Report on comparative analysis of wines sealed with Vinolok and alternate closures
Report on analysis of wines after 60-month storage period bottled for testing in May 2006

In a letter dated May 17, 2006 you received a report on the bottling of three types of wines, which were stored in our institute with various closures. Per your order the following wines were stored at various storage temperatures (variable warm temperature: 22-28 °C, cold temperature: 15 °C) in both horizontal and upright positions.

1. **Riesling**
   - LATE HARVEST
   - DRY, 2005

2. **Pinot Blanc**
   - LATE HARVEST
   - DRY, 2005

3. **Pinot Noir**
   - LATE HARVEST
   - DRY, 2004
   - Quality Red Wine, (Protected Designation of Origin, PDO)
Per your order bottles with these three wines were sealed with closures in the following variations:

1. **Screw Cap**
   (5 – SE, Silver, Seal: DF 2000B, N2 – 202)

2. **Screw Cap D1**
   (LongCap 30 x 60, Black, Saran tin sealing liner)

3. **Screw Cap D2**
   (LongCap 30 x 60, Yellow, Saranex PVDC sealing liner)

4. **VLG D1**
   (Vinolok selection, Glass, Black cover, PVC Poly One seal)

5. **VLG D2**
   (Vinolok selection, Glass, Manincor silver cover, PVC seal, DS Chemie)

6. **VLG D3**
   (Vinolok selection, Glass, Manincor gold cover, EVA copolymer seal, Elvax 550)

7. **VLP D1**
   (Vinolok classic, Organic glass, Black cover with tax label, EVA copolymer seal, Elvax 550)

For purposes of comparison with these closures, bottles were also sealed with natural cork closures and Nomacorc synthetic cork closures as follows:

8. **Natural cork**
   45 x 24 mm

9. **Nomacorc**
   Classic, 43 x 22 mm

The testing plan assumed comparative evaluation of the test samples sealed with various closures based on different analysis parameters.

For each sample group, sets of bottles were stored under different conditions: horizontal, upright, warm temperature and cold temperature. As assumed in the testing plan, after the 60-month storage period, analyses were performed on bottles stored in a horizontal position at a cold temperature with closure samples 1, 2, 3, 6, 7, 8 and 9.

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Analysis parameters

1. **Visual description of the bottles**
   This parameter checks the impermeability of the bottle seal and the condition of the closures under storage.

2. **CO₂ level in wine**
   This analysis parameter was not investigated after the 60-month period because experience has shown that after 60 months there is no longer a measurable level of dissolved CO₂, and as a result it cannot affect the sensory perception of freshness in any way.

3. **Comparative measurement of wine color**
   Wine color is considered an important evaluation criterion and is directly related (among other things) to the storage method and the sealing capability of the given closure. Along with sensory evaluation of color, spectrophotometric measurement was used to examine changes in color.

4. **SO₂ level in wine (impact of oxygen, oxidation)**
   Analysis of the amount of SO₂ consumed in wines may be used to draw conclusions about the impermeability of closures. The less gas the closure allows to permeate through, the less sulfur dioxide is consumed by oxidation.

5. **Comparative sensory testing of samples**
   The ranking test method was used to evaluate differences between the samples. The analyses were carried out by specially trained wine tasters in the sensory analysis laboratory in the Enology and Wine Technology Department at the Geisenheim Institute.
Analysis results

1. Visual description of the bottles

For each closure sample set, the filled bottles were analyzed after a storage period of 60 months, during which they were tightly sealed and kept at a warm or cold temperature. The closures were positioned fully in the bottleneck and did not show any mechanical damage. All of the screw cap closures were correctly screwed on to the bottle and the glass and organic glass closures were also correctly positioned. There were no externally visible signs of permeability among these test samples either.

2. CO₂ level in wine

As described in the previous section, this analysis parameter was not taken into account following the five-year storage period.

3. Comparative measurement of wine color

The enclosed color value charts show changes in wine color over the five-year storage period for each wine and storage method. Color in both white wines was measured at 420 nm. The default extinction value in the Riesling was 0.081. In bottles stored in a horizontal position, the color values for all closure samples increased to 0.125 on average, i.e. the relative increase was approximately 55%. The slightest color increase was noted in bottles sealed with screw caps (Long screw cap D1) and the greatest color change was noted in test samples sealed with “natural cork” and “Nomacorc Classic” closures.

However, since the color changes were not very considerable, the comparative observation of the closure samples cannot be considered “significant”. The chart below gives a comprehensive picture of the color values obtained in color structure measurements in sets of 10 bottles for each sample group and the default values for Riesling, Late Harvest, Dry, 2005. Average color values measured for various closure samples and different storage methods after five years of storage (15 °C) for Riesling, Late Harvest, Dry, 2005

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11/2011
The default color value for Pinot Blanc was 0.067. On average the color value in bottles stored horizontally/at a cold temperature for all closure samples increased to 0.101, i.e. the relative increase was approximately 50% on average.

As clarified by the following chart, the total color change in this wine was even smaller than in the Riesling. Average color values measured for various closure samples and different storage methods after five years of storage (15°C) for Pinot Blanc, Late Harvest, Dry, 2005

Measurement of default levels in the Pinot Noir red wine identified the following color values at various wavelengths:

<table>
<thead>
<tr>
<th>Wavelength</th>
<th>Color Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>420 nm</td>
<td>0.798</td>
</tr>
<tr>
<td>520 nm</td>
<td>0.685</td>
</tr>
<tr>
<td>620 nm</td>
<td>0.148</td>
</tr>
<tr>
<td>Sum of colors, color intensity</td>
<td>1.631</td>
</tr>
<tr>
<td>Color hue (ratio 420 nm/520 nm)</td>
<td>1.16</td>
</tr>
</tbody>
</table>

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After five years of bottle storage in a horizontal position/at a cold temperature, the color intensity for all closures increased on average to 1.913. The slightest change in color intensity with a value of 1.764 was measured for Long screw cap D2 samples. The greatest change in color intensity with a value of 2.009 was measured in test samples with Nomacorc Classic.

The 420/320 extinction ratio changed in all samples on average to 1.352. In general, wines with a color hue lower than 0.8 were identified as “purple”, those in the 0.8 – 1.2 range as “red” and wines with values greater than 1.2 as “brown-red”. Since in this case the bottles were filled with Pinot Noir Late Harvest Red Wine 2004, the color hue was in between “red” and “brown-red” in general.

Since the changes during the storage period monitored to date had been relatively low, no significant difference between the analyzed test samples could be found. The following chart illustrates the results for Pinot Noir Late Harvest Red Wine.

Measurement of color (color intensity) for various closure samples and different storage methods after five years of storage (15 °C) for Pinot Noir Late Harvest Dry Quality Red Wine PDO, 2004
4. **SO₂ level in wine (impact of oxygen, oxidation)**

Analysis of the amount of SO₂ consumed in wines may be used to draw conclusions about the impermeability of closures. The less gas the closure allows to permeate through, the less sulfur dioxide is consumed by oxidation.

After the 60-month storage period, free and total sulfur dioxide levels were measured in 10 bottles of each sample for each type of wine.

The following charts and graphs illustrate the average values (from n=10) of our measurements in comparison to levels measured for natural cork samples. In addition to the average values, the deviation range for each measurement is shown as minimum and/or maximum data.

Values measured in analysis of Riesling, Late Harvest, Dry are shown first.

Free and total sulfur dioxide levels after 60 months of storage (15 °C) (Average values from n = 10). Riesling, Late Harvest, Dry, 2005
Free and total sulfur dioxide levels after 60 months of storage (15 °C) (Average values from n = 10), Riesling, Late Harvest, Dry, 2005

After the five-year storage period, the test samples for each closure recorded significant decreases in sulfur dioxide levels. One of the notable points was the higher SO2 level in bottles sealed with short screw caps in comparison to bottles sealed with long screw caps. Bottles with Nomacorc Classic synthetic closures showed significantly lower levels than test samples with other closures.

Comparison of total SO2 levels showed similar ratios as for free SO2 levels.
Overview of average values, minimum and maximum values for free and total sulfur dioxide for various bottle closures, 60-month storage period (15 °C)

The text below lists the values measured for Pinot Blanc, Late Harvest, Dry, 2005.

### Riesling, Late Harvest, Dry, 2005

<table>
<thead>
<tr>
<th>Closure</th>
<th>Free sulfur dioxide [mg/l]</th>
<th>Total sulfur dioxide [mg/l]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Average value</td>
<td>Minimum</td>
</tr>
<tr>
<td>Natural cork</td>
<td>16.9</td>
<td>14.5</td>
</tr>
<tr>
<td>Short screw cap</td>
<td>23.7</td>
<td>23.1</td>
</tr>
<tr>
<td>Nomacorc Classic</td>
<td>8.4</td>
<td>7.7</td>
</tr>
<tr>
<td>Long screw cap D1</td>
<td>20.5</td>
<td>19.9</td>
</tr>
<tr>
<td>Long screw cap D2</td>
<td>19.3</td>
<td>17.6</td>
</tr>
<tr>
<td>Vinolok glass D3</td>
<td>18.5</td>
<td>16.6</td>
</tr>
<tr>
<td>Vinolok organic glass D1</td>
<td>16.8</td>
<td>14.4</td>
</tr>
</tbody>
</table>

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Free and total sulfur dioxide levels after 60 months of storage (15 °C) (Average values from n = 10). Riesling, Late Harvest, Dry, 2005

The default levels for Pinot Blanc at bottling were 59 mg/l free SO2 and 138 mg/l total SO2.

After the five-year storage period, the test samples for each closure recorded significant decreases in sulfur dioxide levels.

Similarly to the Riesling test samples, the Late Harvest wine samples also showed a higher SO2 level in bottles with short screw caps in comparison to bottles with long screw caps. Bottles with Nomacorc Classic synthetic closures again showed the lowest levels of free and total sulfur dioxide.

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11/2011
Overview of average values, minimum and maximum values for free and total sulfur dioxide for various bottle closures, 60-month storage period (15 °C)

Free and total sulfur dioxide levels in bottles of Pinot Noir Late Harvest Red Wine sealed with all closure samples were also measured after the 60-month storage period.

Free and total sulfur dioxide levels after 60 months of storage (15 °C). (Average values from n = 10), Pinot Noir Late Harvest Dry Red Wine 2005

<table>
<thead>
<tr>
<th>Closure</th>
<th>Free sulfur dioxide [mg/l]</th>
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<tr>
<td></td>
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</tr>
<tr>
<td>Natural cork</td>
<td>18.2</td>
<td>14.3</td>
</tr>
<tr>
<td>Short screw cap</td>
<td>27.9</td>
<td>26.6</td>
</tr>
<tr>
<td>Nomacorc Classic</td>
<td>10.2</td>
<td>9.7</td>
</tr>
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<td>Long screw cap D1</td>
<td>21.9</td>
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<td>Long screw cap D2</td>
<td>19.3</td>
<td>18.0</td>
</tr>
<tr>
<td>Vinolok glass D3</td>
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<td>17.2</td>
</tr>
<tr>
<td>D1</td>
<td>19.2</td>
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</tr>
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</table>

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Free sulfur dioxide levels after 60 months of storage (15 °C). (Average values from n = 10), Pinot Noir Late Harvest Dry Red Wine 2004

The default levels for Pinot Noir Late Harvest Red Wine at bottling were 73 mg/l free SO₂ and 134 mg/l total SO₂. Given the level of “reductones” in the red wine (which stimulate free sulfur dioxide and which were measured independently) at 19 mg/l, the actual active free sulfur dioxide level at bottling should be calculated as 54 mg/l.

After the five-year storage period, the test samples of red wine for all closure samples showed the most dramatic decreases in sulfur dioxide levels when the three types of wine were compared.

Analysis of the red wine also showed the lowest values of free and total sulfur dioxide in test samples sealed with Nomacorc Classic synthetic closures and natural cork. The most considerable decrease in free SO₂ level, particularly in comparison with the previous time of analysis, was identified in wines sealed with natural cork. In this case, the average value from n = 10 in test samples that was classified as very low was caused by two test samples with extraordinary values of 11.1 mg/l that were included in the calculation.
Free and total sulfur dioxide levels in bottles of Pinot Noir Late Harvest Red Wine sealed with all closure samples were also measured after the 60-month storage period.

Overview of average values, minimum and maximum values for free and total sulfur dioxide for various bottle closures, 60-month storage period (15 °C)

<table>
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<td>Vinolok organic glass D1</td>
<td>20.0</td>
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</tr>
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5. Comparative sensory testing of samples

The analyses were carried out in the sensory analysis laboratory in the Enology and Wine Technology Department at the Geisenheim Institute. Geisenheim Research Institute scientists and staff specially trained in sensory evaluation served as the wine tasters.

Samples of wines stored in a horizontal position/at a cold temperature were selected for tasting after the 60-month storage period. The sensory tests were performed in April 2011 with a set of 12 (Riesling and Pinot Blanc), or 13 (Pinot Noir Late Harvest) trained wine tasters.

The ranking test method enables comparison of several products standing alongside one another. The method is described in DIN 10963, ISO 8587, in S. 35 of the Act on Foods and Items of Daily Consumption (LMBG, i.e. German “Lebensmittel- und Bedarfsgegenstaendegesetz”, method 00.60 4). The ranking test enables classification of two or more test samples into a ranking order according to assigned criteria.

Testing procedure:

The tasters are given two or more test samples in any sequence. The wine tasters must then rank the test samples according to the assigned ranking criteria (e.g. sugar level, acidity, bouquet intensity). With electronic data processing software support, the test samples are assigned random three-digit codes and the samples are given to the tasters in a random sequence.

Properties such as concentration, quality, sensory impressions (such as color, bouquet, flavor) and personal preference can be included in the ranking.

Statistical evaluation:

The tasting results are statistically evaluated according to the calculation procedure stipulated in the DIN standard. First a “F-test” (Friedmann Test) is performed to ascertain whether the group of wine tasters were able to identify generally statistically significant differences between the test samples in the ranking test. If the wine tasters identified differences between the test samples, their responses can be assigned a confidence level of 95%, 99% or 99.9%, in relation to the calculated “F value”. If the test result is not significant, further calculations cannot be performed. In that case the test must be evaluated by stating that the wine tasters ranked the test samples as “equal”.

If the F-test shows that the wine tasters discerned generally significant differences between the test samples, a second calculation procedure called the “comparison of test samples” follows, which consists of analyzing which test samples were identified as significantly different by the wine tasters. The result of this second calculation clarifies whether the wine tasters for example in a test with four products (A, B, C, D) could or could not differentiate test sample A from B, A from C, A from D, B from C, etc. At this point the calculation procedure again differentiates whether the identified difference between two test samples has a confidence level of 95% or 99%.

The sensory tests performed here included four degustation tests for each of the three types of wine, with ranking of test samples stored in a horizontal position and sealed with various closures.
Ranking test result

**Riesling**

**LATE HARVEST**

**DRY, 2005**

In this ranking test with five test samples, wines sealed with natural cork, short screw caps, long screw caps D1, long screw caps D2 and the Nomacorc Classic synthetic closures were first compared to one another.

The wine tasters’ task was to rank the test sample that they perceived as “freshest” and having the “purest color” in bouquet and flavor in first place and to rank the remaining test samples in second to fifth place according to their impressions. In this initial comparison, the wine tasters were unable to find any significant difference between the test samples after the five-year storage period.

In the second ranking test for the Riesling, the test samples with short screw caps were compared with the Vinolok glass and organic glass closures. In this test a significant difference was found between the test samples (95% confidence level of responses). The subsequent comparison of test samples with a 95% level of statistical significance showed a difference between test samples sealed with natural cork, the Vinolok D3 glass closure and the Vinolok organic glass closure. In this test the Vinolok D3 closure sample was evaluated as significantly fresher and purer in color than the other two samples with a 95% level of statistical significance. The graph below illustrates this result.

The third ranking test was structured like the second, except that the comparison included the short screw cap instead of the natural cork closure. The third ranking test was structured like the second, except that the comparison included the short screw cap instead of the natural cork closure. No significant differences between the test samples were found in this test. In the fourth ranking test, a tasting of the three closure samples with Vinolok was performed again, but in this case they were compared to the Nomacorc Classic closure. No significant differences between the test samples were found in this test either.
In this ranking test with five test samples, wines sealed with natural cork, short screw caps, long screw caps D1, long screw cap D2 and the Nomacorc Classic were first compared to one another. Once again the wine tasters’ task was to rank the test sample that they perceived as “freshest” and having the “purest color” in bouquet and flavor in first place and to rank the remaining test samples in second to fifth place according to their impressions.

Here too the wine tasters did not find any significant difference between the test samples. In the second ranking test for the Pinot Blanc, the test samples with short screw caps were compared with the Vinolok glass and organic glass closures. The wine tasters did not find any significant differences between the test samples in this comparison.

Likewise the third ranking test, which included a taste comparison of the two Vinolok samples with the natural cork closure, did not show any significant result.

In the fourth Pinot Blanc ranking test, which like its Riesling counterpart included a tasting of the Vinolok samples and the Nomacorc Classic closure sample, the F-test calculation showed that the wine tasters found a difference between the test samples with a 95% confidence level. The comparison at this level of statistical significance shows that the Vinolok D3 sample was evaluated significantly higher than the other two samples in this comparison.

Generally speaking, after the five years of storage at a cold temperature, there were not clear differences in the sensory character and properties of Pinot Blanc bottled with various closure samples. Significant differentiation between samples in the sensory analysis did not point out any preference on the part of the wine tasters for a particular closure. The only significant difference found was in the comparison between the two Vinolok closures and the synthetic closure.
Pinot Noir
LATE HARVEST
DRY RED WINE, 2004

Sensory ranking tests were also performed for the analyzed red wine comparing various closure samples in the same manner as for the two white wines.