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Gas Chromatographic Quantification of Major Volatile Compounds and Polyols in Wine by Direct Injection

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Methanol, propanol, isobutanol, isoamyl alcohol, 2-phenylethanol, acetaldehyde, 1,1-diethoxyethane, acetoin, ethyl acetate, ethyl lactate, and ethyl succinate and the polyols 2,3-butanediol (*levo* and *meso* forms) and glycerol were quantified by direct injection of wine samples. Linear responses over the usual concentration ranges for these compounds and r^2 values from 0.9932 to 0.9998 were obtained. The confidence limits for the mean values ranged from 2.34% for diethyl succinate to 8.52% for 1,1-diethoxyethane, both at a probability level of 0.05. Relative errors ranged from 8 to 10% for the polyols and 1,1-diethoxyethane and were all less than 5% for alcohols and acetaldehyde. The proposed method is useful with a view to identifying relationships between alcoholic fermentation byproducts and controlling biological or chemical aging in wines.

KEYWORDS: Wine; analysis; volatile compounds; polyols; gas chromatography

INTRODUCTION

Gas chromatography is a widely used technique for determining volatile compounds in wines and alcoholic beverages. Such compounds are important for the classification, quality control, and sensory evaluation of wines (*1*, *2*). However, exhaustive analyses for volatiles are very complex and expensive; also, they usually require some pretreatment for isolation from the wine matrix. The pretreatment often involves liquid−liquid extraction with an organic solvent such as carbon disulfide, diethyl ether, Freon, pentane, dichloromethane, or a pentane−dichloromethane mixture. Distillation or microextraction (*3*) have also been used for this purpose. By contrast, only a few methods involving direct injection of the wine samples have so far been reported (*4*−*9*). Direct injection of the wine sample is advisable in quantifying very polar volatile compounds, which are very readily dissolved in water and ethanol−water mixtures and are scarcely extracted by organic solvents as a result. Methanol, propanol, acetaldehyde, 1,1-diethoxyethane, and acetoin are major examples in this respect (*10*−*13*), all with marked sensory and biochemical properties (*14*−*16*).

The injection of samples containing a high proportion of water into a chromatographic column results in degradation of the stationary phase, which in turn leads to low peak resolution, poor reproducibility, and shortened column lifetime. According to Blomberg (*17*), the stationary phases currently used in capillary columns should be structurally improved to increase the reproducibility of their chromatographic properties and their thermal stability. However, capillary columns made with polyethyleneglicol (PEG) or WAX as stationary phase exhibit a high chemical inertness, thermal stability, and endurance over repeated injections of aqueous samples (*18*). These columns are recommended by the OIV (*19*) for the separation of alcohols, aroma compounds, fatty acids methyl esters, and essential oils in distillates from alcoholic beverages.

The chromatographic methods commonly used for the analysis of major volatile compounds in alcoholic beverages and wines, which possess high contents of soluble solids, use a sample distillation step (*19*). Injecting the distillate rather than the sample directly into the gas chromatograph reduces contamination of the injection chamber and in the head of the column; however, it alters the equilibrium between volatile compounds in the wine matrix. In addition, nonvolatile compounds such as glycerol and 2,3-butanediol are not present in the distillate, so they cannot be quantified in this way.

There are various gas chromatographic methods for the determination of polyols in wines and fermented beverages (*2*, *4*, *8*, *20*, *21*), some of which involve direct sample injection. The most widely studied method entails the prior formation of silyl derivatives (*2*), which is rather complex and labor-intensive (*20*).

Major volatile compounds in wines and alcoholic beverages are mainly higher alcohols, which are so-called because they have more carbon atoms than ethanol, the wine alcohol. 1-Propanol, isobutanol, isoamyl alcohol, and 2-phenylethanol are those typically present at the highest concentrations (from 10 to 500 mg/L). Methanol is present in all types of wine. It comes from the enzymatic hydrolysis of the methoxyl groups of the pectins during fermentation and its content depends on the extent to which the grape solids, especially skins that have

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Table 1. Chemical Standards Used and Retention Times Obtained on a CP-WAX 57 CB Column (60 m × 0.25 mm × 0.40-μm Film Thickness)

compound	supplier	purity (%)	retention time (min)
acetaldehyde	Merck	99	8.323
ethyl acetate	Aldrich	99.5	13.250
1,1-diethoxyethane	Fluka	97	13.496
methanol	Merck	99.9	14.578
1-propanol	Riedel de Haën	99	22.412
isobutanol	Carlo Erba	99	25.346
4-methyl-2-pentanol	Merck	97	28.656
isoamyl alcohol	Carlo Erba	99	30.660
acetoin	Aldrich	99.9	34.203
ethyl lactate	Sigma	98	36.140
2,3-butanediol (*levo* and *meso* forms)	Fluka	99	43.355 (*levo*) 44.466 (*meso*)
diethyl succinate	Merck	99	47.001
2-phenylethanol	Merck	99	54.567
glycerol	Fluka	99.5	78.169

a high pectin contents, are macerated. The use of pectolytic enzymes may cause an increase in methanol as a result of the pectin esterase activity. Methanol's toxicity is well known, so its concentration must be checked. Ethyl esters such as ethyl acetate, ethyl lactate, and diethyl succinate, which result from the activity of acetic and lactic acid bacteria, are highly abundant. Some special wines such as the sherry-type wines "fino" and "amontillado" exhibit high concentrations of acetaldehyde and its derivatives 1,1-diethoxyethane and acetoin as a result of the biological aging process used in their production. Finally, all types of wine typically contain high concentrations of the polyols glycerol and 2,3-butanediol (5−11 g/L and 1 g/L, respectively).

The aim of this work is to develop and validate a simple method for quantifying many of the most important volatile compounds and polyols in wine in a single chromatographic run.

MATERIALS AND METHODS

Reagents, Samples, and Standards. *Solvents.* Absolute ethanol (analytical reagent grade, ACS-ISO quality) from Merck (Darmstadt, Germany) and water purified through a Milli-Q system (Millipore, Bedford, MA), were used to prepare the standard solutions.

Reagents. Solid anhydrous calcium carbonate was purchased in analytical grade (ACS-ISO quality) from Panreac (Barcelona, Spain).

Standards. Their sources and purity are shown in **Table 1**.

Samples. All wine samples were from Spain and included fino, amontillado, oloroso, young white wine, and young red wine, with 14.5, 17.0, 18.0, 12.0, and 12.0% v/v ethanol, respectively. All were dry-type wines with residual sugar contents below 5 g/L and pH 3.2−3.5.

Standard Solutions. Accurately weighed amounts (≈0.001 g) of the chemical standards were dissolved in water, absolute ethanol, or a 14% ethanol−water mixture. The solutions included the following:

Standard A: acetaldehyde (6.5 g), methanol (1.1 g), 1-propanol (0.8 g), isobutanol (1.0 g), isoamyl alcohol (4.2 g), 2-phenylethanol (0.8 g), and acetoin (1.6 g) in 1 L of water.

Standard B: ethyl acetate (2.2 g), ethyl lactate (5.9 g), diethyl succinate (0.6 g), and 1,1-diethoxyethane (1.1 g) in 1 L of absolute ethanol.

Standard C: glycerol (103 g) and 2,3-butanediol (30 g, 35% in levo form and 65% in meso form) in 1 L of 14% ethanol.

Synthetic Wine Samples. Standards A, B, and C were mixed and diluted with water and ethanol to the concentrations typically found in wines, the final ethanol content being adjusted to 14% v/v.

The internal standard was a solution of 4-methyl-2-pentanol (1 g/L) in 14% (v/v) ethanol. Both samples and standard solutions were stored refrigerated at 4 °C.

Gas Chromatography. An Agilent 6890 series plus gas chromatograph (Agilent Technologies, Waldbronn. Germany) with electronic pressure control was used. The column, a CPWAX-57 CB model from Chrompack (Middelburg, The Netherlands), was fused silica 60 m × 0.25 mm and 0.40-μm film thickness. The temperature program was as follows: 50 °C for 15 min and then raised to 190 °C at 4 °C/min for 35 min. The flow rate of the carrier gas (helium) was held at 0.7 mL/min for 16 min and then raised at 0.2 mL/min^2 to 1.1 mL/min for 52 min. The injector was equipped with an open tubular liner type in borosilicate glass, 4 mm of i.d., using glass wool at the center to ensure repeatability in the injection volume and no tapers for consistent split injection. A 1:30 split ratio and an injector temperature of 275 °C were used. The flame ionization detector temperature was 300 °C, and the hydrogen and air flow rates were 40 and 400 mL/min, respectively. A postrun purge program at 200 °C for 35 min and a helium flow rate of 1.3 mL/min were used after the chromatographic peaks of interest were eluted. The chemstation software package (Agilent Technologies, Waldbronn. Germany) for gas chromatograph control and detector signal treatment was used.

Proposed Method. To a 15-mL screw-capped centrifuge tube containing 10 mL of freshly prepared synthetic wine or wine sample, 1 mL of internal standard solution of 4-methyl-2 pentanol and 0.2 g of calcium carbonate were added. The tubes were then shaken for 15 s in an ultrasonic bath and centrifuged at 3500 rpm for 5 min. Once the solid-phase had been separated, the liquid phase was transferred to a 15-mL tube and a volume of 0.5 μL was injected into the gas chromatograph. The peak area relative to the internal standard for each volatile compound and polyol in the wine was calculated and interpolated into the corresponding calibration graph, which was constructed as described below.

Calibration Graphs. Synthetic standard solutions for calibration containing known concentrations of the target volatile compounds and polyols within the determination ranges listed in **Table 2** were prepared by mixing variable volumes of solutions A, B, and C and adjusting their ethanol content to 14% (v/v). A volume of 10 mL of each standard solution was subjected to the sample preparation and analysis procedures.

Method Development and Validation. Three synthetic standard solutions of known concentration were prepared separately in triplicate and injected into the gas chromatograph to check for linearity. The analysis of the data obtained for the nine standard solutions for each compound provided the linear equations shown in **Table 2**.

Repeatibility and Reproducibility. The five selected wines and the synthetic wine were analyzed in triplicate on 3 different days. Data from 54 analyses (6 wines × 3 injections × 3 days) were processed as follows: the square root of the arithmetic mean of the variance for each of the three replicates (one triplicate per day) for each wine was used to assess the repeatability of the method for each compound in each wine sample. The standard deviation obtained from the three mean values for each wine (one per day) multiplied by the square root of 3 was taken to be reproducibility value; if such value exceeded the repeatability, however, this was also taken as the reproducibility.

RESULTS AND DISCUSSION

The typical wine chromatogram obtained (**Figure 1**) reveals that the 14 analytes can be efficiently separated within 80 min. The separation of ethyl acetate and 1,1-diethoxyethane, which exhibited absolute retention times of 13.250 and 13.496 min, respectively, was also accomplished. The difference is large enough for accurate quantification of both compounds by capillary gas chromatography. Isoamyl alcohol isomers (viz., 2-methyl-1-butanol and 3-methyl-1-butanol) exhibited a difference of less than 0.1 min between their respective peaks; this precluded their individual quantification, so the two had to be quantified together. By contrast, the levo and meso forms of 2,3-butanediol had a retention time of 43.355 and 44.466 min, respectively, so the peaks were processed separately. 4-Methyl-2-pentanol, which was used as the internal standard, was eluted

Table 2. Figures of Merit of the Proposed Method[a]

compound	slope	intercept	r^2 ($n = 9$)	DR	$S_{x/y}$	QL	±CL
acetaldehyde	4.23×10^{-3}	-7×10^{-4}	0.9993	44–653	0.045	31	7.2
ethyl acetate	4.96×10^{-3}	-3×10^{-4}	0.9986	20–202	0.010	12	2.9
1,1-diethoxyethane	5.92×10^{-3}	1×10^{-2}	0.9995	8–86	0.007	7	1.7
methanol	5.04×10^{-3}	6×10^{-3}	0.9995	22–220	0.013	19	4.4
1-propanol	9.1×10^{-3}	5×10^{-3}	0.9994	18–90.0	0.022	16	3.7
isobutanol	9.5×10^{-3}	3×10^{-3}	0.9997	10–120	0.010	6	1.4
isoamyl alcohol	8.7×10^{-3}	9×10^{-3}	0.9998	42–416	0.021	14	3.3
acetoin	5.08×10^{-3}	-1×10^{-3}	0.9976	25–164	0.032	13	3.0
ethyl lactate	5.6×10^{-3}	3×10^{-2}	0.9954	32–900	0.048	24	5.7
2,3-butanediol *levo*	4.79×10^{-3}	-5×10^{-2}	0.9939	82–816	0.099	60	14
2,3-butanediol *meso*	5.18×10^{-3}	-2×10^{-2}	0.9977	144–2248	0.122	64	15
diethyl succinate	5×10^{-3}	2.8×10^{-2}	0.9935	13–130	0.018	10	5.0
2-phenylethanol	14×10^{-3}	2×10^{-3}	0.9957	20–160	0.038	16	3.8
glycerol	2.1×10^{-3}	-4.4×10^{-1}	0.9932	1493–10308	1.376	1190	279

[a] r^2 = regression coefficient. DR = determination range. $S_{x/y}$ = standard deviation of the estimate. QL = quantification limit (mg/L) = 10 σ/S. σ = standard deviation intercept of the calibration curve. S = sensitivity (slope of the calibration curve). CL = confidence limits (mg/L) for QL at a probability $p \leq 0.05$ with 8 degrees of freedom.

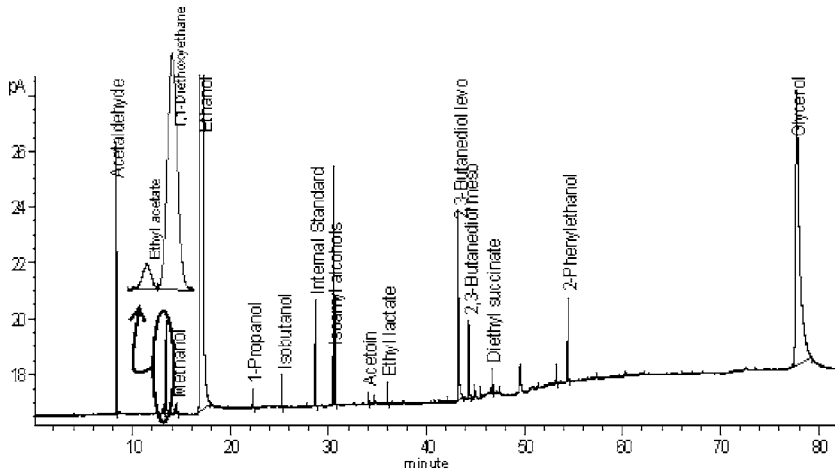

Figure 1. Chromatogram for wine sample as obtained using a CP-WAX 57CB column (60 m × 0.25 mm and 0.40-μm film thickness).

after 28.6 min (a time in the middle of the chromatogram). The other compounds studied had the retention times listed in **Table 1**.

Standard solutions of known concentration were used to establish the calibration equation for each compound. Thus, synthetic samples prepared by mixing variable volumes of solutions A, B, and C were analyzed to obtain the results of **Table 2**. The table shows the figures of merit of the proposed method. The correlation coefficient (r^2) was good in all cases and ranged from 0.9932 for glycerol to 0.9998 for isoamyl alcohols; these values are related to the proportion of the sample variance accounted by the equations which corresponded to r (%) values of 99.69 and 99.99%, respectively. The standard deviation of residuals was always less than 2 and ranged from 1.376 for glycerol to 0.007 for 1,1-diethoxyethane.

The slope of a straight calibration curve is a measure of method sensitivity and is dependent on the response of the flame ionization detector to each compound, which is related to the carbon/oxygen ratio in hydrocarbon compounds. In our case, the lowest sensitivity was that for glycerol and acetaldehyde, and the highest was that for isobutanol and 1-propanol.

The quantification limit (QL) is defined as the compound concentration giving a signal that is taken to be the lower limit of the linear range. Such a limit for each compound is calculated as the intercept plus 10 times its standard deviation. The reliability of the QL values was calculated as the confidence limit at the 95% probability level for each compound. As can be seen in **Table 2**, QL ranged from 1190 mg/L for glycerol to 6 mg/L for isobutanol, with a confidence limit of 279 mg/L and 1.4 mg/L, respectively. The average concentrations usually found in wines are higher than the respective QL values for the compounds studied.

The precision, expressed as the relative standard deviation RSD (%), and the accuracy (% error) of the method were established by determining the compounds in synthetic samples of known concentration prepared from one standard solution (**Table 3**). The results obtained for nine aliquots of the same sample had a deviation below 6% for acetoin, ethyl lactate, 2,3-butanediol (levo and meso), diethyl succinate, and all the alcohols except 2-phenylethanol (7.52%). By contrast, acetaldehyde, ethyl acetate, glycerol, and 1,1-diethoxyethane exhibited deviations from 7.03% for acetaldehyde to 11.07% for 1,1-diethoxyethane. Such relatively low deviations suggest a high precision in the quantification of methanol, higher alcohols, acetoin, 2,3-butanediol (levo and meso), and the ethyl esters of lactic and succinic acids. The relative error for each compound was calculated by using the concentrations obtained for dilute solutions of the commercial products listed in **Table 1** as reference. Acetaldehyde, methanol, propanol, isobutanol, isoamyl alcohol, acetoin, and 2-phenylethanol provided errors less than 4%, while ethyl acetate, ethyl lactate, and diethyl succinate exhibited error from 6.3 to 8.3%. Only 1,1-diethoxyethane and polyols (butanediol and glycerol) provided errors values in the region of 10%.

Table 3. Precision (as RSD %), Accuracy (Relative Error, %), and Confidence Limits of the Proposed Method over the Linear Range for Each Compound

	concentration (mg/L)				
compound	added[a]	found[b]	RSD %[c]	error %	±CL
acetaldehyde	326.5	318.5	7.03	–2.45	38
ethyl acetate	43.5	49.9	7.98	6.34	6.7
1,1-diethoxyethane	77.6	70	11.07	–9.79	13
methanol	35.8	35.2	3.69	–1.67	2.2
1-propanol	41.2	42.3	4.78	2.57	3.4
isobutanol	59.5	58	5.53	–1.46	5.4
isoamyl alcohol	208	207.8	4.27	–0.1	15
acetoin	83.5	86.4	5.61	3.47	8.1
ethyl lactate	161.5	174.9	4.95	8.3	14
2,3-butanediol *levo*	244.5	270.3	5.26	10.5	24
2,3-butanediol *meso*	448.5	492.1	4.12	9.74	34
diethyl succinate	21.1	22.6	3.04	7.36	1.2
2-phenylethanol	65.5	63.1	7.52	–3.66	8.0
glycerol	5285	5815.8	9.26	10.04	905

[a] Concentration values obtained by dilution of commercial products. [b] Mean of $n = 9$ injections. [c] RSD % = standard deviation of the mean. CL = confidence limits (mg/L) of the means at a probability $p \leq 0.001$, with 8 degrees of freedom.

Bias was negative for acetaldehyde, 1,1-diethoxyethane, methanol, isobutanol, isoamyl alcohol, and 2-phenylethanol only, so the mean concentrations found for these compounds were always lower than the reference concentrations.

Confidence limits were also calculated at a probability of 0.001 (99.9% confidence level) with 8 degrees of freedom. **Table 3** summarizes the results for diethyl succinate, methanol, propanol, isobutanol, ethyl acetate, 2-phenylethanol, and acetoin, all with low confidence limits (<10 mg/L), in contrast to others such as glycerol, which exhibited the highest value (905 mg/L). The confidence limits indicate the range where the actual concentration of each compound can be found with a probability of 99.9%, when the proposed method is repeatedly applied to the same matrix. Only the reference concentrations for 2,3-butanodiol (levo and meso forms) and diethyl succinate lay outside their respective confidence intervals; however, the errors for these compounds were 10.5, 9.74 and 7.36%, respectively.

The method proposed was used to determine volatiles and polyols in synthetic and real wine samples. **Table 4** shows the repeatability and reproducibility data for each compound studied. Repeatability is a measure of dispersion in the results of independent analyses, carried out by the same analyst using the same material in the same laboratory in a short interval of time. On the other hand, the reproducibility is related to dispersion in the results of analyses conducted by the same or different analysts using different calibration curves on different days. For practical purposes, reproducibility can be considered uncertainty in the measurements performed by a laboratory.

Repeatability data are listed in **Table 4** as the square roots, of the arithmetic mean, of the variances obtained from three replicate samples analyzed in triplicate on the same day. The reproducibility data in this table are the average standard deviations of the means obtained from the analysis of several replicate samples analyzed by different analysts on different days multiplied by the square root of 3. These data were calculated for five wines used to validate the proposed method and also for a synthetic wine; it provided an estimation of both parameters at different concentrations in different types of matrixes. Four white wines were used for this purpose, namely, young white wine and fino, amontillado, and oloroso sherry type wines, all from the Montilla-Moriles Denomination of Origin, and a young red wine from Rioja Denomination of Origin. All wines had residual sugar concentrations below 5 g/L. The production of fino wine involves a biological aging process, oloroso wine production involves a chemical aging process, and amontillado wine production involves a combination of both aging processes. Biological aging is effected by special yeasts called "flor yeasts" which form a film on the wine surface. Their influence on wine composition is exerted via the production of acetaldehyde and its derivatives and by the consumption of glycerol (*22*−*25*). Chemical aging takes place in American oak casks and is an oxidative process. The young wines studied were fresh, fruity wines obtained from recently harvested grapes and were subject to no aging process.

All compounds in the synthetic wine sample exhibited relative standard deviations less than 10% (from 0.48% for 2,3-butanediol to 9.11% for acetoin) in the repeatability study. The deviations obtained in the reproducibility study were always higher than the repeatability values for all compounds; ethyl lactate, diethyl succinate, glycerol, and acetoin had RSD values above 10%. In general, all wines and compounds exhibited repeatability values below 10%; by exception, acetoin in fino wines (31.2 mg/L) and glycerol in red wines (10514 mg/L) provided deviations of 11.86% and 10.22%, respectively, as a likely result of the nearness of their concentrations to the determination range limits used to construct their respective calibration curves. Reproducibility values exhibited higher

Table 4. Mean, Repeatability, and Reproducibility for Five Wine Samples and a Synthetic Wine[a]

	synthetic wine			fino wine			amontillado wine			Oloroso wine			young white wine			young red wine		
compound	mean	Srt %	Srd%	mean	Srt %	Srd %	mean	Srt %	Srd %	mean	Srt %	Srd %	mean	Srt %	Srd %	mean	Srt %	Srd %
acetaldehyde	558	1.02	1.95	234.9	1.40	6.26	186	1.77	2.63	95.6	4.50	7.01	90.2	6.54	6.54	67.2	6.25	7.29
ethyl acetate	71.9	1.11	4.31	66.3	5.28	12.52	266	1.32	2.71	109.5	4.47	9.32	60.9	3.61	13.46	59.7	6.53	12.06
1,1-diethoxyethane	108	0.56	3.33	9.2	4.35	18.48	29.3	1.37	1.37	10.9	8.26	13.76	ND[b]	ND	ND	ND	ND	ND
methanol	113	1.33	2.21	70.3	1.56	6.83	89.7	1.23	12.93	96.7	4.03	6.51	57.1	3.15	6.83	191	3.09	6.07
1-propanol	52	0.77	2.31	48	0.63	6.46	75.1	0.40	2.53	38.1	2.62	6.04	24.7	4.05	5.67	31	3.23	6.13
isobutanol	56.7	1.06	2.47	65.9	3.03	4.70	98.2	2.04	3.56	45.2	2.43	8.41	18.8	6.91	6.91	49.6	1.41	7.06
isoamyl alcohol	273	0.73	2.01	358	1.37	2.71	490	1.00	1.59	285	1.47	2.98	155	2.65	3.61	222	1.67	3.51
acetoin	157	9.11	19.49	31.2	11.86	13.14	62.2	5.95	12.06	38.8	2.58	12.63	56.4	3.72	19.33	54.1	6.28	13.86
ethyl lactate	188	3.14	11.70	400	6.43	9.90	895	2.87	3.40	270	4.78	10.89	99.6	3.41	21.69	168	4.05	18.10
2,3-butanediol *levo*	363	3.25	5.67	913	4.49	10.41	2530	1.62	4.17	820	7.34	10.76	806	8.91	8.00	1047	8.42	10.03
2,3-butanediol *meso*	665	0.48	6.71	273	5.27	14.62	1061	1.36	4.74	281	8.97	2.67	246	4.84	21.50	316	3.80	15.92
diethyl succinate	58.4	7.71	12.84	39	4.87	9.74	215	0.88	2.37	66.8	7.93	2.84	32.4	8.33	9.88	65	7.69	7.85
2-phenylethanol	80.5	1.37	2.48	54.3	3.13	12.34	121	1.40	3.47	45.1	7.54	10.64	17.6	4.55	6.25	31	6.77	13.55
glycerol	9467	4.90	17.03	4477	8.64	8.64	13276	9.47	9.47	8069	7.45	16.77	6693	9.47	15.36	10514	10.22	10.22

[a] Mean = mean of three wine analyses in triplicate for each wine type ($n = 9$). Srt: Repeatability (square root of arithmetic mean of the variance of triplicates in each type of wine). Srd: Reproducibility (standard deviation of the three mean values for each wine multiplied by the square root of 3). [b] Not detected.

deviations than the repeatability values in all compounds and wines. Ethyl lactate, butanediol (meso), and 2-phenylethanol exhibited the highest reproducibility deviations in the young red wine by effect of the high sugar concentration (5 g/L) and polyphenol levels in this red wine. Improving the reproducibility here entails cleaning the injector liner more frequently.

Special caution must be exercised when complex matrix solutions are to be directly injected. Thus, injection of the sample is a delicate process owing to the high variability in boiling points among the analytes. Therefore, injections should be made with great caution, always in the same way, and after checking that the septum is in good condition to avoid increased losses of the more volatile compounds. Also, although wine samples are injected after acid compounds have been removed with calcium carbonate, the accumulation of residues of nonvolatile compounds in the injection liner and in the column head can detract from reproducibility. This problem can be avoided by injecting a small sample volume (0.5 μL), changing the glass wool placed in the liner after 10 injections, and placing a precolumn without stationary phase before the analytical column. Purging the column at a temperature near its maximum limit of use is also advisable, using a postrun program for at least 30 min after the peaks of interest are eluted.

In conclusion, the proposed method allows the fast quantitative determination of major volatile compounds and polyols in dry wines. Of special interest is the quantification of various compounds highly soluble in water and ethanol−water mixtures (e.g., acetaldehyde, methanol, and propanol); these are poorly extracted by organic solvents, leading to poor gas chromatographic quantification. In addition, polyols are quantified with acceptable errors relative to other chemical methods. Its good analytical performance in terms of linearity, precision, and accuracy make the proposed method useful for routine wine quality control and classification analysis, as well as for extracting useful information about wine aging processes.

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