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BIOGENIC AMINES IN WINE : A REVIEW OF ANALYTICAL METHODS

MÉTHODES D'ANALYSE DES AMINES BIOGÈNES DANS LES VINS

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Abstract : The content of biogenic amines in wines and other fermented beverages has been extensively studied over the last 25 years because of the physiological effects which their consumption might lead to, in particular in the presence of alcohol. From the health point of view, this may indicate that the hygienic conditions while the beverage in question was being produced were deficient. Thanks to the rapid evolution of analytical techniques, there is now a better knowledge of the origin and the content of biogenic amines in wines. At present, data about concentrations are sufficiently reliable for rules to be established about the maximum recommendable limits in each country, although as yet there are no legal, unified regulations. The most widely used analytical methods for determining biogenic amines nowadays are based on chromatographic separation procedures and, in particular, on High Performance Liquid Chromatography (HPLC). This study reviews research which has compiled data about the presence of biogenic amines in wines and about the analytical techniques used to determine them, putting special emphasis on HPLC methods and the sampling treatments necessary to carry out the analyses.

Résumé : La teneur des amines biogènes dans les vins et dans les autres boissons fermentées a été largement étudiée ces 25 dernières années en raison des effets physiologiques entraînés par leur consommation et, en particulier, en présence d'alcool. D'un point de vue santé, cela peut révéler que les conditions d'hygiène dans lesquelles la boisson en question a été élaborée étaient insuffisantes. En raison de l'évolution rapide des techniques analytiques, on connaît maintenant mieux l'origine et la teneur en amines biogènes dans les vins. Les données sur les concentrations sont actuellement suffisamment fiables pour que des règlements sur les limites maximum recommandées, soient établis dans chaque pays. Malgré cela, il n'existe pas encore de règlements légaux et unifiés. De nos jours, la méthode analytique la plus largement utilisée pour la détermination des amines biogènes est basée sur les procédures de séparation chromatographique et, en particulier, sur la chromatographie en phase liquide à haute performance (HPLC). Ce travail fait le bilan des recherches qui ont rassemblées des données sur la présence des amines biogènes dans les vins et sur les techniques analytiques utilisées pour les déterminer, en mettant l'accent en particulier sur les méthodes par chromatographie en phase liquide à haute performance (HPLC) et les traitements des échantillons necessaires pour mener les analyses.

Mots clés : amines biogènes, vin, méthode analytique
Key words : biogenic amines, wine, analytical method

INTRODUCTION

There are a great deal of substances which are physiologically active in the human metabolism and which are incorporated into it by the ingestion of food of an animal or vegetable origin. These substances usually have a positive effect on the physiological mechanism of organisms, although if the quantity ingested is quite high, they may have a harmful effect, above all on the nervous system.

The content of biogenic amines in food may be related to the quality of the raw material and to the technological process to which it has been subjected (KARMAS, 1981 ; BERTRAND and DELAS, 1992 ; KRIEGER, 1993). In most cases, the content shows how well conserved the food is. The content of bioge-

nic amines in food of animal origin is not very significative (HENRY, 1960). Most of the biogenic amines which have any scientific interest are present in food of a vegetal origin.

Biogenic amines characteristically appear in fermented food. The activity of microbes on the proteins in the food, both in the ripening or fermenting process as well as in the possible process of decomposition, is the main cause of these biogenic amines. This activity initiates the sequence in which the proteolythic reactions lead to the precursor aminoacids, the decarboxylation of which results in the formation of the respective amines (VIDAL, 1987).

Of all the amines present in food, histamine is the one which has been studied most, although since the end of the 60's ethanolamine, putrescine, cadaverine, tyramine, methylamine and butylamine have also been the object of scientific studies, the main aim of which has been to determine their content in different food types such as olives (HORNERO and GARRIDO, 1994), cheese (MENTASTI et *al.*, 1991 ; MORET et BORTOLOMEAZZI, 1992), fish (TAKATSUKI and KIKUCHI, 1990 ; GIORGIO et *al.*, 1993), meat (SHALABY, 1993 ; STRAUB et *al.*, 1993 ; EEROLA et *al.*, 1993) and fermented beverages (HERRAIZ et *al.*, 1975 ; YAMAMOTO et *al.*, 1980 ; CERUTTI et *al.*, 1987b), among which wine and all its varieties and production stages stands out. Subsequently, phenetylamine, amylamine, ethylamine, tryptamine, propylamine and hexylamine have been incorporated as amines which are present more sporadically.

Interest in the presence of biogenic amines in food increased after 1946 when they were diagnosed as possibly being toxic. So, they have been the object of several bibliographic reviews focused on different food such as chocolate, (HURST et *al.*, 1982 ; JOOSTEN and OLIEMAN, 1986), lactic products (SIEBER and LAVANCHY, 1990 ; STRATTON et *al.*, 1991), meat (SCHMITT et *al.*, 1988), seafood (GILL, 1990 ; HORNER, 1992) or food in general (LOVENBERG, 1974 ; SMITH and PALUMBO, 1981 ; INGLES et *al.*, 1985 ; HURST, 1990 ; HALASZ et *al.*, 1994). However, few of these revisions focus on the specific analysis of wine (FRÖLICH and BATTAGLIA, 1980 ; SILVA and COLAGRANDE, 1987) and it is particularly noteworthy that only histamine, and in some cases tyramine, have been worthy of any special attention.

REGULATIONS

In general, it can be said that biogenic amines have toxic effects and may affect blood pressure. CABANIS, in an excellent study published in 1985, reviewed the origin and physiological and toxicological properties of histamine. Phenetylamine and tyramine are considered to be hypertensive amines. The latter also affects the absorption into the blood of histamine (BAUZA et *al.*, 1992) which, unlike the other two, is hypotensive (YEN and CHANDRA, 1988). Putrescine and cadaverine may change into pyrrolidine and piperidine, by heat, which may react with nitrites in acidic conditions and form carcinogenic compounds such as nitropyrrolidine and nitropiperidine. As well as these effects, there is the possibility that some of the biogenic amines found in wines (serotonin among them) may be involved in alcohol addiction (SUOMALAINEN et *al.*, 1974) which can increase the effect of these amines (CHAYTOR et *al.*, 1975). Migraines have been related with the consumption of half a litre of wine with histamine concentrations higher than 5 mg/l (BATTAGLIA and FRÖLICH, 1978).

The presence of biogenic amines in wines has also been associated to bacterial contamination during the production process (BUTEAU et *al.*, 1984b) and so there is considerable interest in histamine, and other amines, as indicators of defective production from a hygienic point of view (JACOB, 1969). In this respect, it is considered that a wine produced in correct hygienic conditions should be practically amine-free (CERUTTI and REMONDI, 1972).

For these reasons, some countries have established regulations and legal requirements about the limits for biogenic amine content, mainly histamine, in several foods (table I), although there is no unified criteria about the maximum limit allowed to be sold or consumed (LEHTONEN et *al.*, 1992).

Actually, the only country to pronounce on the maximum admissible limit of biogenic amines has been U.S.A., which has set the value, for tuna, at 20 mg/100 g of fish, only taking into account histamine levels, although this value is reduced to 10 mg/100 g when there are traces of decomposition (STRATTON et *al.*, 1991). For the rest of food, only Swiss legislation sets a value for histamine concentration above which products for human consumption can be rejected, although for sea produce, Finland, Sweden and Austria have also passed restrictive laws (WHO, 1991-1993).

The lack of legislation about the content of biogenic amines which can be found in food in general and wines in particular, makes it difficult to correctly judge the imports and exports of these products (INGARGIOLA, 1992). So, Holland rejects wines which contain more than 3.5 mg/l of histamine and checks for the presence of other biogenic amines, while Switzerland accepts all those that do not have more

TABLE I

Maximum recommendable limits (MRL) of histamine in food

Country	Food	MRL	Reference
Germany	Wine	2 mg/l	(MILLIES and ZIMLICH, 1988) (LEHTONEN, 1992)
	Fish	200 mg/kg	(WHO, 1991)
Switzerland	Wine	10 mg/l 4 mg/l	(OIV, 1989) (LEHTONEN, 1992)
	Fish	10 mg/100 g	(VIDAL, 1987)
Belgium	Wine	5-6 mg/l	(MILLIES and ZIMLICH, 1988) (LEHTONEN, 1992)
France	Wine	8 mg/l	(MILLIES and ZIMLICH, 1988) (LEHTONEN, 1992)
USA	Tuna Food	10 mg/100 g 50 mg/100 g	STRATTON et al., 1991 (WILLS et *al.*, 1987)
Sweden	Fish	200 mg/kg	(WHO, 1992)
Finland	Fish Wine	10 mg/100 g 5 mg/l	(VIDAL, 1987) (GARCIA et *al.*, 1994)
Czechoslovakia	Fish	10 mg/100 g	(VIDAL, 1987)
Canada	Fish	10 mg/100 g	(VIDAL, 1987)
Denmark	Tuna and bonito Fish	20 mg/100 g 200 mg/kg	(VIDAL, 1987) (WHO, 1993)
Austria	Wine	10 mg/l	(GARCIA et *al.*, 1994)
Holland	Wine	3.5 mg/l	(OIV, 1989, 1991a)
EC	Fish	200 mg/kg	(GIORGIO et *al.*, 1993)

than 10 mg/l of histamine and gives no importance to other biogenic amines which they regard as being minorities (OIV, 1989, 1991a).

AMINES IN WINES

Interest in the presence of biogenic amines in wines and their possible effect on quality was made quite clear by the preferential treatment which the subject was given by the Agreement Sub-Commission for the Unification of Analysis Methods and Appreciation of Wines of the Office International de la Vigne et du Vin (OIV). In particular, the histamine content of wines was discussed in length between 1983 and 1985 (OIV, 1983-1985) and, since 1989, the subject has been dealt with in all the annual meetings (OIV, 1989-1994).

There is great controversy about the origin of amines in wines largely because they seem to appear at random. In many studies carried out amines are formed in some wines, while in others of similar characteristics they do not appear. Obviously, there is a factor which is not exactly known or certain conditions which cause them to appear.

There is not much information available about the presence of amines in musts, although it is known that concentrations of histamine are rarely above 10 mg/l (LAFON-LAFOURCADE, 1975 ; DESSER et *al.*, 1981 ; AERNY, 1985, 1990) and that there are traces of others, such as tyramine and ethanolamine (BUTEAU et *al.*, 1984b). These concentration levels are very low when compared to the ones found in wines so we can conclude that amines are formed in the fermenting stages.

During alcoholic fermentation, the only amine which is formed in substantial concentrations is ethanolamine, which may reach values of up to 20 mg/l by the end, together with small quantities of histamine, tyramine, cadaverine and putrescine (DESSER et *al.*, 1981 ; BUTEAU et *al.*, 1984b ; AERNY, 1990).

TABLE II

Content of biogenic amines (mg/l) in red wines

Country	MET	HIS	IPR	TIR	ETA	CAD	PUT	FEN	ETY	IAM	Reference
France		2.2									LAFON-LAFOURCADE, 1975
		6.5									SCHNEYDER, 1973
		1.1		5.4		2.3	3.6	1.0			WOIDICH et *al.*, 1980
	0.1		0.2		2.1		1.7	0.4	0.4	0.1	LEHTONEN, 1986
	0.1	3.6		3.2		0.2	10.8	0.9	0.9		TRICARD and CABAZEIL, 1990
	0.1	1.2		0.7		0.3	4.8	0.2	0.5	0.2	TRICARD and *al.*, 1991
		0.4		1.0	0.4		0.4		1.2	2.5	PUPUTTI and SUOMALAINEN, 1969
		8.1		7.3		1.0	7.6				ZEE et *al.*, 1983
Switzerland		5	2		5		1			1	MAYER and PAUSE, 1984a
		28		8	17	0	9	8		3	WALTHER et *al.*, 1987
Japan	0.2	1.9		1.9		0.2	4.8	1.3	0.8	2.0	IBE et *al.*, 1991
Austria		3.3		1.7		0.7	7.6	0.9			WOIDICH et *al.*, 1980
		3.1		1.5		0.8	7.3	1.4			PFANNHAUSER and PECHANEK, 1984
Hungary		4.9		0.1		1.3	16.2	0.1			WOIDICH et *al.*, 1980
	0.2		0.2		5.4		2.2	2.2	1.2	0.7	LEHTONEN, 1986
		6.6									MACK, 1973
Italy		0.1		0.1		0.1	0.9	0.1			WOIDICH et *al.*, 1980
	0.2	2.2		3.2		0.2	6.4	1.0	1.3		TRICARD and CABAZEIL, 1990
	0.1		0.3		4.1		1.0	0.4	1.1	0.1	LEHTONEN, 1986
		0.3									MACK, 1973
		4.1		4.0		0.5	3.1				ZEE et *al.*, 1983
Spain		5.6									TEJEDOR and MARINÉ, 1979
	< 0.4	4.7		3.4		0.5	7.8	0.6	1.2		IÑIGUEZ and VAZQUEZ, 1994
	0.3	5.0		6.3		0.2	16.6	0.9	2.0		TRICARD and CABAZEIL, 1990
	0.1	0.8	0.3		6.3	0.2	13.0	0.8	1.5	5.9	LEHTONEN, 1986
		1.9		1.2							VIDAL et *al.*, 1991
		3.9	0.3	2.2			4.6	1.2	0.7	2.8	BUSTO et *al.*, 1995a
		8.9		4.2							VIDAL et *al.*, 1991
	0.1	1.1			1.9	0.3	9.0		0.9		BUSTO et *al.*, 1994
		7.6		5.2							VIDAL et *al.*, 1991
				2.4							RIVAS et *al.*, 1982
	0.7	5.1	ND	ND	2.1	ND	3.8	ND	0.7	0.2	BUSTO et *al.*, 1996a (*)
	0.8	6.3		ND		0.6	4.4	0.6	0.5	0.1	BUSTO et *al.*, 1996b (*)
Portugal		1.2					0.9				ZEE et *al.*, 1983
Yugoslavia	0.1	1.1	0.3		2.5		3.0	0.8	0.9	4.6	LEHTONEN, 1986
USA		15.1		6.3		1.1	35.8	1.1		1.0	LEHTONEN et *al.*, 1992
		5.6		ND		21.8	1.1				BAUCOM et *al.*, 1986
		3.3		ND		19.5	2.6				BAUCOM et *al.*, 1986
		2.0		ND		17.5	2.6				BAUCOM et *al.*, 1986
		1.0									OUGH, 1971
		7.3		8.6		0.8	5.5				ZEE et *al.*, 1983
Canada		3.7		4.3		0.3	2.2				ZEE et *al.*, 1983
Germany		2.1									MACK, 1973
Algeria		6.6		8.2		0.2	16.0			0.1	LEHTONEN et *al.*, 1992
Australia		1.4		2.4		0.1	6.7			0.1	LEHTONEN et *al.*, 1992
North Africa		1.0									MACK, 1973
Bulgaria		2.9		5.5		0.1	6.6	2.6		4.7	LEHTONEN et *al.*, 1992

MET: methylamine - HIS : histamine - IPR: isopropylamine - TIR: tyramine - ETA: ethanolamine - CAD: cadaverine - PUT: putrescine
FEN: phenethylamine - ETY: ethylamine - IAM: isoamylamine

ND = not detected; (*) Median

Studies carried out using different yeasts have reached the conclusion that during alcoholic fermentation, histamine is not formed in any appreciable quantity (LAFON-LAFOURCADE, 1975), not even in musts which are rich in histidine, its natural precursor. Therefore, it can be concluded that this does not appear to be the origin of most of the amines found in wines.

In general, it is accepted that histamine is produced mainly by bacterial activity during malolactic fermentation (AERNY, 1990 ; COLAGRANDE, 1992 ; KRIEGER, 1993 ; FAETH and RADLER, 1994), although how it is formed and the conditions in which it is generated have yet to be fully clarified. This point has been particularly controversial and various authors have pointed out that the concentration of histamine during malolactic fermentation actually decreases (LAFON-LAFOURCADE, 1975 ; BUTEAU et *al.*, 1984b). Bacteria of the species *Pediococcus cerevisae* seem to be mainly responsible for the formation of histamine (AERNY, 1990). Unlike the species *Leuconostoc oenos*, these bacteria can develop at pH over 3.3 and in the presence of sulphur dioxide, so at the end of the fermentation they may decarboxylate the aminoacids.

The pH and the content of sulphur dioxide only affect the bacterial flora which will guide the malolactic fermentation. So, to minimise the formation of histamine and other amines it is recommended (AERNY, 1990) that wines of pH below 3.3 should be obtained before malolactic fermentation and that sulphur dioxide should only be added in quantities lower than 50 mg/l of must and with not at all after alcoholic fermentation. So, malolactic fermentation will be driven by *Leuconostoc oenos* and histamine and the other amines will only be minimally formed.

Red wines have higher concentrations of amines than white wines (SCHNEYDER, 1973 ; CILLIERS and VAN WYK, 1985). This may be due to the greater development of bacteria during malolactic fermentation (AERNY, 1985), which may give rise to greater decarboxylasic activity and more biogenic amines. Another cause may be the use of bentonite, which is normally used in the clarification of white wines and which at doses of over 50 g/hl decreases the content of biogenic amines. Finally, it has been proposed that yeast, the extracts of which are rich in nitogenated compounds, should be autolysed as a source of amines during the vinification of red wines since these are not normally clarified before malolactic fermentation.

Histamine was the first biogenic amine to be found in wine by TARANTOLA in 1954 (CABANIS, 1985). Amine content in wines was first systematically determined mainly between the beginning of the 60s (MARQUARDT et *al.*, 1963) and the beginning of the 70's (PUPUTTI and SUOMALAINEN, 1969 ; OUGH, 1971 ; ZAPPAVIGNA and CERUTTI, 1973). The studies carried out by MAYER (1974) on the content of histamine in Swiss wines and by CILLIERS and VAN WYK (1985) on the content of histamine and tyramine in South African wines are of special interest. Subsequently, these studies were extended to research other biogenic amines. Nowadays, the presence of biogenic amines in wine is beyond question, so much so that it is mentioned in many general reviews about the constituents of wine (SCHREIER, 1979 ; NYKÄNEN and SUOMALAINEN, 1983 ; MONTEDORO and BERTUCCIOLI, 1986 ; OUGH and AMERINE, 1988).

Tables II and III summarize the data about the principal biogenic amine average contents in white and red wines from different countries. Those references (ZEE et *al.*, 1983 ; LEHTONEN et *al.*, 1992) which indicate the concentration ranges of some of the amines are not included. As well as the amines included in the previous tables, the following have also been detected, although rarely and only in low concentrations: agmatine, spermine, spermidine, octapamine, dimethylamine, propylamine, pyrrolidine, diethylamine, amylamine, butylamine, morpholine, tryptamine, iso-butylamine and hexylamine. The studies published by FRÖLICH and BATTAGLIA (1980), PFANNHAUSER and PECHANEK (1984) and LEHTONEN (1986) are worthy of special mention because they include data about more than twenty biogenic amines in wines.

The study of biogenic amines in Spanish wines also began in the 70's with research into histamine (MARTIN, 1975 ; TEJEDOR and MARINÉ, 1978, 1979 ; BURDASPAL et *al.*, 1979 ; IÑIGO and BRAVO, 1980 ; BRAVO et *al.*, 1983; VIDAL and MARINÉ, 1985 ; CALULL et *al.*, 1991) which then was subsequently extended to other amines among which was tyramine (RIVAS et *al.*, 1982 ; RIVAS et *al.*, 1983 ; VIDAL et *al.*, 1990, 1991). More recently, the content of biogenic amines has been studied more generally and all those that are regarded as being of major (GARCIA et *al.*, 1994 ; BUSTO et *al.*, 1994 ; IÑIGUEZ and VAZQUEZ, 1994) and minor presence (BUSTO et *al.*, 1994-1995, 1996a) have been included.

ANALYSIS OF BIOGENIC AMINES IN WINES

The two most pressing problems faced by the qua-

TABLE III

Content of biogenic amines (mg/l) in white wines

Country	MET	HIS	IPR	TIR	ETA	CAD	PUT	FEN	ETY	IAM	Reference
France		0.8									LAFON-LAFOURCADE, 1975
		4.9									SCHNEYDER, 1973
		0.1		0.1	0.1				0.5	1.0	PUPUTTI and SUOMALAINEN, 1969
				0.8		0.2	1.8	1.1		1.0	LEHTONEN et *al.*, 1992
	0.1		0.5		2.8		0.4	0.3	0.4	1.0	LEHTONEN, 1986
		4.4		6.5		1.4	2.3				ZEE et *al.*, 1983
Japan	0.1	1.1		1.0		0.1	1.2	2.3	0.3	5.1	IBE et *al.*, 1991
Australia		ND		0.1		0	0.1	0.1			INGLES et *al.*, 1985
Austria		0.5		0.5		0.8	0.9	0.7			WOIDICH et *al.*, 1980
		0.5		0.5		0.8	0.9	0.7			PFANNHAUSER and PECHANEK, 1984
Italy					6.1						MARGUERI et *al.*, 1983
				0.7		0.1	1.8	0.2		0.2	LEHTONEN et *al.*, 1992
		0.9		1.4		0.1	1.0				ZEE et *al.*, 1983
	0.1		0.2		2.1		0.6	0.2	0.4	0.8	LEHTONEN, 1986
Switzerland		12	1.0	2.0	20			13.0	0.5	20.0	MAYER and PAUSE, 1984a
				3.0		0.5	4.8	0.5			WALTHER et *al.*, 1987
Canada		1.9					1.2				ZEE et *al.*, 1983
Portugal		1.1		4.4		1.4	2.4				ZEE et *al.*, 1983
Spain		2.6		4.7							VIDAL et *al.*, 1991
		2.1									TEJEDOR and MARINÉ, 1979
		2.1		1.3			1.9				ZEE et *al.*, 1983
	0.5	0.5			0.7		0.6		0.1		BUSTO et *al.*, 1994
	0.5	1.2	0.1	ND	0.1	ND	3.0	ND	ND	0.5	BUSTO et *al.*, 1996a (*)
	0.8	0.3		<0.4		<0.2	2.0	0.8	0.8		IÑIGUEZ and VAZQUEZ, 1994
				2.5							RIVAS et *al.*, 1982
USA		3.5		0.1		37.3	2.7				BAUCOM et *al.*, 1986
		1.1		0.3		17.6	2.4				BAUCOM et *al.*, 1986
		2.4		0.1		29.4	2.7				BAUCOM et *al.*, 1986
		3.6		3.2			1.7				ZEE et *al.*, 1983
Germany	0.1				3.2		0.4	1.2	0.3	6.3	LEHTONEN, 1986
				2.8			2.0	2.7		3.7	LEHTONEN et *al.*, 1992
		3.7		5.9		1.3	1.3				ZEE et *al.*, 1983
Hungary	0.1		0.2		2.5		1.8	0.7	0.1	3.6	LEHTONEN, 1986

MET: methylamine - HIS : histamine - IPR: isopropylamine - TIR: tyramine - ETA: ethanolamine - CAD: cadaverine - PUT: putrescine - FEN: phenethylamine - ETY: ethylamine - IAM: isoamylamine

ND = not detected; (*) Median

lity control of food in general, and of wine in particular, are usually the complexity of the sample matrix and the low concentration levels which have to be determined. Both variables mean that new systems are continually being developed with the aim of achieving greater accuracy, sensitivity and specificity of the analytical methods.

There are a great deal of methods developed for determining biogenic amines in food. Although at first, as has been mentioned above, specific methods for analysing a single amine (normally histamine or tyramine) were developed, as both extractive and chromatographic separation techniques have been perfected, the number of analytical methods which can simultaneously determine several amines has gradually increased. At present HPLC is the most used technique because of its great versatility, and is in fact the technique used in the reference methods of official analysis laboratories (FEIER and GOETSCH, 1993).

Table IV summarizes the methods used to determine biogenic amines in wines which use techniques other than HPLC. Initially, the most usual methods for

these determinations used techniques such as spectrofluorimetry (SANTOS et *al.*, 1985 ; VIDAL, 1987 ; VIDAL et *al.*, 1989) which give the total value of the biogenic amine content in the sample to be analyzed and are often preceded by a derivatization method.

More recently, thin-layer chromatography (CERUTTI et *al.*, 1987a), column ionic chromatography (INGLES et *al.*, 1985), gas chromatography and basically HPLC have enabled biogenic amines to be determined individually working with different eluents and mobile phases.

Capillary electrophoresis (MA et *al.*, 1992) and mass spectrometry (BURLINGAME et *al.*, 1982) have also been used in the analysis of biogenic amines, although they have not been specifically applied to wine.

Practically all the analytical techniques mentioned have an associated pre- or post- chromatographic treatment to improve the selectivity and the sensitivity of the different methods. However, it is not always necessary to resort to these treatments because the sample can be directly injected as long as there is a suitable detection system, both in GC and in HPLC (SEILER, 1977).

Most of the analytical methods of biogenic amines are based on derivatization reactions which improve the two quality parameters mentioned. These reactions can be carried out either pre- or post- column, which

TABLE IV

Generally applicable analytical methods of biogenic amines in wines

Technique	N	Derivatizing reagent	Detection	Clean-up	Reference
Spectrometry	1	o-phthalaldehyde	FL	IEC*	LAFON-LAFOURCADE, 1974
	1	o-phthalaldehyde	FL	IEC*	LAFON-LAFOURCADE, 1975
	1	o-phthalaldehyde	FL	IEC*	PLUMAS and SAUTIER, 1973
	1	o-phthalaldehyde	FL	IEC*	TEJEDOR and MARINÉ, 1979
	1	o-phthalaldehyde	FL	LLE*	VIDAL et *al.*, 1989-1991
	1	o-phthalaldehyde	UV	E*	MAYER and PAUSE, 1973a
	5	Ninhydrin	Visible	IEC*	INGLES et *al.*, 1985
	10	Ninhydrin	Visible	IEC*	SAYEM-el DAHER et *al.*, 1983
	6	Ninhydrin	Visible	IEC*	WOIDICH et *al.*, 1980
	1	α-nitroso-β-naphthol	FL	LLE*	VIDAL et *al.*, 1990-1991
	1	α-nitroso-β-naphthol	FL	LLE*	RIVAS et *al.*, 1979, 1982
	1	α-nitroso-β-naphthol	FL	LLE*	ULLA et *al.*, 1989
Thin Layer	5	—	—		CERUTTI et *al.*, 1987a
Chromatography	2	Ninhydrin/Pauly	—	LLE* - IEC*	ZAPPAVIGNA et *al.*, 1974
	4	Ninhydrin	—	—	PUPUTTI and SUOMALAINEN, 1969
	9	Ninhydrin	Visible	LLE*	MAYER and PAUSE, 1973b
	5	Ninhydrin	Visible	IEC*	CERUTTI et *al.*, 1986 VECCHIO et *al.*, 1989
	1	Ninhydrin	Visible	LLE* - IEC*	CERUTTI, 1975
	19	Dansyl chloride	MS	LLE* / IEC*	ADDEO and MALORNI, 1974
	1	α-nitroso-β-naphthol	FL	LLE*	RIVAS et *al.*, 1979, 1982
	1	o-phthalaldehyde	FL	IEC*	TEJEDOR and MARINÉ, 1979
Gas Chromatography	10	Trifluoroacetic anhydride	NPD	DST* - LLE **	DAUDT and OUGH, 1980
	10	Trifluoroacetic anhydride	MS	DST* - LLE **	OUGH et *al.*, 1981
	2	Trifluoroacetic anhydride	MS	DST* / LLE* LLE **	ALMY et *al.*, 1983
	8	Trifluoroacetic anhydride	MS	DST* - LLE **	OUGH and DAUDT, 1981
	4	—	FID	DST - LLE -IEC	PUPUTTI and SUOMALAINEN, 1969
Mass Spectrometry	8	Dansyl chloride		LLE **	WALTHER et *al.*, 1987

Detection : FL : fluorescence - UV : ultraviolet - MS : Mass Spectrometry - FID : flame ionization detector - NPD : nitrogen/phosphorus detector
Clean-up : IEC : ion exchange chromatography - LLE : liquid-liquid extraction - DST : distillation - E : electrophoresis
(*) pre-derivatization - (**) post-derivatization
N : number of biogenic amines analysed

often conditions the concentration techniques which are applied during the analysis of minority amines. So, trifluoracetic anhydride (TFA) is normally used in gas chromatography, while dansyl chloride (DnsCl) or o-phthaldialdehyde (OPA) are normally used in liquid chromatography, the latter being particularly sensitive and quick at reacting, despite the limited stability of its derivatives.

Other occasionally used derivatizing reagents are phenylisothiocyanate (PITC) (CALULL et *al.*, 1991) and 9-fluorenylmethoxycarbonyl chloride (FMOC) (PFEIFFER and RADLER, 1992), both utilised in liquid chromatography. More recently, 6-aminoquinolinol-n-hydroxysuccinimidyl-carbamate (AQC) (BUSTO et *al.*, 1996a-b), commercialized as AccQ.TagTM, has been used and its high sensitivity gives more stable derivatives than the ones from the reaction with the traditional OPA.

ANALYZING BIOGENIC AMINES IN WINES USING HPLC

Biogenic amines are normally analysed by ionic chromatography or by reverse phase chromatography. In the first case, the ionic character of the amines at an acidic pH is used. In the second, the amines are normally derivatized previously, although the chromatographic separation can also be carried out using reagents which form ionic pairs with the amines, followed by a post-column derivatization.

Prior to the chromatographic separation, it is usual to clean up the samples to eliminate any interference. This clean-up may also be used to simultaneously concentrate the amines and by so doing there are considerable improvements in the sensitivity of the methods applied. Liquid-liquid and, more recently, solid-phase extraction have been the most used techniques in this respect and are applied to the sample itself as well as its derivatization products. If the aim is to determine contents of principal amines in wine as is the case of histamine, tyramine, putrescine, cadaverine and/or phenetylamine, a filtration prior to sample injection is usually sufficient. If contents of minority amines are to be determined or if the detection technique is not sensitive enough, there will have to be processes of sample extraction/concentration prior to the analysis.

Table V summarizes the different methods found in the bibliography of analysing biogenic amines using HPLC and gives the derivatizing reagents, the chromatographic conditions and the detection systems used.

In general, the limits of detection (LOD) of the HPLC techniques are not significantly different from the ones obtained using the techniques in table IV, the lowest values being about 5 µg/l (PEREIRA and BERTRAND, 1994) and the highest about 500 µg/l (AERNY, 1982 ; IÑIGUEZ and VAZQUEZ, 1994). However, in practically all studies the LOD for the techniques used are calculated from biogenic amine chromatographic standards and do not take into account the effect of the matrix which normally increases these LOD (BUSTO et *al.*, 1995b). It is difficult to find a solution to the problem of determining LOD in chromatography, especially if we take into account that it is not easy to obtain an analyte-free wine with the same characteristics as the working matrix in biological samples in which these analytes are natural components. PEREIRA and BERTRAND (1994) solve this problem by determining the LOD of each amine with a synthetic wine solution which has a similar matrix but which, of course, does not have its specific characteristics.

I — CLEAN-UP SYSTEMS

As well as filtering the sample, the most usual clean-up techniques are liquid-liquid extraction (LLE) and solid-phase extraction (SPE).

As far as LLE techniques are concerned, the most widely used solvents are butanol (PAUSE and MAYER, 1972 ; MAYER and PAUSE, 1973b ; CERUTTI, 1975 ; LEHTONEN, 1986), ethyl acetate (BUTEAU et *al.*, 1981 ; RIVAS et *al.*, 1982 ; BAUCOM et *al.*, 1986), ethyl ether (PAUSE and MAYER,1972 ; MAYER and PAUSE, 1973b ; OUGH and DAUDT, 1981 ; OUGH et *al.*, 1981 ; VECCHIO et *al.*, 1989) and dichloromethane (LEHTONEN, 1986), which, although they have not always preceded an HPLC, could be used just the same if the extract is suitably adapted to the type of chromatography to be carried out. All these methods are characterized by a long sample treatment which uses large volumes of organic solvents and which has high, very variable percentages of amine recuperation, depending on their polarity.

SPE techniques have developed quite considerably in recent years, in particular in connection with the perfection and the specifity of the stationary phases which are used in the solid phase extraction cartridges. GAWDZIK and CZERWINSKA-BIL (1991) and JANDERA et *al.* (1994) have made a comparative study of different adsorbents for the clean-up of amines in hydrophilic samples. In wine analysis, SPE techniques are being slowly introduced, the most widely used adsorbents being the weak cationic exchangers formed by carboxylic groups (LAFON-LAFOURCADE, 1975 ; IBE et *al.*, 1991 ; ZAPPAVIGNA et *al.*, 1974; PLUMAS and SAUTIER, 1973 ; CERUTTI, 1975),

the strong cationic exchangers made up of sulphonic groups (CERUTTI, 1975 ; PFEIFFER and RADLER, 1992 ; BUSTO et *al.*, 1995a, 1996b) and octadecylsilane (C18) exchangers on silicon (SUBDEN et *al.*, 1978 ; BUSTO et *al.*, 1994, 1995b, 1996c).

The cationic exchange resins cannot simultaneously concentrate biogenic amines without considerable losses if the elution is carried out in a strong acidic medium (BUSTO et *al.*, 1995a) although this concentration is achieved if the elution is carried out at a basic pH (BUSTO et *al.*, 1996b).

The amines can also be concentrated in two other ways : by using C_{18} cartridges if previously the amines are derivatised with derivatisants such as dansyl chloride which give stable derivatives (BUSTO et *al.*, 1994) or directly, without derivatizing, by adding ionic pair forming reagents (BUSTO et *al.*, 1995b).

Normally post-derivatization is more advisable because it gives all the amines an apolar, homogeneous chemical structure which is also different from the structure of the rest of the components of the matrix. So an extractant of greater specificity can be chosen, both in LLE and in SPE. However, a post-derivatization

TABLE V

HPLC analytical methods for determining biogenic amines in wines

N	SP	Mobile phase (MP)	DR	Det	Clean-up	Reference
4	C_{18}	salicylate/phosphate	—	UV	—	GENNARO and ABRIGO, 1991
19	C_{18}	ACN-water	DnsCl (1)	UV	PVP* - SPE**	BUSTO et *al.*, 1994
1	S5W	cyclohexane-AcEt	DnsCl (1)	FL	—	AERNY, 1982
16	C_8/C_{18}	ACN- water	DnsCl (1)	FL	IEC* - LLE**	IBE et *al.*, 1991
10	C_{18}	HAc/ACN-MeOH/ACN	DnsCl (1)	FL	LLE**	BUTEAU et *al.*, 1984a
1	S5W	cyclohexane -AcEt	DnsCl (1)	FL	LLE**	BATTAGLIA and FRÖLICH, 1978
26	C_{18}	ACN- ACN/EtOH	DnsCl (1)	UV-FL	LLE* - LLE**	LEHTONEN, 1986
7	C_{18}	ACN/MeOH/HAc/ water	DnsCl (1)	UV	LLE**	VECCHIO et *al.*, 1989
8	C_{18}	phosphate -OctOH/ACN	OPA (1)	FL	—	TRICARD et *al.*, 1991
1	C_{18}	ACN/ phosphate	OPA (1)	UV	SPE*	SUBDEN et *al.*, 1978
7	C_{18}	NaAc/THF-MeOH	OPA (1)	FL	—	DROZ and TANNER, 1983
7	C_{18}	phosphate -OctOH/ACN	OPA (1)	FL	—	TRICARD and CABAZEIL, 1990
7	C_{18}	MeOH/THF/NaAc-NaAc	OPA (1)	FL	—	IÑIGUEZ and VAZQUEZ, 1994
7	#	#	OPA (1)	FL	—	INGARGIOLA and BERTRAND, 1991
10	C_{18}	HAc/ACN-MeOH/ACN	OPA (1)	FL	LLE**	BUTEAU et *al.*, 1984a
7	C_{18}	HAc/ACN-MeOH/ACN	OPA (1)	FL	LLE**	BUTEAU et *al.*, 1984b
17	C_{18}	NaAc/THF-MeOH	OPA (1)	FL	PVPP*	MAYER and PAUSE, 1984a-b
6	C_{18}	HAc-ACN	OPA (1)	FL	—	LEHTONEN et *al.*, 1992
8	C_{18}	phosphate -OctOH/ACN	OPA (1)	FL	—	PEREIRA and BERTRAND, 1994
16	C_{18}	THF/TEA-MeOH	OPA (1)	FL	SPE*	BUSTO et *al.*, 1995a
15	C_{18}	THF/NaAc-MeOH	OPA (1)	FL	SPE-IPR*	BUSTO et *al.*, 1995b
8	C_{18}	OPA/Borate- ACN	OPA (2)	FL	SPE	BUSTO et *al.*, 1996c
1	C_{18}	NaAc/ HAc - ACN	PITC (1)	UV	SPE**	CALULL et *al.*, 1991
3		Pico Tag Waters™ System	PITC (1)	FL	LLE	BAUCOM et *al.*, 1986
10	C_{18}	HAc/ACN-MeOH/ACN	FLURAM (1)	FL	—	BUTEAU et *al.*, 1984a
5	C_{18}	—	FLURAM (1)	FL	IEC*	INGLES et *al.*, 1985
16	C_{18}	THF/NaAc-MeOH	AQC (1)	FL	—	BUSTO et *al.*, 1996b
16	C_{18}	THF/NaAc-MeOH	AQC (1)	FL	SPE*	BUSTO et *al.*, 1996c
4	C_{18}	Ninhydrin/DMSO	—	UV	TCA*	JOOSTEN and OLIEMAN, 1986
1	C_8	NaAc/ACN-NaAc/ACN	FMOC (1)	FL	SPE* - LLE**	PFEIFFER and RADLER, 1992

Mobile phase : HAc : acetic acid - NaAc : sodium acetate - ACN : acetonitrile - THF : tetrahydrofuran - MeOH : methanol EtOH : ethanol - OctOH : octanol - TEA : triethanolamine - DMSO : dimetil sulfoxide - AcEt : etyl acetate - #: not indicated. The hyphens indicate binary systems

Detection (Det) : FL : fluorescence - UV : ultraviolet

Clean-up : IEC : ion exchange chromatography - LLE : liquid-liquid extraction - SPE : solid-phase extraction - IPR : ion pair reagent - PVPP : polyvinylpoly-pyrrolidone - PVP : polyvinylpyrrolidone. (1) pre-column - (2) on-column *pre-derivatization - **post-derivatization

N : number of biogenic amines analyzed - SP : stationary phase - DR : derivatizing reagent

extraction cannot be applied when the derivatives suffer from limited stability, as is the case for the derivatives from the OPA reaction.

Another factor which has to be taken into account when working with wines is the specific interference of polyphenolic substances. Polyvinylpyrrolidone (PVP) (BUSTO et *al.*, 1994) and polyvinylpolypyrrolidone (PVPP) (MAYER and PAUSE, 1984a-b) are normally used to remove these substances, although other procedures have been tried, e.g. SPE with anionic exchanger cartridges (SAX) carried out before neutralizing the amines (BUSTO et *al.*, 1995b, 1996c) or, for simple samples, SPE with octadecylsilane cartridges (PFEIFFER and RADLER, 1992 ; BUSTO et *al.*, 1995b, 1996c).

II — DERIVATIZING REAGENTS

Since the aliphatic biogenic amines do not have chromophore groups which give pronounced absorption in the UV-Vis region, derivatization techniques have to be resorted to in order to increase the sensitivity of the detection (SEILER and DEMISCH, 1978 ; KNAPP, 1979). Obtaining fluorescent derivatives is the most widely used method since it can determine low concentrations of amines with great sensitivity and selectivity.

Table VI shows the derivatization conditions and the characteristics of the most used derivatizing reagents in the determination of biogenic amines in wine, as well as the properties of the reaction product obtained, since these will condition the type of sample clean-up and the amine concentration technique to be applied.

DnsCl has the advantage of giving stable derivatives with primary and secondary amines which can be determined both by UV detection and fluorescence, although it is not very sensitive (pmol) and has the drawback that it gives complex chromatograms because it gives more than one reaction product, so interfering in the detection of the amines. It also reacts with phenols, aliphatic alcohols and some sugars (BARTOK et *al.*, 1992).

The advantage of OPA is its great sensitivity (fmol) and speed of reaction, although it only reacts with primary amines and gives some derivatives (isoindoles) of limited stability. This is the great handicap which all the researchers working with OPA (BUTEAU et *al.*, 1981 ; ALLISON et *al.*, 1984) find and there are a wide range of studies which try to find solutions to this problem. OPA is not a reagent which is intrinsically fluorescent, although the isoindoles resulting from the reaction are. Their stability depends on the amine itself, on the excess of OPA, and above all, on the nature of the nucleophile (thiol) used in the cycling reaction. One way of approaching the problem of instability of the OPA-derivatives will be, then, to study different nucleophiles (KEHR, 1993) or to vary the reaction medium once the reaction has been carried out (BUTEAU et *al.*, 1981). The most widely used nucleophile is mercaptoethanol (ME). Below, if we do not say otherwise, when we refer to the OPA derivatizing agent, we shall be talking of OPA/ME.

Even so, OPA is the most widely used derivatising reagent. The instability of the derivatives can be avoided by using automatic injectors in which the derivatisation takes place in the injection block itself (LEHTONEN et *al.*, 1992 ; PEREIRA and BERTRAND, 1994 ; BUSTO et *al.*, 1995a-b, 1996a-b) or by carrying out a post-column derivatisation, although so far it has not been applied to wines (ALLENMARK et *al.*, 1985 ; STRAUB et *al.*, 1993; IZQUIERDO et *al.*, 1993). Recently, it has been proposed that OPA could be used in an on-column derivatisation system in which the reagent is a part of the mobile phase (BUSTO et *al.*, 1996c).

The reagent AQC has been used recently in the derivatisation of aqueous samples as an alternative to OPA (NIMURA et *al.*, 1986 ; COHEN and MICHAUD, 1993 ; DIAZ et *al.*, 1996). This derivatising agent reacts with primary and secondary amines, giving stable derivatives although the excess of reagent and the reaction by-products make it difficult to detect some amines (BUSTO et *al.*, 1996a-b). Sensitivity is of the same order as that obtained with OPA (fmol).

Likewise, there is no need to resort to derivatising agents. Ionic pair chromatography can be used with chromophores as the reagents (GENNARO and ABRIGO, 1991).

III — MOBILE AND STATIONARY PHASES

As can be seen in table VI, amines are usually analysed by HPLC using reverse phase chromatography, and the stationary phase normally used is octadecylsilane (C18). As an exception, IBE et *al.* (1991) and PFEIFFER and RADLER (1992) have used a phase of similar characteristics (C8) and BATTAGLIA and FRÖLICH (1978) and AERNY (1982) have used normal phase chromatography.

The mobile phases mainly consist of buffered binary systems which combine water with different modifiers such as tetrahydrofuran and/or triethanolamine, and methanol and acetonitrile as organic eluents. The predominant mobile phase in all these studies is close to being neutral or is slightly basal, which has the drawback that it limits the average life of the silica based

TABLE VI

Derivatizing reagents usually used in the HPLC analysis of biogenic amines in wines

	PITC	FLURAM	FMOC	OPA/ME	AQC	DnsCl	Ninhydrin
Usual detection	UV	UV/FL	FL.	UV/FL	FL	UV/FL	FL
Optimum detection sensitivity	pmol	pmol	fmol	fmol	fmol	pmol	nmol
Derivative stability	good	excellent	poor	excellent	excellent	good	good
Kinetics of the reaction	average	fast	fast	fast	fast	slow	slow
Secondary amines	Yes	No	Yes	No	Yes	Yes	Yes
Single product reaction	Yes	No	No	Yes	Yes	No	Yes
Specificity	Yes	Yes	Yes	No	Yes	No	No
Usual derivatization	1	1, 2	1	1, 2, 3	1	1	1, 2
pH of reaction medium	8-9	7.0	7.7	9-11	8.2-10	9.5-10	5.5
Solubility in a medium...	aqueous	organic	organic	aqueous	organic	organic	organic
Excess reagent peak	Yes	No	Yes	No	Yes	Yes	No
Reaction temperature	r.t.	r.t.	r.t.	r.t.	r.t.	variable	variable
Cost of the reagent	average	high	average	low	high	average	low

1 : pre-column - 2 : post-column - 3 : on-column - r.t. : room temperature

columns. The use of polymer based columns partially gets round this drawback (BUSTO et *al.*, 1996c).

IV — DETECTION SYSTEMS

The most usual methods of detection in the analysis of biogenic amines are UV-Vis spectrophotometry (lapprox 250 nm) and fluorimetry. The latter method has been more widely used both in the direct detection of biogenic amines and their chemical derivatives (HURST, 1990) because of its greater specificity and sensitivity. The excitation and emission wavelengths which are most used in the detection of the dansyl derivatives are 360 and 460 nm, respectively, whereas for the OPA-derivatives the most usual ones are 350 and 445 nm.

Other detectors, such as the electrochemical (WHEATLEY and TIPTON, 1987 ; YI and BROWN, 1990 ; WASHINGTON et *al.*, 1991) and the conductometric (GENNARO and MARENGO, 1988), have been used to analyse amines in different types of aqueous samples although, up to the moment, they have not been applied to wines.

CONCLUSIONS

Of all the different analytical techniques used to determine biogenic amines in wines, HPLC has imposed itself on the others (HURST, 1990). This technique stands out because it enables a great deal of amines to be simultaneously determined quickly and relatively simply.

In general, the approach to the problem of determining biogenic amines in wines by HPLC, should be based on their concentration level. Should we be interested in determining majority amines such as histamine, putrescine and tyramine, which are normally found at levels of higher than 1 mg/l, direct determination by means of derivatisation with OPA can be used. A statistically validated procedure is the one described by PEREIRA and BERTRAND (1994), although the instability of the OPA derivatives make it absolutely necessary to use automatic injectors for the on-line derivatisation.

To analyse the minority amines, with concentrations that are usually below 1 mg/l, a previous concentration treatment is needed. SPE is, at present, the most versatile technique since it allows for pre- and post-derivatisation treatments using commercial SPE cartridges with different polarities (BUSTO et *al.*, 1994-1995, 1996b-c).

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