

Hydroxylated Stilbenes in Selected American Wines

Olusola Lamikanra,^{*,†} Casey C. Grimm,[‡] J. Ben Rodin,[†] and Inyang D. Inyang[†]

Center for Viticultural Science and Small Farm Development, Florida A&M University, Tallahassee, Florida 32307, and Southern Regional Research Center, Agricultural Research Service, U.S. Department of Agriculture, 1100 Robert E. Lee Boulevard, New Orleans, Louisiana 70124

Hydroxylated stilbene contents of a number of commercial American red wines were determined by gas chromatography–mass spectrometry. Six stilbenols were eluted. Two of these were identified as *cis* and *trans* isomers of 3,5,4'-trihydroxystilbene (resveratrol), while the others were tentatively identified as mono-, di-, tri-, and tetrahydroxystilbenes. *cis*- and *trans*-resveratrol were present in the wines at concentrations ranging from 0.1 to 31.9 ppm for *cis* and from 0.0 to 13.4 ppm for *trans*. The concentrations of resveratrol in wines made from *Vitis rotundifolia* (muscadine) grapes were higher ($P > 0.05$) than those present in wines made from *Vitis vinifera* and *Vitis labruscana* grapes. Tetrahydroxy contents of Muscadine and Concord wine were, in general, lower than that of the *V. vinifera* wines. A method for analyzing hydroxylated stilbene compounds in wine by flame ionization detection is reported.

Keywords: *Resveratrol*; *stilbenol*; *red wine*; *muscadine*; *Vitis*

INTRODUCTION

The production of hydroxylated stilbene compounds by woody plants in response to fungal infection and other forms of stress has been investigated for many years (Creasy and Coffee, 1988; Jeandet et al., 1991; Langcake and Pryce, 1976; Jeandet et al., 1995a). These stilbenols and related oligomeric compounds exhibit antifungal and antibacterial activities (Lins et al., 1990; Pool et al., 1981; Dercks and Creasy, 1989; Langcake, 1981). Interest in *trans*-resveratrol (3,5,4'-trihydroxystilbene) has increased considerably following reports suggesting potential health benefits, such as the inhibition of platelet aggregation in rats, lowering of lipid levels in rat liver following oral administration, and inhibition of human low-density lipoprotein oxidation (Kimura et al., 1985; Frankel et al., 1993; Waterhouse, 1995). Resveratrol has also been identified as the active ingredient in some Chinese and Japanese folk medicines used for the treatment of a number of diseases and infections (Siemann and Creasy, 1992). In the grapevine (*Vitis* sp.), resveratrol serves as a precursor to potent stilbene phytoalexins such as viniferins (Langcake and Pryce, 1977).

The association of red wines with platelet hypocoagulation and an increase of high-density lipoprotein (HDL) cholesterol in humans has been attributed to the presence of resveratrol in these wines (Goldberg et al., 1993; Waterhouse, 1995). Wines and grape skin extracts have also been reported to have vasorelaxing properties (Fitzpatrick et al., 1993). The concentration of resveratrol in wine varies considerably and appears to be influenced by geographic location, wine type, and grape cultivar. Resveratrol has been shown to exist in wine as both the aglycon and the bound glucoside called piceid (Jeandet et al., 1994; Waterhouse and Lamuela-Raventos, 1994; Lamuela-Raventos et al., 1995). The aglycon resveratrol content of *Vitis vinifera* and *Vitis*

labruscana red wines has been reported to range between 0.1 and 15 ppm (Lamuela-Raventos and Waterhouse, 1993; Mattivi, 1993; Goldberg et al., 1993, 1994, 1995a,b; Jeandet et al., 1995b,c; Peret et al., 1994; Siemann and Creasy, 1992; Soleas et al., 1994).

Vitis rotundifolia (muscadine) grapes are grown throughout the southeastern United States. They are highly resistant to many pathogenic fungi and bacteria that infect many grape species and are traditionally used for wine and juice making in the region. There is no report concerning the resveratrol aglycon content of wines made from muscadine grapes. The objective of this study is to determine the hydroxylated stilbenes contents of representative commercial red muscadine wines and to compare them with those present in nonmuscadine red American wines.

MATERIALS AND METHODS

Wine Samples. Three bottles of each brand of nonmuscadine wines were purchased from retail stores. Muscadine table wines were made primarily from the Noble cultivar and were obtained from retail outlets at wineries. Muscadine port wine was vinified as described by Lamikanra (1987) except that the sugar level, after the initial skin fermentation, was raised to 24 °Brix and fermentation was terminated at 5 °Brix by addition of absolute ethanol to give a final alcohol content of 18%.

Grape Samples. Noble muscadine grapes were harvested from the variety trial vineyard at Florida A&M University, Tallahassee, FL. At harvest, the average water-soluble content of the fruits was 14.5%, titratable acidity was 0.67% (as tartaric acid), and pH was 2.97. The fruits were rinsed with sulfite solution (100 ppm). Undeseeded berries (500 g) were pulverized in a Vita Mix blender for 2 min. Blended grapes were lyophilized and stored at -80 °C until analyzed.

Sample Preparation for GC Analysis. Extraction of hydroxylated stilbenic compounds from the wines was carried out using C₁₈ extraction cartridges (Soleas et al., 1993). The cartridge was preconditioned with methanol and water. Wine samples (12 mL), to which stilbene solution was added as internal standard to give a total stilbene concentration of 3 ppm, were passed through the cartridge, and hydroxylated stilbenic compounds were eluted from the cartridge with ethyl acetate (1 mL). The eluate was evaporated to dryness with nitrogen gas, and to the dry residue was added 0.5 mL of bis-

* Author to whom correspondence should be addressed [telephone (904) 599-3996; fax (904) 561-2617; e-mail olamikan@ns1.famu.edu].

† Florida A&M University.

‡ U.S. Department of Agriculture.

Table 1. Mass Spectral Data of Trimethylsilyl Derivatives of Stilbenols in Muscadine Wine^a

retention time (min)	compound	concentration (ppm)	characteristic MS data <i>m/z</i> ^b
13.38	monohydroxystilbene	<1.0	M ⁺ = 268; 73 (100), 207 (26); <i>m/z</i> 268 SIM only
16.15	dihydroxystilbene	<1.0	M ⁺ = 356; 73 (100), 207 (31); <i>m/z</i> 356 SIM only
18.19	dihydroxystilbene derivative	~20.0	M ⁺ = 414; 73 (100), 281 (63), 414 (29), 133 (7), 239 (6), 179 (6), 311 (6), 147 (5), 207 (3), 399 (2), 369 (2)
22.48	<i>cis</i> -resveratrol	21.7	M ⁺ = 444; 73 (100), 444 (23), 5 (12), 207 (8), 147 (5), 133 (4), 208 (3), 281 (2), 355 (1)
25.81	<i>trans</i> -resveratrol	13.4	M ⁺ = 444; 73 (100), 207 (35), 75 (16), 96 (12), 133, 208, 281 (9), 444 (7), 191 (6), 135 (4), 103 (3)
27.43	tetrahydroxystilbene	4.1	M ⁺ = 532; 73 (100), 207 (27), 368 (10), 96, 147, 281 (7), 133, 179, 355 532 (5)

^a Muscadine wine sample. ^b Relative intensity.

[trimethylsilyl]trifluoroacetamide (BSTFA). The solution was held at 60 °C for 30 min and then left at room temperature overnight.

For analysis of dry grapes, methanol (5 mL) was added to the dry powder (500 mg) and stirred for 30 min. The mixture was centrifuged and the supernatant passed through a C₁₈ extraction cartridge. The ethyl acetate eluate from the column was treated as described above for the wine samples.

Gas Chromatography Analysis. Injections of 2 μ L of the derivatized solution were made into a Hewlett-Packard 5988A GC-MS. A 50 m, 0.25 mm i.d., DB-5 capillary column was held for 1 min at 150 °C then ramped at 10 °C/min to 300 °C. Selected ions were monitored for 100 μ s each on *m/z* 179, 180, 268, 276, 326, 356, 414, 444, and 446. Total cycle time for the mass spectrum was approximately 1 s. Stilbene was quantified on *m/z* 180, while the tris(trimethylsilyl)stilbene was quantified on *m/z* 444. Standards of stilbene and *trans*-resveratrol were acquired from Sigma Chemical Co., St. Louis, MO. Isomerization of *trans*-resveratrol to *cis* was accomplished by exposing *trans*-resveratrol solution to UV light (254 nm) for 12 h (Siemann and Creasy, 1992). Stock solutions were prepared by solvation in ethyl acetate with the aid of a sonic water bath. A 50 μ L aliquot of the standard was then treated with 50 μ L of BSTFA and heated at 60 °C for 30 min. Serial dilutions were then made from the stock solution. All values were normalized to the response of a standard of 0.3 ng of stilbene. Wines analyzed without the internal standard had stilbene contents that were negligible and below the detection level of the equipment. The adjusted resveratrol peak areas were converted to nanograms of resveratrol using the calibration curve generated from the serial dilutions of the resveratrol standard. Correlation coefficient for the curve was 0.99578. Concentrations are reported in parts per million (ppm), and values were calculated on the basis of the initial starting volume of 12 mL of wine. Standards of mono-, di-, and tetrahydroxystilbene were not available, and their retention times could not be confirmed. Tentative identifications of these compounds were thus based on their molecular ions and fragmentation patterns (Table 1). MS data of trimethylsilyl derivatives of these compounds do not appear to have been published.

A 30 m, 0.53 mm i.d., capillary column consisting of 95% dimethyl and 5% diphenyl stationary phase was used for the flame ionization detection (FID) of hydroxylated stilbene compounds. The column was held at 50 °C for 1 min, after which time it was ramped to 250 °C at 5 °C/min. It was then held at 250 °C for 5 min, after which time it was increased to 300 °C. The split injector temperature was at 220 °C and closed for 1 min following the injection. Detector temperature was held at 280 °C. The head pressure on the column was increased to 10 psi. Peaks of *cis*- and *trans*-resveratrol were identified from retention times of standards.

RESULTS AND DISCUSSION

The total ion chromatogram of the trimethylsilyl (TMS) derivative of ethyl acetate extract of muscadine wine and the chromatogram detected with flame ionization detector (FID) are shown in Figures 1 and 2, respectively. In addition to *trans*-trihydroxystilbene, its

Table 2. Amounts of Resveratrol and Tetrahydroxystilbene in Wines (Parts per Million)

wine	<i>cis</i> -resveratrol	<i>trans</i> -resveratrol	tetrahydroxystilbene
muscadine A ^a	31.9 ^a	12.2 ^a	27.4 ^a
muscadine B	23.6 ^b	9.2 ^b	2.0 ^{e,f}
muscadine C	9.2 ^c	4.9 ^{c,d}	0.4 ^f
muscadine D	21.7 ^b	13.4 ^a	4.1 ^{d,e,f}
muscadine E	19.3 ^b	5.8 ^c	6.2 ^{c,d,e,f}
Cabernet Sauvignon A	1.0 ^d	1.3 ^{g,h}	16.6 ^b
Cabernet Sauvignon B	3.3 ^{c,d}	4.5 ^{c,d}	13.1 ^{b,c}
burgundy A	0.9 ^d	1.3 ^{f,g,h}	8.5 ^{c,d,e}
burgundy B	0.8 ^d	1.1 ^{g,h}	10.1 ^{b,c,d}
merlot A	2.6 ^d	3.5 ^{d,e,f}	11.1 ^{b,c,d}
merlot B	3.3 ^{c,d}	3.8 ^{c,e,d}	13.4 ^{b,c}
zinfandel A	1.9 ^d	3.6 ^{e,d}	13.1 ^{b,c}
zinfandel B	1.7 ^d	2.0 ^{e,f,g,h}	12.6 ^{b,c}
Concord A	1.5 ^d	1.1 ^{g,h}	0.9 ^{e,f}
Concord B	4.0 ^{c,d}	2.7 ^{d,e,f,g}	6.2 ^{c,d,e,f}
muscadine port	3.3 ^{c,d}	3.6 ^{e,d}	3.4 ^{d,e,f}
<i>V. vinifera</i> port A	0.3 ^d	0.0 ^h	0.2 ^f
<i>V. vinifera</i> port B	0.1 ^d	0.0 ^h	0.0 ^f

^a Capital letters indicate different brand names for each wine type; lower case letters in column without the same letters are significantly different ($P > 0.05$, $n = 3$).

cis isomer and other hydroxylated stilbene compounds were present in each wine studied. Mass spectra of *cis*- and *trans*-resveratrol were identical. On the basis of the molecular ions and fragmentation patterns (Table 1), peaks eluted after 13.38, 16.15, and 27.43 min were tentatively identified as monohydroxystilbene (*m/z* 268), dihydroxystilbene (*m/z* 356), and tetrahydroxystilbene (*m/z* 532), respectively. In addition, another dihydroxystilbene compound with an additional moiety of 43 amu (MW 414 amu) was observed. The mass spectrum would suggest a trihydroxystilbene with two TMS moieties and one alditol acetate. Mass spectral data for these stilbenols and their retention times are shown in Table 1. Three of these compounds [*cis*-resveratrol, *trans*-resveratrol, and tetrahydroxystilbene (GC-MS retention times 27.06, 32.12, and 34.37 min, respectively)] were identified in the FID trace. The elution profile also suggests that other compounds identified by the mass spectrometric method were also present at low concentrations. An FID detection method for the determination of resveratrol concentration in wines is particularly useful because equipment for such analysis is relatively common and widely used for analyzing volatile compounds.

A comparison of the *cis*-, *trans*-, and total resveratrol contents of the 18 different wines analyzed showed that muscadine table wines had relatively higher resveratrol contents than nonmuscadine grape wines (Table 2). Tetrahydroxystilbene contents in muscadine wines were in most cases lower than the amounts determined for *V. vinifera* wines but comparable to the amounts

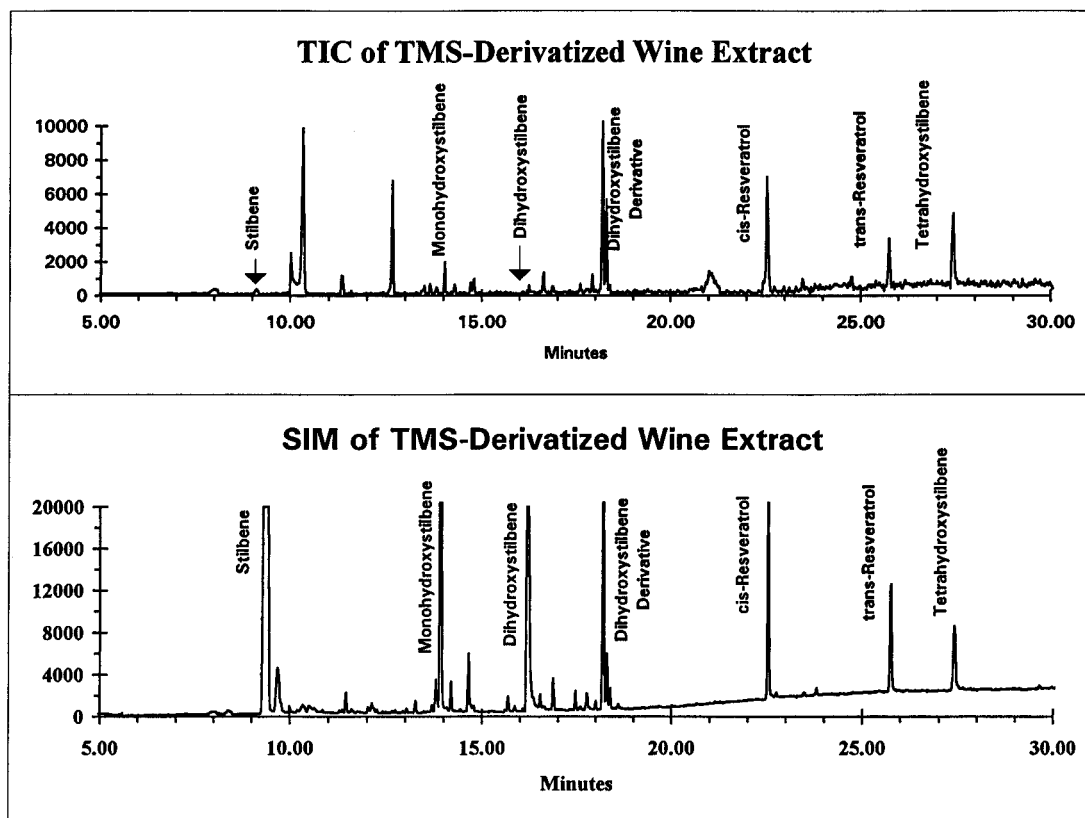


Figure 1. Total ion and selected ion chromatograms of derivatized muscadine wine extract.

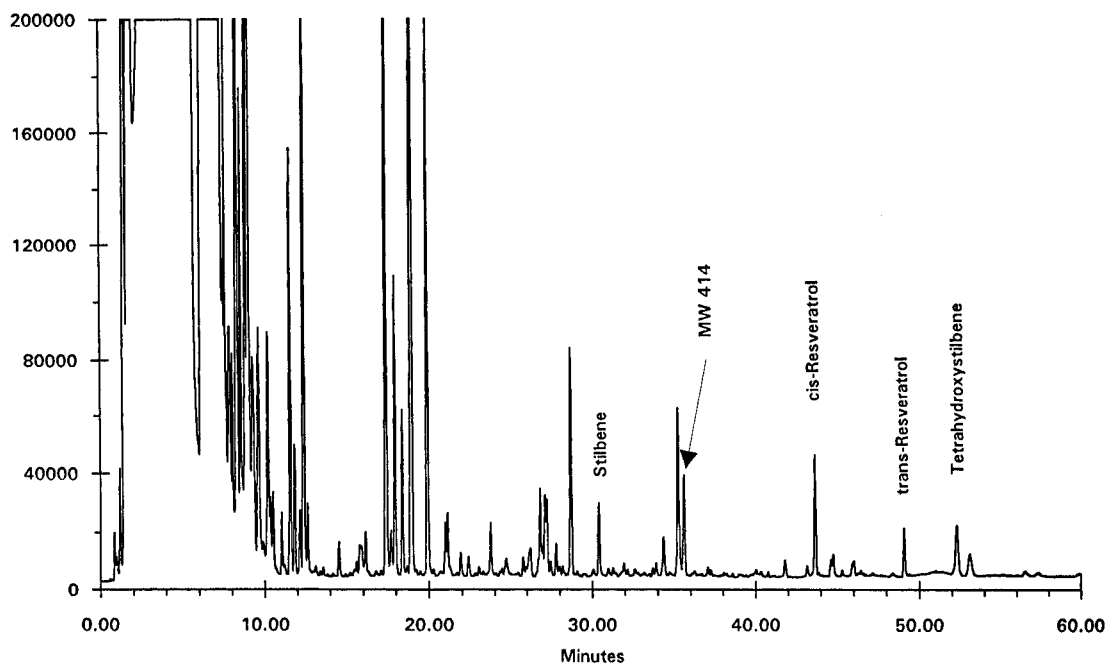


Figure 2. Flame ionization detector trace of TMS derivatized wine extract.

detected in the only *V. labruscana* wine (Concord) studied. The muscadine wine (sample A) that had a relatively high level of tetrahydroxystilbene also had the highest level of resveratrol. This was the only muscadine wine that was barrel fermented. Consistent with earlier reports (Goldberg et al., 1993), *V. vinifera* port wines had very little resveratrol and tetrahydroxystilbene contents were relatively low. The muscadine port wine (sample) had lower stilbenol content than the muscadine table wines. The concentration of these compounds in the muscadine port wine was, however,

much higher than those of the *V. vinifera* port wines and comparable to the amounts present in *V. vinifera* table wines. Analysis of the average mean obtained for table wines of each species (i.e. *V. rotundifolia*, *V. vinifera* and *V. labruscana*) also showed a significantly higher value for muscadines than the other grape species (Table 3). In cv. Noble muscadine grape berry the trans isomer is the dominant hydroxystilbenic compound, with 5.6 mg/100 g of the fresh berry weight. *cis*-Resveratrol and tetrahydroxystilbene contents are 0.08 and 0.05 mg/100 g of fresh fruit, respectively.

Table 3. Average Mean Values of Resveratrol and Tetrahydroxystilbene in *V. rotundifolia*, *V. vinifera*, and *V. labruscana* Table Wines (Parts per Million)

species	<i>cis</i> -resveratrol	<i>trans</i> -resveratrol	tetrahydroxystilbene
<i>V. rotundifolia</i>	21.2 ^a	9.1 ^a	11.9 ^a
<i>V. vinifera</i>	3.3 ^b	3.6 ^b	8.0 ^a
<i>V. labruscana</i>	2.7 ^b	2.6 ^b	3.5 ^a

^aLetters in each column without the same letters are significantly different ($P > 0.05$).

Most reports concerning resveratrol have focused on the *trans* isomer content, apparently because of its dominance in nonwine grape products. Recent evidence indicates that the *cis* isomer may be as effective in lowering serum lipid levels as *trans*-resveratrol (Jayatilake et al., 1993). *cis*-Resveratrol is observed in all wines analyzed in this study. Since *trans*-resveratrol can be converted to the *cis* isomer by UV light, the total resveratrol content would seem to be the more informative value. Additionally, this is the first report found concerning the presence of other hydroxylated stilbene compounds in wines. Similar in structure, these compounds would normally be expected to complement the effects of resveratrol. Monohydroxystilbene, for example, was demonstrated to possess high antimicrobial properties (Langcake and Pryce, 1976). In the same study, resveratrol was shown to have insignificant antimicrobial activity. Other reports, however, suggest fungitoxic properties of resveratrol (Creasy and Coffee, 1988). The four hydroxy groups on tetrahydroxystilbene should increase its antioxidant activity compared to that of resveratrol.

The linkage between resveratrol production in grape species and their disease resistance mechanism is well established (Langcake and Pryce, 1976; Langcake, 1981; Blackburn and Timmon, 1969; Jeandet et al., 1995c). It is quite interesting that wines made from muscadine grapes have the highest resveratrol levels. Muscadine grapes are known to be highly resistant to most bacterial and fungal diseases of grapes (Fry and Milholland, 1990) and are grown in regions of high disease pressure. In particular, they are resistant to Pierce's disease bacterium that would kill *V. vinifera* cultivars within 2–5 years of infection.

The relatively high levels of tetrahydroxystilbene in *V. vinifera* red wines, which are traditionally fermented and/or aged in wood barrels, and the fact that the only muscadine wine with a high tetrahydroxystilbene content was barrel fermented appear to indicate that barrel fermentation might influence the amount of this compound present. Our results, however, indicate that the grape berry has significant quantities of tetrahydroxystilbene. The muscadine wines used in this study were all from the same cultivar (Noble). The fact that all of the non-barrel-fermented wines have relatively lower tetrahydroxystilbene levels than the corresponding barrel-fermented wines suggests that wood barrels play a role in providing and/or stabilizing tetrahydroxystilbene in wine. Further work is needed to determine the influence of wood contact on hydroxystilbene content of wines and the potential of the non-resveratrol stilbenol compounds to reduce serum lipid levels.

ACKNOWLEDGMENT

We gratefully acknowledge the assistance of Daphne Ingram for statistical analysis of our data.

LITERATURE CITED

- Blackburn, E. V.; Timmons, C. J. The phytocyclisation of stilbene analogues. *Q. Rev.* **1969**, *23*, 484.
- Creasy, L. L.; Coffee, M. Phytoalexin production potential of grape berries. *J. Am. Soc. Hortic. Sci.* **1988**, *113*, 230.
- Dercks, W.; Creasy, L. L. The significance of stilbene phytoalexins in the *Plasmopara viticola*-grapevine interaction. *Physiol. Mol. Plant Pathol.* **1989**, *34*, 189.
- Fitzpatrick, D. F.; Hirschfield, S. L.; Coffey, R. G. Endothelium-dependent vasorelaxing activity of wine and other grape products. *J. Am. Physiol. Soc.* **1993**, H774.
- Frankel, E. N.; Waterhouse, A. L.; Kinsella, J. E. Inhibition of human LDL oxidation by resveratrol. *Lancet* **1993**, *341*, 1103.
- Fry, S. M.; Milholland, R. D. Multiplication and translocation of *Xylella fastidiosa* in petioles and stems of grapevine resistant, tolerant, and susceptible to Pierce's disease. *Phytopathology* **1990**, *80*, 61.
- Goldberg, D. M.; Karumanchiri, A.; Eng, E.; Diamandis, E. P.; Yan, J.; Soleas, J.; Waterhouse, A. L. Resveratrol content of wines assayed by a direct gas chromatographic-mass spectrometric technique. *Am. J. Enol. Vitic.* **1993**, *44*, 344.
- Goldberg, D. M.; Yan, J.; Ng, E.; Diamandis, E. P.; Karumanchiri, A.; Soleas, G.; Waterhouse, A. L. Direct injection gas chromatographic mass spectrometric assay for trans-resveratrol. *Anal. Chem.* **1994**, *66*, 3959.
- Goldberg, D. M.; Karumanchiri, A.; Ng, E.; Yan, J.; Diamandis, E. P.; Soleas, G. J. Direct gas chromatographic-mass spectrometric method to assay *cis* resveratrol in wines. Preliminary survey of its concentration in commercial wines. *J. Agric. Food Chem.* **1995a**, *43*, 1245.
- Goldberg, D. M.; Yan, J.; Ng, E.; Diamandis, E. P.; Karumanchiri, A.; Soleas, G.; Waterhouse, A. L. A global survey of trans resveratrol concentrations in commercial wines. *Am. J. Enol. Vitic.* **1995b**, *46*, 159.
- Jayatilake, G. S.; Jayasuriya, H.; Lee, E. S.; Koochanock, N. M.; Geaheln, R. L.; Ashendel, C. L.; McLaughlin, J. L.; Chang, C. J. Kinase inhibitors from *Polygonum cuspidatum*. *J. Nat. Prod.* **1993**, *56*, 1805.
- Jeandet, P.; Bessis, R.; Gautheron, B. The production of resveratrol (3,5,4'-trihydroxystilbene) by grape berries in different development stages. *Am. J. Enol. Vitic.* **1991**, *42*, 41.
- Jeandet, P.; Bessis, R.; Sbaghi, M.; Meunier, P. Occurrence of a resveratrol β -D-glucoside in wine: preliminary studies. *Vitis* **1994**, *33*, 183.
- Jeandet, P.; Bessis, R.; Sbaghi, M.; Meunier, P. Production of phytoalexin resveratrol by grapes as a response to Botrytis attack under natural conditions. *J. Phytopathol.* **1995a**, *143*, 135.
- Jeandet, P.; Bessis, R.; Maume, B. F.; Meunier, P.; Peyron, D.; Trollat, P. Effect of enological practices on resveratrol isomer content of wine. *J. Agric. Food Chem.* **1995b**, *43* (2), 316.
- Jeandet, P.; Bessis, R.; Sbaghi, M.; Meunier, P.; Trollat, P. Resveratrol content of wines of different ages: relationship with fungal disease pressure in the vineyard. *Am. J. Enol. Vitic.* **1995c**, *46*, 1.
- Kimura, Y.; Okuda, H.; Arichi, S. Effects of stilbenes on arachidonate metabolism in leukocytes. *Biochim. Biophys. Acta* **1985**, *834*, 275.
- Lamikanra, O. Aroma constituents of muscadine wines. *J. Food Qual.* **1987**, *10*, 57.
- Lamuela-Raventos, R. M.; Waterhouse, A. L. Occurrence of resveratrol in selected California wines by a new HPLC method. *J. Agric. Food Chem.* **1993**, *41*, 521.
- Lamuela-Raventos, R. M.; Romero-Perez, A. I.; Waterhouse, A. L.; de la Torre-Boronat, M. C. Direct HPLC analysis of *cis*- and *trans*-resveratrol and piceid isomers in Spanish red *Vitis vinifera* wines. *J. Agric. Food Chem.* **1995**, *43*, 281.
- Langcake, P. Disease resistance of *Vitis* spp. and the production of the stress metabolites resveratrol, epsilon-viniferin, alpha-viniferin and pterostilbene. *Physiol. Plant Pathol.* **1981**, *18*, 213.

- Langcake, P.; Pryce, R. J. The production of resveratrol by *Vitis vinifera* and other members of the Vitaceae as a response to infection or injury. *Physiol. Plant Pathol.* **1976**, *9*, 77.
- Langcake, P.; Pryce, R. J. A new class of phytoalexins from grapevine. *Experientia* **1977**, *33*, 151.
- Lins, A. P.; Felicio, J. D.; Braggio, M. M.; Roque, L. C. A resveratrol dimer from *Parthenocissus tricuspidata*. *Phytochemistry* **1991**, *30*, 3144.
- Mattivi, F. Solid phase extraction of *trans*-resveratrol from wines for HPLC analysis. *Z. Lebensm. Unters. Forsch.* **1993**, *196*, 522.
- Peret, R.; Pont, V.; Cuenat, P. Method to determine resveratrol and pterostilbene in grape berries and wines using HPLC and highly sensitive fluorimetric detection. *J. Chromatogr.* **1994**, *663*, 191.
- Pool, R. M.; Creasy, L. L.; Frackelton, A. S. Resveratrol and the viniferins, their application to screening for disease resistance in grape breeding programs. *Vitis* **1981**, *20*, 136.
- Siemann, E. H.; Creasy, L. L. Concentration of the phytoalexin resveratrol in wine. *Am. J. Enol. Vitic.* **1992**, *43*, 49.
- Soleas, G. J.; Goldberg, D. M.; Diamandis, E. P.; Karumanchiri, A.; Ng, E.; Yan, J. A gas chromatographic mass spectrometric method for the analysis of resveratrol in juice samples. *Am. J. Enol. Vitic.* **1993**, *44*, 344.
- Waterhouse, A. L. Wine and heart disease. *Chem. Ind.* **1995**, 338.
- Waterhouse, A. L.; Lamuela-Raventos, R. M. The occurrence of piceid, a stilbene glucoside in grape berries. *Phytochemistry* **1994**, *37*, 571.

Received for review May 5, 1995. Revised manuscript received November 16, 1995. Accepted December 11, 1995.[⊗]

JF950274J

[⊗] Abstract published in *Advance ACS Abstracts*, March 15, 1996.