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Sensory and chemical changes of young white wines stored under oxygen. An assessment of the role played by aldehydes and some other important odorants

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Abstract

The aroma of young white wines altered by oxygen was described by a sensory panel which defined the terms: cooked vegetables, liquor, woody, cider and pungent. Twenty-seven young white wines stored under oxygen for 1 week were analyzed by the sensory panel and were further analyzed by gas chromatography (GC)-Ion trap mass spectrometry (MS) to determine their contents in hexanal, 4-hydroxy-4-methylpentanone, 2-nonanone, 2-buthoxyethanol, *t*-2-octenal, 1-octen-3-ol, furfural and 5-methylfurfural, benzaldehyde, *t*-2-nonenal and eugenol. The degrees of aroma degradation induced by oxidation and the acetaldehyde concentration of the wines were measured before and after the oxidation process. The sensory analysis showed that wine aroma degradation is primarily caused by the appearance of a cooked-vegetable odour nuance. The acetaldehyde content of the wines did not vary significantly during the oxidation process, and thereby, cannot be related to the appearance of any of the aroma nuances. Regression data confirm the important role played by eugenol in the woody aromatic nuance, but suggest that important odorants, responsible for the other aromatic nuances, remain unidentified. Some of the compounds analyzed may be used as chemical markers for wine oxidative deterioration. The cooked-vegetable odour nuance can be satisfactorily predicted with quantitative measurements of *t*-2-nonenal, eugenol, benzaldehyde and furfural. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Wine; Oxidation; Aroma; Acetaldehyde; Aldehydes; Eugenol

1. Introduction

The shelf life of wine is a primary concern of the wine industry and, leaving aside the problems caused by different sources of yeast and bacteria, the shelf life of a young wine is directly related to its resistance to oxidation. In fact, wine contains a great number of natural antioxidants belonging to different families of phenolic compounds. These classes of components undergo different oxygen-mediated condensations until they finally precipitate as brown pigments, which cause serious changes in the physical appearance of wine (Cheynier, Basire, & Rigaud, 1989; Fernández Zurbano, Ferreira, Pena, Escudero, Serrano, & Cacho, 1995; Simpson, 1977, 1980, 1982; Singleton & Kramling, 1976; Singleton, Trousdale, & Zaya, 1979). However, very often, wine quality is lost before the changes in colour become apparent, as a consequence of the appearance of several oxygen-related off-flavours. To our knowledge, these off-flavours have not been described, and different descriptors have been used to define what could be the same thing. Aroma terms, such as toasted dry fruits, caramel, overripe fruit, apple, oxidized apple, acetaldehyde, solvent, cement, woody, rancid (Renouil, 1988), vegetative aroma resembling asparagus or straw (Chisholm, Guiher, & Zaczkiewicz, 1995), cooked and rancid (Toukis, 1974), acetaldehyde (Noble, Arnold, Buechsenstein, Leach, Schmidt, & Stern, 1987), or aldehyde (Halliday & Johnson, 1992), have been used to describe wine aroma oxidation. However, there are no precise descriptions of those odours, or estimations of their role in the process of wine aroma degradation.

It is generally believed that acetaldehyde is the main aroma generated during wine oxidation (Baro & Quiros Carrasco, 1977; Meirland & Pernot, 1992; Ribéreau-Gayon, Peynaud, Ribéreau-Gayon, & Sudraud, 1977;

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Usseglio-Tomasset, 1985), although this has not been demonstrated conclusively. On the other hand, several odorants, whose genesis is related to the effect of oxygen, have been identified in wines (Escudero, 1996; Escudero, Cacho, & Ferreira, 2000; Ferreira, Escudero, Lopez, & Cacho, 1998). Some of these compounds are products of the oxidative degradation of unsaturated fatty acids, such as hexanal, *t*-2-octenal, *t*-2-nonenal and 1-octen-3-ol, and others have a miscellaneous origin, such as benzaldehyde, furfural and 5-methylfurfural, 4-hydroxy-4-methylpentan-2-one, 2-nonanone, 2-buthoxyethanol and eugenol. All of them have important sensory properties, but it is not clear whether they play an effective role in the generation of the oxidized aroma.

The goals of the research presented in this paper are to define the aroma notes of white wines oxidized in short periods of time, and to determine the role of acetaldehyde other odorants, previously identified in the perception of those aroma notes. In order to meet the objectives, wines showing different oxidation aromas were first described by a sensory panel. After this, a sample of 27 young white wines was oxidized in the laboratory in a short period of time (1 week). The oxidized samples were scored by the tasting panel and analyzed by a gas chromatography (GC)-mass spectrometry (MS) to determine the targeted aroma compounds. Acetaldehyde was measured before and after the oxidation process. Finally, data have been processed by different statistical techniques.

2. Material and methods

2.1. Wines

As shown in Table 1, 27 bottled young dry white wines were produced in different parts of Spain with non-aromatic grapes. All the wines belonged to the 1999 vintage and the experiment was carried out during May, June and July 2000.

2.2. Wine oxidation

Bottles of wine (750 ml) were transferred to a 1 l sterilized amber bottle and saturated with pure oxygen (99.999%). The remaining headspace of the bottle was also saturated with oxygen to ensure enough oxygen availability during the oxidation process. The bottles were sealed and stored at 20 °C in the dark for 1 week.

2.3. Acetaldehyde determination

A Shimadzu GC-9A with a FID detector was used. The column was from Tecknokroma (2 m×0.32 cm; 5% CW20M in Carbopack BAW 80/120). The carrier gas was N_2 at 20 ml/min. The column was initially at 100 °C and after 2.5 min, was then raised at 25 °C/min up to 180 °C. The injection volume was 1 μl. Fifty microlitres of the Internal Standard Solution (Acetone HPLC quality from Fischer Scientific, 5% v/v in water) were added to 10 ml of the wine sample. Acetaldehyde concentration was determined by interpolation of the relative area in a calibration graph. Analyses were carried out in duplicate. Mean values before and after the oxidation process were compared by a *t*-test.

2.4. Aroma analysis

Concentrations of 11 aroma compounds were obtained, following the procedure described by Ferreira et al. (1998). According to this method, the alcoholic degree of the wine is first adjusted to 12.3% (v/v), spiked with the Internal Standard (2-octanol), and 385 ml of wine are salted out with 43 g of H_2NaPO_4–H_2O and 176.7 g of $(NH_4)_2SO_4$. Two millilitres of the supernatant organic phase are then transferred to a 15-ml tube, diluted with 5 ml of salt solution (35 g NH_4SO_4 in 100 ml water) and extracted with 0.1 ml of Freon 113. This extract is analyzed by GC-MS under the conditions described here:

GC-MS conditions were as follows: A Star 3400CX Gas Chromatograph fitted to a Saturn 4 electronic impact Ion Trap Mass Spectrometer from Varian was used. The column was a DB-WAX from J&W (Folsom, USA), 60 m×0.32 mm; 0.5 μm film thickness, and was preceded by a 3 m×0.32 mm uncoated (deactivated, intermediate polarity) precolumn. The carrier was He at 1 ml/min. The column was initially at 40 °C and after 5 min was then raised to 190 °C at 2 °C/min. The transfer line was kept at 220 °C. A 1093 SPI (Septum-equipped Programmable Injector) from Varian was used for the injection of 1 μl of sample. The injector was initially at 30 °C for 6 s, and was then raised to 190 °C at 200 °C/min. Mass Spectrometry: Mass Range, 35–200 m/z, 1 scan s^{-1}. Ionization was carried out via automatic gain control. Quantitative data were obtained by interpolation of the peak relative areas in the calibration graph prepared by analyzing synthetic wines containing known amounts of the volatile components.

2.5. Sensory analysis

The tasting panel was composed of 15 people (eight male and seven female, average age 35 years) selected by their ability to memorize and recognize odours and because of their experience in wine tasting. Descriptors were defined in five previous sessions. No pre-established list of sensory terms was used. First, each individual was asked to describe a series of oxidized wines with the terms of his/her choice. At the end of each session, the hedonic and the a priori non-pertinent terms were eliminated in a general discussion with the coordinator of the panel. Six terms were finally selected and, to ensure unique and common meanings for all of the terms, reference standards were proposed by the coordinator of the panel, following suggestions given by the members of the panel. Once the sensory descriptive terms were fixed, four additional training sessions were run with different oxidized wines and the six reference standards defined in Table 2. Tasters were asked to mark with one x the most important descriptor of the wine from a list including the six sensory terms previously defined.

Finally, the 27 wines of the sample were sensorially evaluated in duplicate in 12 sessions. In each session, the tasters had at hand the six reference standards defined in Table 2. Wines (30 ml, 20 °C) were presented in random order in covered standard tulip cups marked with a three-digit code. Tasters were asked to mark the most important note of those of the training sessions.

The members of the panel were trained in other sessions to evaluate the degree of deterioration of the wine aroma with different oxidized wines. A six-point aroma degradation scale (AD) was defined as follows: 0, no sign of oxidation; 1, very slight oxidation symptoms; 2, slight oxidation symptoms; 3, enough level of oxidation to reject the wine; 4, strong level of aroma deterioration by oxidation and 5, very strong level of aroma deterioration by oxidation. Once the AD scale was fixed, two additional training sessions were run with different oxidized wines.

Table 1
Sample identity and acetaldehyde concentration[a] before and after the oxidation

Wine	Origin[b]	Grapes	Acetaldehyde in non oxidized wine	Acetaldehyde in oxidized wine
V1	Calatayud	Macabeo	45.5	38.9[c]
V2	La Mancha	Airen	44.6	44.8
V3	Alella	Pansa blanca	79.9	77.0
V4	Rioja	Viura	46.4	48.7
V5	Rioja	Viura	48.2	46.1
V6	La Mancha	Airen/Macabeo	79.9	79.7
V7	La Mancha	Airen	47.4	41.2[c]
V8	Montilla	Macabeo	107	102
V9	Penedés	Parellada	33.0	28.3[c]
V10	Cariñena	Macabeo	61.8	55.2[c]
V11	Cariñena	Macabeo	44.1	48.1
V12	Cariñena	Macabeo	87.2	99.8[c]
V13	Cariñena	Macabeo	194	205
V14	Cariñena	Macabeo	48.5	49.4
V15	Cariñena	Macabeo	39.2	41.2
V16	Cariñena	Macabeo	42.6	44.9
V17	Borja	Macabeo/Moscatel	15.8	17.5
V18	Somontano	Macabeo/Alcañón	74.5	75.6
V19	Somontano	Macabeo	67.9	76.3[c]
V20	Somontano	Macabeo	39.4	44.0[c]
V21	Cariñena	Macabeo	87.9	84.0
V22	Cariñena	Macabeo	98.7	101
V23	Costers Segre	Chardonnay/Macabeo/Xarello	58.2	57.5
V24	Somontano	Macabeo	72.3	74.9
V25	Borja	Macabeo	66.6	71.4
V26	Borja	Macabeo	63.4	65.5
V27	Borja	Macabeo	65.7	65.9

[a] Acetaldehyde concentration in mg/l (average of two determinations).
[b] All of them are Spanish regions within the system of "Appelations de origin Controlés".
[c] A *t* test comparison of the means before and after the oxidation gave a significant value ($P<0.05$).

Table 2
Aromatic terms used in sensory analysis and reference standard composition

Terms	Reference standard composition
"Cooked vegetables"	2 g of cooked green bean macerated in 50 ml of white wine during 2 h
"Liquor"	5% of Bourbon in white wine
"Woody"	5% of whisky in white wine
"Rancid"	2 ppb of *t*-2-nonenal in white wine
"Pungent"	2 g of overripe melon macerated in 50 ml of white wine with 10 mg/l of butiric acid during 2 h
"Cider"	15% of cider in white wine

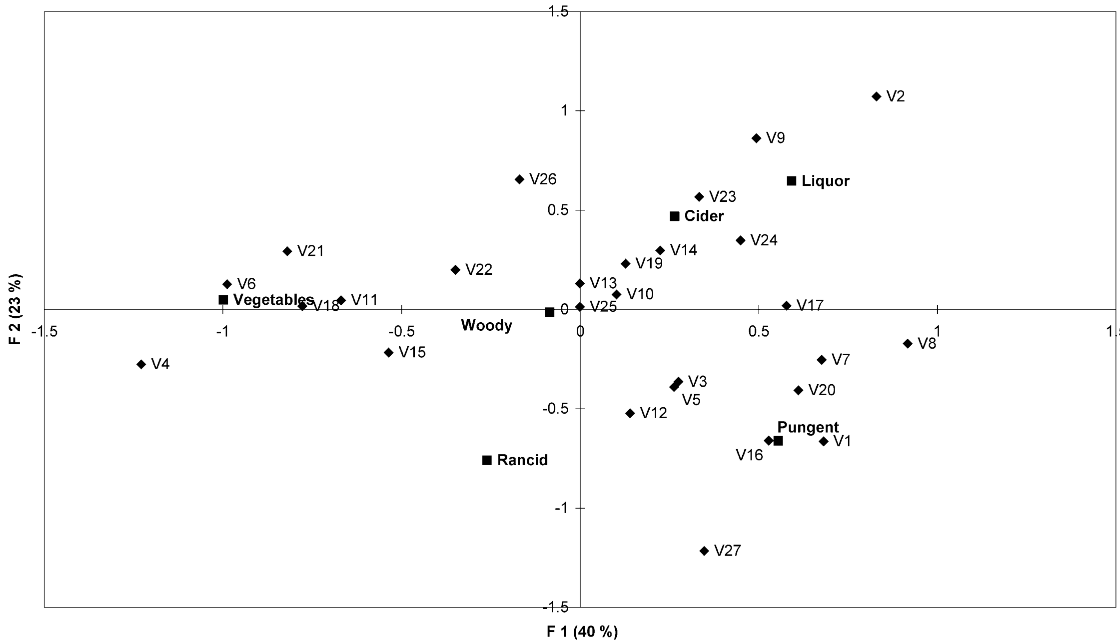

Fig. 1. Correspondence factorial analysis: projection of the 27 wine samples and the six odour descriptors in the plane formed by the first and the second axis.

The 27 wines of the sample were sensorially evaluated in duplicate in 20 sessions. Samples were controlled by the panel coordinator before and after the oxidation process to ensure that off-odours had really been generated before presenting the samples to the sensory panel.

Wines (30 ml, 20 °C) were presented in random order in covered standard tulip cups marked with a three-digit code. Tasters were asked to score the AD of the wines.

The data matrix was built by counting the number of x with which each sample was marked for each descriptor. With this contingency matrix, a Factor Correspondence Analysis (FCA) was carried out to verify the degree of convenience of the different sensory descriptors.

Different multiple and simple lineal regression studies were carried out, searching for some link between the AD scale and both the sensory data and chemical quantitative data.

The software package used was StatBox 2.1 (Grimmer logiciel, Paris, France).

3. Results and discussion

The descriptors finally used by the members of the panel to describe the aroma of the oxidized samples, together with the reference standards used to define them, are shown in Table 2. Out of the six terms in the table, only the term ‘‘cooked vegetables’’ has some correspondence with the aroma terms defined by Noble et al. (1987) in the wine aroma wheel. It is also worth mentioning that the term ‘‘acetaldehyde’’, the only term of the aroma wheel directly related to oxidation, was not used to describe the oxidized wines of this study. This is due to the fact that such a term is used to describe the aroma of wines that have undergone an oxidative aging, such as Sherry or Port wines. This study is, however, focussed on the off-flavours that appear in the first stage of the spontaneous and undesired oxidation of young wines.

It became obvious, since the beginning of the experiment, that the appearance of some of the aroma nuances described in Table 2 can deeply distort the aromatic profile of wine, and that some of the oxidized samples could develop very strong and dominant off-flavours. Consequently, it was considered to be more meaningful to assign the wine to an off-flavour category rather than to score all the studied nuances of the wine as is done in normal quantitative descriptive analysis (Stone & Sidell, 1993). Sensory data were subsequently obtained by counting the number of times that each wine was assigned to a given category.

The pertinence of the six sensory descriptors selected was studied by a FCA performed on the contingency matrix. Fig. 1 shows the projection of the different samples and sensory descriptors on the plane formed by the two first axes (with 63% of the total variance of the system). The terms ‘‘cooked vegetables’’, ‘‘pungent’’, ‘‘woody’’, ‘‘cider’’ and ‘‘liquor’’ can be considered pertinent since, as it is shown in Fig. 1, there are a good number of samples in the regions close to them. However, the descriptor ‘‘rancid’’ appears in an area of the plane in which there are no samples. In fact, this term was used only 3% of the time. Consequently, this descriptor was considered to be non-pertinent and was excluded from the analysis. The discriminant power of the different terms is related to their distance from the centre of the space in the FCA representation. As can be seen in Fig. 1, the term "cooked vegetables" is the most discriminant, followed by "pungent" and "liquor". The term "woody", although in the plane shown in Fig. 1 seems to be very close to the center of the plane, is also discriminant. This becomes obvious when the terms are represented in the plane formed by the first and the third Factors (figure not shown). Fig. 1 and data in Table 3 allow us to conclude that the five selected terms are not redundant, since they are not correlated. In fact, the highest correlation coefficient between sensory terms was −0.391 ("pungent" vs. "cooked" vegetables).

The loss of quality induced by the oxidation of the wine was measured through an aroma degradation scale, defined in the experimental section. Before the oxidation period, all the wines scored 0 or 1, which means that they were not affected by oxidation. After one week under oxygen, 18 out the 27 wines were rejected, and their AD scores in all cases, had increased. A correlation study between these AD scores and the different flavour nuances is shown in Table 3. As can be seen in italic format in Table 3, the only term which shows a direct and significant correlation with the aroma degradation score is the term "cooked vegetables" ($r = 0.596$, significant at $P < 0.001$), which, thereby, can be regarded as the main contributor to the loss of wine quality. Another important observation is the fact that the term "liquor" is negatively correlated with the aroma degradation scale ($r = -0.348$, significant at $P < 0.1$), which seems to indicate that this flavour nuance can not be considered as an off-flavour of wine.

A stepwise multiple linear regression study allowed us to develop a function able to predict the AD score of a wine from its aromatic profile. This function is:

$$\text{AD} = 2.74 + 0.067\text{``Cooked Veg''} + 0.067\text{``Pungent''}$$

$$r = 0.728, \text{ significant at } P < 0.05$$

which clearly indicates that the aromatic nuances generated in a short oxidation that induce a stronger effect on wine quality are "cooked vegetables" and "pungent". A plot of the residuals (difference between the estimated and the measured AD scores) vs. the estimated values, showed a random distribution (figure not shown), which indicates that the regression errors are independent.

Wine acetaldehyde concentrations, before and after the oxidation, are shown in Table 1. Table 3 shows the correlation coefficients between these concentration data, the different flavour nuances and the AD scores. Table 1 indicates that acetaldehyde concentration surprisingly does not change significantly in the process. Only in some wines is there a small increase, but never

Table 3
Correlation matrix between the aromatic notes, the aromatic degradation (AD) scores and the level of acetaldehyde

	"Cooked vegetables"	"Liquor"	"Woody"	"Pungent"	"Cider"	Acetaldehyde in non-oxidised wine	Acetaldehyde in oxidised wine	Acetaldehyde increment	AD
"Cooked vegetables"	1								
"Liquor"	−0.341137	1							
"Woody"	0.007615	−0.248079	1						
"Pungent"	−0.391723	−0.10194	−0.280661	1					
"Cider"	−0.180242	0.091685	−0.039906	−0.152638	1				
Acetaldehyde in non-oxidised wine	0.059073	−0.14307	0.1811232	−0.333149	0.302805	1			
Acetaldehyde in oxidised wine	0.065981	−0.151423	0.198812	−0.341728	0.247489	0.9911354	1		
Acetaldehyde increment	0.073955	0.20119	0.20119	−0.20093	−0.266436	0.358383	0.47778	1	
AD	*0.596316*	−0.0153	−0.0153	0.150848	−0.051712	0.0668588	0.069602	0.036017	1

Table 4
Concentration (μg/l) of 11 odorants quantified in 27 oxidized wines

	Range[a]	Mean	S.D.[b]
Hexanal	<M.Q.L.–112	62.3	38.9
4OH-4 M-Pentanone	<M.Q.L.–297	54.3	100
2-Nonanone	10.3–49.2	15.8	12.2
2-Butoxgethanol	<M.Q.L.–73.0	66.1	64.1
t-2-Octenal	<M.Q.L.–10.8	3.2	3.3
1-Octen-3-ol	5.1–16.2	9.7	2.8
Furfural	15.8–342	90.2	22.3
Benzaldehyde	20.0–313	165	24.1
t-2-Nonenal[c]	30.1–151.2	62.6	24.8
5M-Furfural	9.9–59.9	40.5	12.4
Eugenol	1.6–53.7	6.2	14.4

[a] <M.Q.L.: Less than minimum quantitative level.
[b] Standard deviation of the mean obtained by averaging the results from the 27 wines.
[c] Concentration in ng/l.

Table 5
Simple regression between the descriptors and the quantified compounds

Descriptor	Compounds with which it is correlated
"Cooked vegetables"	Furfural*
"Liquor"	Hexanal*
	Benzaldehyde**
"Woody"	Hexanal**
	t-2-Octenal**
	Furfural***
	Eugenol***
	5M-Furfural***
"Pungent"	Hexanal*

* Significant at $P>90\%$.
** Significant at $P>95\%$.
*** Significant at $P>99\%$.

Table 6
Stepwise linear regression analysis

Functions	*r*
"Cooked vegetables" = 1710 *t*-2-nonenal − 24272 eugenol + 132 benzaldehyde + 27188 furfural − 11.6	0.617
"Liquor" = 28059 hexanal + 32382 *t*-2-octenal + 8.79	0.495
"Woody" = −6882 furfural + 4694 2-nonanone + 100 benzaldehyde + 1012 *t*-2-nonenal − 3.26	0.627
"Pungent" = −27301 hexanal − 9623 furfural + 5473 4OH4M2pentanone − 0.91 acetaldehyde increment + 24.7	0.563

higher than 14% of the original concentration. This clearly demonstrates that acetaldehyde cannot be an important contributor to the aroma of the wines oxidized in short periods of time. This is corroborated by data in Table 3 which, as expected, show that neither the acetaldehyde concentration, nor its increase during the oxidation, are correlated with the flavour nuances or with the AD score. It can therefore be concluded, that the changes induced in wines in short periods of oxidation have nothing to do with acetaldehyde concentration.

With regard to the other 11 odorants measured in the study, Table 4 shows their concentration range, their mean and their standard deviation. Table 5 summarizes the results of the correlation study between these components and the different aromatic nuances previously measured. None of the 11 odorants was correlated with the term "cider", and only furfural was correlated with the descriptor "cooked vegetables". The term "liquor" is correlated to hexanal and to benzaldehyde. The aromatic term with higher correlation coefficients any of the chemical compounds is the term "woody". The most remarkable result is its high correlation with eugenol. In a previous study (Ferreira, Escudero, Fernandez, & Cacho, 1997) it was shown that the levels of eugenol increased upon wine oxidation. Moreover, eugenol was detected by gas chromatography-olfactometry (GCO) as an important odour linked to oxidation (Escudero et al., 2000). Data in the table suggest that it has an actual role in the perception of the woody note. This could explain why old and oxidized wines are often described as "maderized", even though they have not had any contact with wood. Furfural and 5-methylfurfural could also be involved in the perception of that note. It is remarkable that some important odorants quantified, such as *t*-2-nonenal or 1-octen-3-ol were not correlated with any of the odor nuances generated in the wines. This means that their role had been previously overemphasized or that several volatile compounds combined together are responsible for the off-odours detected in the oxidized wines.

Finally, a stepwise multiple linear regression analysis was carried out in order to search for models that could link the different odour nuances with the contents of some chemicals generated in the oxidation process, independently of their sensory properties. Those results are presented in Table 6 and show that some of the compounds analyzed could be considered as chemical markers for wine oxidative deterioration. Particularly, the odor nuance "cooked vegetables" can be satisfactorily predicted from the concentrations of *t*-2-nonenal, eugenol, benzaldehyde and furfural.

In conclusion, this research has shown that different aromatic nuances can be produced in young white wines oxidized in short periods of time, and that the odour nuance "cooked vegetable" has a profound effect on wine quality. It has also been demonstrated that acetaldehyde has nothing to do with these aromatic nuances nor with the flavour deterioration of these wines.

Finally, regression data confirm the important role played by eugenol in the ''woody'' aromatic nuance, but suggest that important odorants responsible for the other aromatic nuances remain unidentified.

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