we had some problems in that factory so we asked a French company to audit that factory to see what was going on in the process. And we realised that it was corks that we bought into that factory to send to America; when the corks arrived in the factory they were already contaminated. So we applied the GCMS technique for those corks. And that factory continues to do exactly the same procedure, send corks to America, but there are no more TCA problems in America from that company. No more TCA problems in clients, but there were not before because those corks were stopped by ETS, but the difference is in the values obtained by ETS before and after we applied this kind of control. So it's not negligible at all, this kind of GC-MS control. I agree with you. I think all of us are thinking the same way that this kind of control, it's not perfect, but no doubt it's a huge improvement compared with what's happened before. No doubt.

### **Len Evans**

I think it's been a wonderfully frank and open discussion of this great scourge that we wine lovers and wine suppliers have had for the last few years. Recently I was at a famous household in England with a famous wine writer who claimed to me that the problem was still much higher and that a quotation of two to four per cent of all wines she drank were still contaminated and I said, "That's all very well, but you hardly ever drink a wine younger than 1990 and most of the wine you drink is pre 80." So she said, "I hadn't thought about that," so perhaps she'll change her tone slightly in the future.

I think that enormous advances have been made. We all quite appreciate that. I think Miguel has been terribly honest when he says it's not yet totally eliminated and it will take further time. All I can do is commend Amorim for their leadership in really working so hard. And when I went to see them originally, I said, "You're spending all this money, doing all this damned work and yet you're not telling the world and discussing it freely with all winemakers what the problems are." I think all that's been eliminated now and I commend the company for really having these open discussions and forums to tell you precisely what's going on and to be precisely honest about what is happening. Thank you very much indeed.

[Applause]

### **Tony Telfer**

Well, this concludes the formal part of today's proceedings. We have two requests for you. Firstly if you could be kind enough to fill out the feedback form on the back of the sheets. Secondly if you'd like to join us for some wines and drinks, some red wines from the Duoro Valley and I believe some lovely beer as well, right? Thank you.