RESOLUTION OENO 3/2006

COLLABORATIVE STUDY - TITRIMETRIC DETERMINATION OF CARBON DIOXIDE IN SPARKLING AND SEMI-SPARKLING WINES

THE GENERAL ASSEMBLY,

CONSIDERING Article 2 paragraph 2 iv of the Agreement establishing the International Organisation of Vine and Wine,

UPON THE PROPOSAL of the Sub-commission of Methods of Analysis and Appraisal of Wine,

DECIDES to complete the method of determination of CO2 in the Compendium of international methods of analysis of wines and musts by the following results concerning the validation study and to modify the method in Type II method in the range of concentration up to 1.5 g/l

Collaborative Study - Titrimetric determination of carbon dioxide in sparkling and semi-sparkling wines - Report on Results

1. Goal of the study

The objective of the study is to determine the repeatability and reproducibility characteristics of the reference method (MA-E-AS314-01-DIOCAR) for the titrimetric CO2 determination in sparkling and semi-sparkling wine.

O.I.V. definitions and limits for the CO2 content are given with resolution OENO 1/2002.

2. Needs and purpose of the study

The reference method for the CO_2 determination includes no precision data. This collaborative trial was thus conducted.

Due to the analytical particularity, theconventional validation protocol was not able to be completely respected. Out of one bottle of sample only one independent determination could be done. Each bottle had to be considered as individual. Therefore homogeneity testing within the pre-investigations for collaborative studies was impossible. In order to provide homogenous test material close co-operation with producers was necessary. Samples were obtained during the filling of the bottles on

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the filling line in a very short time space, thus that it must be assumed that the CO_2 is homogeneously distributed in all bottles.

This study was designed to be a blind duplicate test. The complete anonymity of the samples could not be guaranteed because the partners involved used different types of bottles and/or stoppers for the different samples. Therefore we had to rely on the honesty of the participating laboratories which were requested to perform the data analysis independently without any data modification.

3. Scope and applicability

1. The method is quantitative.
2. The method is applicable for the determination of CO_2 in sparkling and semi-sparkling wines to check that standards are respected.

4. Materials and matrices

The collaborative study included 6 different samples. All were sent in blind duplicate, so that in total 12 bottles were distributed to the participants.

Table 1. Samples and coding.

Sample	Bottle Code	Type
SAMPLE A	(Code 1 + 9)	sparkling wine
SAMPLE B	(Code 2 + 5)	semi-sparkling wine (“petillant”)
SAMPLE C	(Code 3 + 4)	sparkling wine
SAMPLE D	(Code 6 + 10)	semi- sparkling wine (“petillant”)
SAMPLE E	(Code 7 + 11)	semi- sparkling wine (“petillant”)
SAMPLE F	(Code 8 + 12)	sparkling wine (red)

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5. Control measures

The method considered is already approved in practice. Only the missing precision data had to be determined within the collaborative study. A pre-trial was not required because most of the laboratories had been already using the reference method in routine analysis.

6. Method to be followed and supporting documents

Supporting documents were given to the participants (Covering letter Reference for method of analysis, Sample Receipt Form and Result Sheet).

The determination of CO_2 content in g/l should be expressed in g/l.

7. Data analysis

1. Determination of outliers was assessed by Cochran, Grubbs and paired Grubbs tests.
2. Statistical analysis was performed to obtain repeatability and reproducibility data.
3. HORRAT values were calculated.

8. Participants

13 laboratories from several different countries participated in the collaborative study. Lab-Code numbers were given to the laboratories. The participating laboratories have proven experience in the analysis of CO_2 in sparkling wine.

Table 2. List of participants.

Landesuntersuchungsamt D-56068 Koblenz GERMANY	Institut für Lebensmittelchemie und Arzneimittelprüfung D-55129 Mainz GERMANY

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Landesuntersuchungsamt D-67346 Speyer GERMANY	Institut für Lebensmittel, Arzneimittel und Tierseuchen D-10557 BERLIN GERMANY
Servicio Central de Viticultura y Enologia E-08720 Villafranca Del Pendes SPAIN	Landesuntersuchungsamt D-54295 Trier GERMANY
Landesuntersuchungsamt D-85764 Oberschleißheim GERMANY	Instituto Agrario di S. Michele I-38010 S. Michele all Adige ITALIA
Chemisches Landes- u. Staatl. Veterinäruntersuchungsamt D-48151 Münster GERMANY	Ispettorato Centrale Repressione Frodi I-31015 Conegliano (Treviso) ITALY
Bundesamt für Weinbau A-7000 Eisenstadt AUTRIA	BgVV D-14195 Berlin GERMANY
Chemisches und Veterinäruntersuchungsamt D-70736 Fellbach GERMANY	

9. Results

The uncertainty data are directly calculated for the CO_2 determination from the results submitted. For the assessment of the collaborative trial the Horrat-ratio is of relevance. For all samples a ratio of < 2 was obtained for r and R, convincing for a collaborative study. Table 3 shows the results of the CO_2 titration for each sample.

Table 3. Summarised results of the CO_2 determination.

CO2	SAMPLE A	SAMPLE B	SAMPLE C	SAMPLE D	SAMPLE E	SAMPLE F

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Mean [g/l]	9.401	3.344	9.328	4.382	4.645	8.642
r [g/l]	0.626	0.180	0.560	0.407	0.365	0.327
sr [g/l]	0.224	0.064	0.200	0.145	0.130	0.117
RSDr %	2.379	1.921	2.145	3.314	2.803	1.352
Hor	0.893	0.617	0.804	1.109	0.946	0.501
R [g/l]	1.323	0.588	0.768	0.888	0.999	0.718
sR [g/l]	0.473	0.210	0.274	0.317	0.357	0.256
RSDR %	5.028	6.276	2.942	7.239	7.680	2.967
HoR	1.245	1.331	0.728	1.599	1.711	0.726

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