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

PART A: PRESENTATIONS

Tony Telfer

Good afternoon ladies and gentlemen and welcome to Amorim's technical seminar on the ROSA process. I'm Tony Telfer, recently appointed as Managing Director of Amorim Cork Australia and it's my pleasant duty to introduce the program and our speakers today. All of them have been involved with the wine industry in one way or another for a long time.

The program is in two parts. Firstly Mr. Antonio de Barros, Executive Vice President of Amorim & Irmãos, will speak briefly about Amorim's commitment to improving the quality of cork closures. Mr. de Barros has made many visits to Australia and will be well known to many of you.

Next, Professor Miguel Cabral, Amorim's head of Research and Development, will present Amorim's most recent advances in the fight against TCA and in particular the new ROSA cork treatment process. Miguel has a PhD in Microbiology and is also a member of the Pharmacy Faculty at the University of Porto. He was recently head of the quality control laboratory at the Vinhos Verdes Official Control Institute in Portugal.

After Miguel's presentation there will be short break. That will be followed by a panel discussion and open forum chaired by Len Evans and involving three of the researchers who have validated the ROSA process. I'll introduce them later.

Noel Heyes, newly appointed chairman of Amorim Cork Australia, has asked me to pass on his apologies. He's been suddenly indisposed and is very disappointed that he was unable to attend today and has handed the introduction job to me. Very kind of him. So without further ado I will hand over to Mr. Antonio de Barros.

Antonio de Barros

Good afternoon ladies and gentlemen and thank you for being with us today.

Two years ago as some of you will recall, Miguel Cabral and I stood before you in Adelaide during the Australian Wine Industry Technical Conference, our purpose being to update wine makers in Australia on Amorim's program to improve the technical performance of cork closures. It was an important occasion for us because we were able to tell you about what we believed was a break through in the fight against TCA. I am referring here to the ROSA process. At that time the process was in the early stages of its development. Miguel seemed to have produced some very exciting results in the laboratory and with industrial prototypes of the process. But we knew that it would not

be a simple matter to scale up to industrial production and of course we were right.

We also promised that we would subject the process to validation by independent researchers to give wine makers confidence that ROSA was indeed a factor in removing TCA from cork. We have done that, and an important part of our discussion today will be about the validation of ROSA treatment. Amorim asked the AWRI, the CamPden and Chorleywood Food Research Association in the UK, the Geisenheim Institute in Germany and Excell Laboratories in France to conduct these tests and I am delighted that Mark Sefton, Martin Hall and Nick Byrd from two of those laboratories are here today.

The development of ROSA has been a high point of Amorim's research and our ongoing efforts to improve the quality of our cork stoppers and the task of bringing ROSA to the market is not finished yet. But ROSA is not the only weapon in Amorim's armoury. It adds an important curative element to a range of preventive measures we have implemented in recent years and let me mention a few. We overhauled our production system establishing new processing factories in the south of Portugal which incorporate new storage and selection methods as well as a new boiling system.

HACCP protocols are being used to identify and manage critical risks across our production and distribution chain and we are working with customers such as the UK supermarkets to ensure that our products comply with their strict quality regimes. We have taken control of our distribution chain, establishing a direct presence in countries such as Australia so that we can better understand and meet your needs. And of course we maintain a large R&D effort led by Professor Cabral and his team and we are delighted with the results they have achieved.

Of all the changes we have introduced, without doubt the most important development is ROSA. While we have been doing much to avoid TCA, for the first time we have a science based method of extracting TCA from contaminated corks and cork products. It is not the whole answer but it is a very important part of the answer. Of course we will not stop here and we have a number of other research projects under way. We will keep you informed about our progress with them and with ROSA.

I am sure you will have many questions about the ROSA process. We know that the Australian market is one of the toughest and most demanding and this has been good for the cork industry in that it has challenged us to constantly improve our performance. I trust that today's discussion will reassure you about the progress we have made.

Thank you. I will now ask Miguel to present the latest information about ROSA and Amorim's fight against TCA.

Miguel Cabral

Ladies and gentlemen, we are here today to talk about what Amorim believes is the most significant advance in cork processing for at least the last 10 years.

Of course I am talking about the ROSA process. But before I get to the description of ROSA it is important for me to briefly explain where ROSA fits into our R&D program and into Amorim's anti-TCA strategy.

Our strategy to defeat TCA has two areas. A prevention area—group of actions to prevent the appearance of TCA—and also a curative area where we want to take out the TCA that is still there. In preventive strategy, we talk about raw materials, cleaning, hygiene and quality control and in each of these areas I will talk about some changes we have made in each area. Probably a lot of you have been in Portugal and already know of these kind of changes.

First, raw materials. Before boiling we select out things like yellow stain and the lower edge of the cork bark that is in contact with soil, which have a higher possibility of being contaminated with TCA. In terms of cleaning, we have the new boiling system that has been installed in our two new factories in the south, Ponte de Sôr and Coruche. One of them has been there since the end of 2000 I think and the other one since the middle of 2001. Those two factories have a new boiling system that is completely different from the previous one and we have already communicated these differences to the Australian market.

INOS II. It is probably one of the reasons why Twin Top corks have such success in Australia. It's a way of washing cork discs with hot water and different pressures. And INOS II has probably been at the base of the success of Twin Top.

Concerning hygiene, we talk about ozone. Actually we tried to see if ozone was able or not to oxidize the TCA molecule. Unfortunately it was not but we use ozone, for instance in our factory in Melbourne, because it's a good way to hygienise our cork stoppers because ozone by its oxidation action is able to kill microorganisms.

But the main part of the prevention strategy that I have to tell you about today are the changes concerning quality control. We used to do quality control by sensory analysis, but since the study made by ETS in California it has been a demand of the cork industry to apply GC-MS coupled with SPME technique to analyse TCA. We have an important tool to measure the amounts of TCA present, not just to see if it is there or not. And so we are doing that in a very strict way and in a very intensive way.

Just for you to understand that we are not talking just about simple results. We are talking about a methodology that is strictly controlled. First of all we validate the method that we are using, the GC-MS coupled with SPME to measure releasable TCA. We use control standards on a daily basis to produce charts, quality control charts, to accept or reject the run of the day. Also we are participating in inter-laboratory tests which are actually a very important way to compare results, for us all to speak about the same numbers.

Here is an example of two of the parameters that have been determined for the validation of the method—the detection limits and the quantification limits of GC-ECD and GC-MS. Control charts have been part of our

validation of the method and on a daily basis we make two control standards in order to control our runs. And so of course if there is any result outside the limit, we have to repeat the run. For instance in this case, and this is a recent control chart from July to the end of September, the tendency starts to appear here and we want to know what's going on. It was a pump on the GC-ECD and we had to replace the pump. But look, with this tool in hand we are able to check the problems and we are able to accept or not the results obtained.

Concerning inter-laboratory tests, from September 2001 we were doing sporadic tests, that's in an unorganized way. We started in July 2003—with Excell, Sabaté and ETS—to do an organized ring test. Also with Cevaque, with whom we started in September 2003. There are lots of laboratories involved in the ring test; I don't know who they are. And so we are very keen to continue with this kind of inter-laboratory test in order to talk about true numbers of TCA.

But all this is applied to a significant number of samples per day. We are talking about something like 5,000 samples that we are analyzing per month and this is to try to control our production of cork stoppers, cork discs and cork granules. Actually we have at this stage 5 GC units (2 GC-MS and 3 GC-ECD) and with this control applied to the great majority of our production we actually are able to separate the TCA-positive lots from the others and not include them in our production chain. There was for instance the case when we had a problem with some corks that we bought through a small factory and we had problems in the American market. Those problems were completely solved applying this kind of methodology to all the corks from that factory. This was the first time we had used this methodology.

A good example of this and the importance of this kind of control is this comparison between some corks that we bought in the market versus some corks produced by our production in our factories at Ponte de Sôr and Coruche where we have our new boiling systems. And actually in spite of having some different numbers in the samples, we are talking about composite samples of 50 corks, we've got a big difference in contamination levels between corks produced from our new factories in Ponte de Sôr and Coruche and the corks bought in the market. This is an indirect way of trying to validate what we already know, that the new way of boiling cork is actually a good way to prevent TCA contamination. Look at the amount of contaminated corks from Ponte de Sôr and Coruche compared to the amount of contaminated corks bought from the market.

So this chemical analysis using GC-MS is the first part of an important strategy, a recent strategy, to defeat TCA but actually we are here essentially to talk about ROSA.

ROSA belongs to the curative strategy. And we started with the basic chemistry. We went to the Merck Index. And in the Merck Index there are not too many things about TCA, but there is one thing which caught our attention. It's volatile with steam. And so from this we started to see what we could achieve using steam distillation. And this progressed to a three year development project where we used steam distillation to clean TCA from cork

granules, cork discs and cork stoppers. And this chart shows where we are concerning the three parts.

Two years ago, here in Adelaide I presented already the results from the laboratory tests. We applied steam to cork granules, discs and stoppers in the laboratory to see if we can go ahead with this process or not and actually the results were very good. So we constructed prototypes to scale up the system. We constructed one prototype first and then we needed a second one for cork discs and stoppers. I will tell why you further on. Then we built industrial pilots and made internal validations. Right now we are already in an industrial routine for cork granules and we hope to be not too far from now in industrial routine for cork discs and cork stoppers. But we will see the results of that.

First of all let's look at a schematic of the ROSA system. These two figures correspond to prototype 1. It's a really simple system. We introduce our granules or cork discs or cork stoppers here. We pass them along this auger and we introduce steam into the tube. And in that way we mix the steam with the granules and remove the TCA.

Three main parameters to control here and this is probably the centre of the question. First of all the flow of the steam, secondly the temperature of the steam and thirdly the extraction of the steam. These are the three main points which have to be really controlled in order to have good results. We used the first prototype for granules, cork discs and cork stoppers and the results concerning TCA were similar. But because of the way we fed the cork through this system, we had significant mechanical damage to the cork stoppers and cork discs too. And so we built a second prototype, not a continuous system as you can see here, but a batch system to avoid the mechanical damage in the cork stoppers and discs. We now have industrial pilots for this purpose.

So getting back to our summary, let's talk about where are we concerning cork granules and some results from our validations—independent validations—of the industrial pilot and industrial routine.

I'm not going to bother you of course with laboratory results and prototype results because you have seen them two years ago. These are our internal validations of our industrial pilot for cork granules. We tested batches of positive cork granules—that is cork granules that we detected as contaminated and put aside to make these validations. And so in different days we did several different validations.

First our internal validation. Here are results for a group of samples, showing an average level of TCA in 10 samples before ROSA and an average level of TCA in 10 samples after ROSA. And you can see the results here; we vary from 70.4 per cent reduction to more than 93 per cent. So very good reductions in general terms. We can talk here about something like 80 per cent on average. So after having our system completely controlled we decided to ask independent institutes, reputed research institutes, to validate our results, because we want you to believe these results.

The first results were from the Geisenheim University in Germany. They took 50 samples before ROSA and 50 samples after ROSA. They were in Portugal, they made the sampling and the result they obtained was a 75.1 per cent reduction.

Campden and Chorleywood Food Research Association was the second laboratory that was with us, in February this year. They did a similar thing. They made 50 samples before ROSA and 50 samples after ROSA and they got reductions of around 80 per cent and of course we are talking about a contaminated lot of granules. Actually they were a highly contaminated lot of granules put aside from production.

The third laboratory that was asked to validate the system was the Australian Wine Research Institute (AWRI). Mark Sefton was with us in June this year and he made a validation of the system with a slightly different approach than the others because AWRI wanted to do an homogenization of the bales of cork granules before ROSA and then after ROSA. And we did that with Mark Sefton. We made three samples from two homogenized bales, plus three other samples from two other homogenized bales. So two through unit one and then we passed these granules into ROSA and took six samples of those granules after the treatment. And so with AWRI we did eight units in one day of work, of ROSA. As you can see, the performance was very good with this one exception. That's Unit 1 where probably there is some problem because the reductions were not as high as all the others, which are very reproducible as you can see. So including Unit 1 we are talking about 69.3 per cent reduction, excluding Unit 1 we are talking about 72.5 per cent reduction in TCA.

So in summary we can see that the reductions obtained with the independent validations and with our internal validation… the results obtained were more or less similar to the ones that individually we have obtained on different days with contaminated lots, a reduction of around 70 to 80 per cent in TCA.

But as we can easily imagine, a steam distillation process is not a process limited to TCA. It's a process that is obviously able to take volatiles out of cork granules. So in two of the internal validations we did, we got methyl-iso-borneol (MIB) and guaiacol in the cork granules. And we obtained similar reductions as we did with TCA. We were expecting this kind of values and in fact we got it with guaiacol and MIB.

Here is a picture of one of the ROSA units for treating cork granules installed in a factory that produces cork granules. At this stage we have three ROSA units in the three main factories which are using cork granules, and by the end of this year we will have one more unit in the fourth factory. And so soon all our production for cork granules will be treated by ROSA.

But while we were making all these validations and the optimization of the process at an industrial scale, we did bottling trials because, we know that what you want to know is not if there is a reduction in the process; what you want to know, is if there is no problem with the wine. These results here are from the prototype (I will show you results from the industrial scale afterwards.) When I was here two years ago in October I said to you that in

November I would start a bottling experiment and I did, on 9th November 2001. And so on 9th November 2003, we will have the two-year bottling results. But we have already the results after one month, three months, six months and 12 months of bottling.

We divided a contaminated batch of cork granules, in two parts. One part we passed through ROSA and we produced agglomerate corks. The other part we didn't pass through ROSA and then we produced agglomerate corks. We used the corks in bottles, and after one month we opened 10 bottles and we analysed the wine for TCA. And we did that after three months, six months and 12 months. We've got these results in terms of TCA. Don't forget that we are talking about a highly contaminated lot of granules and the same granules but treated with ROSA producing these results in the wine. So after one year we have no TCA in the wine; we are talking about less than 2 nanograms per litre because that's our quantification limit. So we were very satisfied and very happy with these results.

In this case we did exactly the same thing but using the industrial pilot. So before it was a prototype, here it's already an industrial pilot and this time instead of making 10 bottles each time, we did 25 bottles. These are the three-month results. We have already completed the six months but actually I haven't done the analysis yet. However we start to have a clear difference between those groups of treated and untreated cork stoppers.

So that's exactly where we are with granules. Now let me talk to you a little bit about cork discs and cork stoppers.

I already showed you two years ago lab results and the prototype results for cork discs and cork stoppers. But then we had to build a second prototype because of the damage, the deformation to the cork. But because we already have the industrial pilot I will just show you those results. Here is the industrial pilot, what we call industrial pilot 2 for ROSA, for cork discs and stoppers. This is a simple tumbler where we have a tube in the middle with different arms, and the steam comes through the arm into the tumbler and is then extracted of course. Here are the results of one of our internal validations concerning individual discs, individual corks, groups of discs and groups of corks. We are talking about 50 corks in a soak, 50 discs in a soak.

As you can see we have good results, however sometimes there are cases like this one, and at the start I didn't know exactly why. Cork as you know is very heterogeneous, but I think I can guess why these kind of cases occur. It's because at this stage in this particular factory where we have this prototype, we do not have enough steam to supply all the ROSAs that are there. So we have to have more steam in the factory. I think it's for that reason that these kind of cases appear. Because you will see in the next slide a validation where the results are much better. And that validation was made with the other ROSA units stopped and just this one operating.

On 1 September this year, two months ago, we started to pass cork stoppers through the ROSA system on a daily basis, so each day we did four ROSA treatments. We sampled before ROSA and after ROSA and these results are the average of those eight samples before and eight samples after. In this case

here, where the reduction was not as good as the others, it was because we had one sample after ROSA with 13.3 nanograms a litre which is very big. And that is obvious because if we have a very highly contaminated cork, from the start we know that we are not able to clean to reasonable levels. We are able to clean it. We are able to reduce a significant amount of TCA, but not enough.

Finally I want to show you the results of the validation from the Excell Laboratories, from Pascal Chatonnet. He was in Portugal in September, the middle of September, and he did validations for cork granules, cork discs, cork stoppers too. And this validation was made with only one ROSA unit operating at a time. We do not have all the results yet. These results came to me last Thursday and we have some results from cork discs and cork stoppers: 25 cork discs before ROSA and after ROSA, and 19 cork stoppers before ROSA and after ROSA. The individual results are very good and we are very happy with them, and we are very keen also to have the results from the granules. However these ones were more important because the other Institutes didn't do the validation for discs and stoppers.

We also ran a bottling trial using natural cork stoppers, cork stoppers that were detected as contaminated in a lot. We divided that lot in two. We treated one part of the lot with ROSA and the other part without ROSA. Unfortunately this lot was not very highly contaminated and as you can see the amount of TCA-positive corks is not fantastically high before ROSA. However we can clearly see a tendency for the difference. Here (with ROSA treatment) we have no TCA in the wines compared to what you can find with the lots not treated with ROSA.

Where are we going from here? We now have commercial production of our Neutrocork and Twin Top with ROSA and we need further development on cork discs and stoppers. Mainly we need to have more data on our industrial pilots. And then of course we will have validations and more validations on cork discs and cork stoppers as we did with granules, because right now we just have the first validation coming from Excell Laboratories.

So you can see that we have achieved very significant results with ROSA. We know that there is still work to do of course, but we believe that we are definitely on the right path to defeating the problem of TCA in cork and of course it's what you wine makers have asked us to do. Thank you very much for your attention.

[Applause]