RESOLUTION OENO 16/2002

DETERMINATION OF D-MALIC ACID IN WINES AT LOW CONCENTRATIONS

THE GENERAL ASSEMBLY,

HAVING CONSIDERED Article 5 of the October 13, 1954 International Convention on Unification of the Methods of Analysis and Appraisal of Wines,

WITH THE PROPOSAL of the Sub Commission on Methods of Analysis and Appraisal of Wines,

DECIDES to complement the determination of malic acid D(-) in Appendix A of the Compendium of International Methods of Analysis of Wines and Musts by the following method:

DETERMINATION OF D-MALIC ACID IN WINES AT LOW CONCENTRATIONS USING THE ENZYMATIC METHOD

1. FIELD OF APPLICATION

The method described is applied to dosage, by the enzymatic means, of malic acid D of wines with contents under 50 mg/l.

2. PRINCIPLE

The principle of the method is based on malic acid D(+) oxidation (D-malate) by nicotinamide-adenine-dinucleotide (NAD) in oxaloacetate that is transformed into pyruvate and carbon dioxide; the formation of NADH, measured by the increase of absorbance in wave length at 340 nm, is proportional to the quantity of D-malate present (principle of the method described for malic acid D determination for concentrations above 50 mg/l), after introducing a quantity of malic acid D of 50 mg/l in a cuvette.

3. REAGENTS

Malic acid D solution of 0.199 g/l, above reagents indicated in the methods described for contents above 50 mg/l.

Certified in conformity Bratislava, 28th June 2002
The Director General of the OIV
Secretary of the General Assembly
Georges DUTRUC-ROSSET

4. APPARATUS

Apparatus indicated in the method described for concentration above 50 mg/l.

5. SAMPLE PREPARATION

Sample preparation is indicated in the method described for concentrations above 50 mg/l.

6. PROCEDURE

The procedure is indicated in the method described for concentrations above 50 mg/l. (Resolution OENO 6/98), but with the introduction in the tank of a quantity of malic acid D equivalent to 50 mg/l. (Introduction of 0.025 mL of malic acid D at 0.199 g/l, substituting the equivalent volume of water); the values obtained are decreased by 50 mg/l.

7. INTERNAL VALIDATION

Summary of the internal validation file on the dosage of malic acid D(+)-after the addition of 50 mg/l of this isomer

Work level	0 mg of 70 mg of malic acid D(+)-per liter. Within these limits, the method is linear with a correlation coeffiency between 0.990 and 0.994
Setting limit	24.4 mg/l
Detection limit	8.3 mg/l
Sensitivity	0.0015 abs / mg/l
Recovery percent range	87.5 to 115.0% for white wines and 75 to 105% for red wines

Certified in conformity Bratislava, 28th June 2002
The Director General of the OIV
Secretary of the General Assembly
Georges DUTRUC-ROSSET

Repeatability	=12.4 mg/l for white wines (according to the OIV method =12,5 mg/l) =12.6 mg/l for red wines (according to OIV method=12,7 mg/l)
Percentage standard deviation	4.2% to 7.6% (white wines and red wines)
Intralaboratory variability	CV=7.4% (s=4.4mg/l; X average=59.3 mg/l)

8. BIBLIOGRAPHY

1. Chretien D., Sudraud P., 1993. Présence naturelle d'acide D(+)-malique dans les moûts et les vins, Journal International des Sciences de la Vigne et du Vin, 27: 147-149.
2. Chretien D., Sudraud P., 1994. Présence naturelle d'acide D(+)-malique dans les moûts et les vins, Feuillet Vert de l'OIV, 966.
3. Delfini C., Gaetano G., Gaia P., Piangerelli M.G., Cocito C., 1995. Production of D(+)-malic acid by wine yeasts, Rivista de Viticoltura e di Enologia, 48: 75-76.
4. OIV, 1998. Recueil des méthodes internationales d'analyse des vins et des moûts.Mise à jour Septembre 1998. OIV, Paris.
5. Przyborski H., Wacha C., Bandion F., 1993. Zur bestimmung von D(+)Apfelsäure in wein, Mitteilung Klosterneuburg, 43: 215-218.
6. Machado M. and Curvelo-Garcia A.S., 1999; FV.O.I.V. N° 1082, Ref. 2616/220199.

Certified in conformity Bratislava, 28th June 2002
The Director General of the OIV
Secretary of the General Assembly
Georges DUTRUC-ROSSET