RESOLUTION OENO 23/2003

DOSAGE OF SUGARS IN WINE BY HPLC

THE GENERAL ASSEMBLY,

CONSIDERING Article 5, paragraph 4 of the International Convention for the Unification of Methods of Analysis and Appraisal of Wine of 13 October 1954,

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

DECIDES to introduce in Annex A of the Compendium of International Methods of Wine and Musts, the following method as a type II method:

Dosage of sugars in wine by HPLC

1. FIELD OF APPLICATION

This recommendation specifies a method for determining fructose, glucose and saccharose in musts and wine by high performance liquid chromatography.

2. PRINCIPLE

Sugars and glycerol are directly determined by HPLC and detected by refractometry.

3. REAGENTS AND PREPARATION OF REACTIVE SOLUTIONS

3.1. Deminiralised water;

3.2. Acetonitrile - Transmission minimum at 200 nm - purity >99%;

3.3. Methanol - purity >99%;

3.4. Ethanol - 95-96%;

3.5. Fructose - Purity > 99%;

3.6. Glucose – Purity > 99%;

3.7. Saccharose D(+) - Purity > 99%;

3.8. Glycerol - Purity > 99%;

3.9. Nitrogen purity > 99%;

3.10. Helium purity > 99%.

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Preparation of reactive solutions

3.11. Demineralised water (3.1) filtered on a 0.45 µm cellulose membrane (4.13) using the filtration system (4.11);

3.12. Eluent: In a 1 litre graduated test tube with a stem (4.7), pour 800 ml of acetonitrile (3.2) in a 1 l flask (4.8) then 200 ml of water (3.11). Continually degas using helium (3.10). In the case if the system works in a closed circuit (eluent going back into the flask), the mixture is renewed every week.

4. APPARATUS

4.1. Conical flasks 100 ml;

4.2. Cylindrical vases 100 ml;

4.3. Cylindrical vases 50 ml;

4.4. Automatic pipette 10 ml;

4.5. Cones for pipette 10 ml;

4.6. Volumetric flask 100 ml;

4.7. Test tube 1 litre;

4.8. Flask 1 litre;

4.9. Syringe 20 ml with needle;

4.10. Syringe 10 ml with needle;

4.11. Filtration system;

4.12. Filter holders;

4.13. Membrane 0.45 µm in cellulose;

4.14. Membrane 0.8 µm in cellulose;

4.15. Membrane 1.2 µm in cellulose;

4.16. Membrane 5.0 µm in cellulose;

4.17. Prefilters in cellulose;

4.18. Filter cartridge of silica grafted by octadecyl groups (e.g. Sep packs C18);

4.19. Strechable film e.g. Parafilm;

4.20. Conical flasks 10 ml;

4.21. Common apparatus for high performance liquid chromatography

4.22. Column, alkylamine 5 µm and 250 □ 4 mm – Conditioning of the columns: the columns are generally filled and tested with hexane. They have to be washed with 50 ml of ethanol (3.4), then 50 ml of methanol (3.3) before undergoing acetonitrile/water mixture 80/20 (3.12). Start at a slow rate, meaning 0.1 ml/min, then progressively

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increasing up to 1 ml/min in order to avoid packing of the phase.

4.23. Refractometric detector - Rinse the reference cell once or twice a day (in between two analyses) with the acetonitrile/water eluant (3.12). Wait about ¼ hour so that the base line stabilises. Adjust the zero of the refractometer.

4.24. Ultrasound bath

5. SAMPLING

The samples are degassed with nitrogen beforehand (3.9) in an ultrasound bath (4.24)

6. PROCEDURE

6.1. Preparation of the sample

6.1.1. Filtration

6.1.1.1. Take 25 ml of the sample using a 20 ml syringe (4.9) with a needle and filter

- on a 0.45 µm membrane (4.13) for a wine
- on a pile of filters 0.45 (4.13) – 0.8 (4.14) – 1.2 (4.15) - 5 (4.16) µm + prefilter (4.17) for a must or a non clarified wine.

6.1.1.2. Dilute five times for musts. Take 20 ml using a 10 ml automatic pipette (4.4) with a cone (4.5), pour into a 100 ml volumetric flask (4.6). Bring to 100 ml with demineralised water (3.11), seal the flask with parafilm (4.19), homogenise.

6.1.2. Elimination of phenolic compounds

For a must or wine: pass over a filter cartridge C18 (4.18).

6.1.2.1. Preparation of the filter cartridge C18

Pass the opposite way (large tip) 10 ml of methanol (3.3), then 10 ml of demineralised water (3.11).

6.1.2.2. Pass on filter cartridge C18

Rinse the 10 ml syringe (4.10) with about 2 ml of sample. Take about 9 ml of the sample. Connect the sep pack C18 (4.18) by the small tip to the 10 ml syringe (4.10), pass the wine through the cartridge while eliminating the first three ml. Gather the remaining 6 ml in an 10 ml conical flask (4.20). Rinse the filtration cartridge C18 in the opposite direction with 10 ml of methanol (3.3), then 10 ml of demineralised water (3.11) after

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each sample. In this case, the cartridges can be reused.

6.1.3. Normal cleaning

Syringes (4.9), (4.10) and needle (4.11) rinsed with demineralised water (3.1) after each sample;
Filter holder is rinsed with hot water, then with methanol (3.3). Let dry naturally.

6.2. Analysis

6.2.1. Analytical conditions

Mobile phase: isocratic eluant acetonitrile/water (80/20, v/v) (3.12);
Flow: 1 ml/mn;
Injected volume 20 µl.
Detector (4.23) to be parameterised according to the apparatus.
Examples of chromatograms are shown in Annex B, figures 1 and 2

6.2.2. External calibration

Synthetic calibration mixture composed of:
fructose (3.5) 10 g/l □ 0.01 g/l;
glucose (3.6) 10 g/l □ 0.01 g/l;
saccharose (3.7) 10 g/l □ 0.01 g/l;
to which glycerol can be added (3.8) (if we wish to quantify it) 10 g/l □ 0.01 g/l.
Calculation of responsitivity

- RF. = surface i/Ci

where
surface i = peak surface of the product in the calibration solution
and Ci = quantity of product present in the calibration solution

7. EXPRESSION OF RESULTS

Calculation of concentrations

- Ce = surface e/RFi

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Where surface e = peak surface of product present in the sample.

The results are expressed in g/l;

Take into account any possible dilutions.

8. CONTROL OF RESULTS

Comparative method;

Indirect connection by mass, volume and temperature;

Synthetic and/or reference controls are inserted among the samples.

9. PERFORMANCES OF METHOD

The analysis takes about 50 min;

Influence of certain wine compounds: glycerol is separated in the same conditions as the sugars. Therefore it can be measured during the same sequence. No known compound co-elutes with fructose, glucose or saccharose.

Robustness: the analysis is sensitive to low variations in temperature. The columns are covered in a foam sheath.

9.1. DETECTION AND QUANTIFICATION LIMITS

- LD fructose = 0.12 g/l
- LD glucose = 0.18 g/l
- LQ fructose = 0.4 g/l
- LQ glucose = 0.6 g/l

see Annex B.2

9.2. FIDELITY

9.2.1. Repeatability

The absolute difference between two individual results obtained on an identical wine submitted for test trial, by an operator using the same apparatus using the shortest time interval will not exceed the repeatability value in more than 5% of cases (See Annex B.3).

The values are:

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For G+F >5 g/l

- RSDr = 1%
- Repeatability limit r = 3% (2.8 RSDr)

For G+F between 2 and 5 g/l

- RSDr = 3%
- Repeatability limit r = 8% (2.8 RSDr)

9.2.2. Reproducibility

The absolute difference between two individual results obtained on an identical wine submitted for test trial in two laboratories will not exceed the repeatability value in more than 5% of cases (See Annex B.4).

For G + F >5 g/l

- RSDR = 4%
- Reproducibility limit R= 10% (2.8 RSDr)

For G + F between 2 and 5 g/l

- RSDR = 10%
- Reproducibility limit R = 30% (2.8 RSDr)

Annex A

Bibliography

1. TUSSEAU D. et BOUNIOL Cl. (1986), Sc. Alim., 6, 559-577;
2. TUSSEAU D., 1996. Limite de détection - limite de quantification. Feuillet Vert OIV 1000.
3. Protocole de validation des méthodes d'analyse. Résolution OIV OENO 6/2000

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4. Exactitude des résultats et méthodes de mesure. Norme NF ISO 5725

Annex B

Statistical results of the interlaboratory test trial

B.1 Samples of the interlaboratory test trial

This study was carried out by the Interregional Laboratory of the Répression de Fraudes in Bordeaux. The test trial involved 12 samples identified from A to J (4 white wines and 4 red wines; 2 white Port wines and 2 red Port wines) containing glucose and fructose and whose content of each sugar was between 2 and 65 g/l. The wines from the region of Bordeaux were supplemented in glucose and fructose and stabilised by 100 mg/l of SO_2.

In fact, it concerned 6 different samples in blind duplicate.

B.2 Chromatographic conditions

Considering the response factors of these two sugars and the scales of the chromatograms, the noise corresponds to a concentration in fructose of 0.04 g/l and in glucose of 0.06 g/l (see figure 3).

Then the limits of detection (3 times the noise) and of quantification (10 times the noise) are obtained:

- LD fructose = 0.12 g/l
- LD glucose = 0.18 g/l
- LQ fructose = 0.4 g/l
- LQ glucose = 0.6 g/l

These results are compliant with those determined by TUSSEAU and BOUNIOL (1986) and are repeatable on other chromatograms.

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B.3 Fidelity

Nine laboratories participated in this study.

The analyses of 3 points of the set of calibration solutions and the 12 samples were carried out successively by applying the method of analysis given.

Five laboratories gave the regression lines obtained after analysis of the 3 points of the set of calibration solutions.

Four laboratories gave the results of 12 samples repeated 3 times, the other laboratories only gave a single result.

The chromatographic conditions were given by all laboratories. All of the laboratories applied the same method principle and the same type of chromatographic column as those defined previously. The only differences concern:

The injection of 50 µl instead of 20 µl for one laboratory,

The calibration solutions with a larger range (5 to 30 g/l of each sugar) for one laboratory.

The results were analysed according to the OIV protocol (Validation Protocol of methods of analysis – Resolution OENO 6/1999).

This protocol requires that the analyses need not be repeated; whereas, 4 laboratories gave results of analyses repeated 3 times. A single series was chosen (the first one) for the analysis of the results in compliance with the OIV protocol.

The calculations of repeatability according to Youden, reproducibility and Cochran and Grubbs tests were performed.

The data on the repetitions allowed to calculate a different way the standard deviations of repeatability (according to ISO 5725).

An invalid result was identified.

The results of the Cochran test has led us to eliminate the results of samples C and J for laboratory 1.

The Grubbs test did not detect outlier results to be excluded.

All results are in table I

Table I - RESULTS OF FRUCTOSE AND GLUCOSE CONTENTS OF THE 12 SAMPLES ANALYSED

Sample	A	B	C	D	E	F	G	H	I	J	K	L

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Sugar	F	G	F	G	F	G	F	G	F	G	F	G	F	G	F	G	F	G	F	G	F	G	F	G
LAB 1	5.9	2.9	9.5	10.4	68.4	56.1	13.0	10.9	5.0	2.5	2.3	2.7	10.4	12.6	13.3	12.0	64.7	43.5	75.2	68.8	65.3	45.7	2.1	2.9
	5.4	2.8	9.3	10.9	73.0	59.7	12.9	11.2	5.3	2.6	2.1	3.1	10.1	12.3	13.3	11.8	64.5	44.2	75.0	68.3	64.5	45.2	2.3	3.0
	5.5	2.9	10.0	11.2	73.6	58.6	12.9	11.0	5.4	2.7	2.2	2.8	9.9	12.2	13.3	12.0	63.9	43.5	77.4	70.5	65.1	45.8	2.1	2.9
	5.6	**2.9**	**9.6**	**10.8**	**71.7**	**58.1**	**12.9**	**11.0**	**5.2**	**2.6**	**2.2**	**2.9**	**10.1**	**12.4**	**13.3**	**11.9**	**64.4**	**43.7**	**75.9**	**69.2**	**65.0**	**45.6**	**2.2**	**2.9**
LAB 2	**5.1**	**2.4**	**10.0**	**12.6**	**74.5**	**67.0**	**13.4**	**12.3**	**5.0**	**2.2**	**1.5**	**2.2**	**10.0**	**13.0**	**13.4**	**12.3**	**64.2**	**42.9**	**76.8**	**69.3**	**64.4**	**43.4**	**1.4**	**0.4***
LAB 3	**5.3**	**3.0**	**9.8**	**12.6**	**72.5**	**66.3**	**13.0**	**12.6**	**5.4**	**3.4**	**1.9**	**3.1**	**10.4**	**14.2**	**13.4**	**13.4**	**63.9**	**45.0**	**73.8**	**69.9**	**65.6**	**47.3**	**2.0**	**3.3**
LAB 4	5.1	3.2	10.3	12.7	71.6	68.2	12.9	12.6	5.0	3.0	1.9	2.9	9.6	12.6	12.7	12.5	62.5	45.4	73.3	70.3	63.4	45.9	1.9	3.0
	5.3	3.0	9.7	12.6	74.0	69.8	12.9	12.6	5.1	2.9	1.8	3.1	10.0	12.7	13.1	13.0	63.0	46.4	74.2	70.6	62.1	46.2	1.9	2.8
	5.2	3.2	9.5	12.5	73.1	69.7	12.8	12.7	5.2	2.9	2.0	2.9	9.7	12.7	13.1	12.8	62.6	45.7	75.0	70.9	61.8	45.3	2.0	2.8
	5.2	**3.1**	**9.8**	**12.6**	**72.9**	**69.2**	**12.9**	**12.6**	**5.1**	**2.9**	**1.9**	**3.0**	**9.8**	**12.7**	**13.0**	**12.8**	**62.7**	**45.8**	**74.2**	**70.6**	**62.4**	**45.8**	**1.9**	**2.9**
LAB 5	**5.4**	**3.2**	**9.8**	**11.3**	**76.1**	**67.5**	**13.3**	**12.0**	**5.1**	**2.9**	**1.9**	**2.5**	**10.0**	**11.6**	**13.1**	**11.8**	**61.6**	**43.4**	**72.1**	**65.3**	**62.5**	**42.5**	**2.0**	**2.3**
LAB 6	**5.6**	**2.9**	**10.5**	**13.0**	**72.2**	**67.9**	**13.5**	**12.1**	**5.2**	**3.0**	**2.0**	**3.1**	**10.4**	**12.9**	**13.3**	**12.4**	**66.8**	**46.9**	**73.9**	**70.3**	**63.6**	**44.1**	**2.2**	**3.1**
LAB 7	**5.1**	**2.9**	**9.8**	**13.6**	**72.0**	**65.4**	**13.1**	**12.6**	**5.1**	**3.0**	**1.6**	**3.9**	**9.7**	**13.9**	**13.3**	**12.7**	**61.8**	**42.9**	**71.5**	**65.9**	**61.7**	**43.5**	**1.6**	**3.9**
LAB 8	5.1	2.8	9.7	12.4	73.7	70.0	13.0	12.7	5.1	2.9	2.0	3.0	10.1	13.0	12.8	12.6	61.6	45.6	71.7	68.6	61.6	45.5	2.1	3.3
	5.0	2.9	9.6	12.9	72.3	68.7	12.3	12.7	5.0	2.9	2.0	3.0	10.0	13.1	12.8	12.9	61.0	44.8	70.6	68.3	61.4	45.1	2.1	3.4
	5.0	3.0	9.6	12.9	72.7	66.7	12.6	12.7	5.0	2.9	2.0	3.0	10.1	13.1	12.6	12.7	61.2	45.4	71.5	68.5	61.2	45.2	2.1	3.3
	5.0	**2.9**	**9.6**	**12.7**	**72.9**	**68.5**	**12.6**	**12.7**	**5.0**	**2.9**	**2.0**	**3.0**	**10.1**	**13.1**	**12.7**	**12.7**	**61.3**	**45.3**	**71.3**	**68.5**	**61.4**	**45.3**	**2.1**	**3.3**
LAB 9	4.9	2.7	9.6	12.6	72.5	69.1	12.1	12.5	5.0	2.6	2.1	2.8	9.7	12.5	12.0	12.6	55.3	44.8	72.0	69.0	57.0	45.0	2.0	2.5
	4.9	2.7	9.0	11.5	79.5	70.2	12.6	12.9	4.8	2.7	2.2	2.5	9.1	11.6	12.5	13.0	60.2	42.6	79.0	70.2	60.3	43.0	2.2	2.5
	5.1	2.6	9.3	12.2	77.5	63.0	12.3	12.0	4.9	2.6	1.9	3.1	9.4	12.1	12.5	12.2	60.9	43.6	77.0	74.1	61.2	43.2	1.9	3.0
	5.0	**2.7**	**9.3**	**12.1**	**76.5**	**67.4**	**12.3**	**12.5**	**4.9**	**2.6**	**2.1**	**2.8**	**9.4**	**12.1**	**12.3**	**12.6**	**58.8**	**43.7**	**76.0**	**71.1**	**59.5**	**43.7**	**2.0**	**2.7**

* invalid result

For the 4 laboratories that gave 3 results, the averages are in bold

Sugar: F fructose, G glucose

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Global results in glucose + fructose

For the 4 laboratories that performed 3 repetitions, only the first series of data was used.

The samples double duplicated are mentioned in the first column and the respective results in the following columns.

Table II – RESULTS OF CONTENT IN G+F IN THE 6 DUPLICATED SAMPLES (in g/l)

	Lab 1		Lab 2		Lab 3		Lab 4		Lab 5		Lab 6		Lab 7		Lab 8		Lab 9	
A/E	8.8	7.5	7.5	7.2	8.3	8.8	8.3	8	8.6	8	8.5	8.2	8	8.1	7.9	8	7.6	7.6
B/G	20.3	23	22.6	23	22.4	24.6	23	22.2	21.1	21.6	23.5	23.3	23.4	23.6	22.1	23.1	22.2	22.2
C/J	125	144	142	146	139	144	140	144	144	137	140	144	137	137	144	140	142	141
D/H	23.9	25.3	25.7	25.7	25.6	26.8	25.5	25.2	25.3	24.9	25.6	25.7	25.7	26	25.7	25.4	24.6	24.6
F/l	5	5	3.8	1.8*	5	4.6	4.8	4.9	4.9	4.3	5.1	5.3	5.5	5.5	5	5.4	4.9	4.5
I/K	108	111	107	108	109	113	108	109	105	105	114	108	105	105	107	107	100	102

* invalid result (wrong integration of peaks)

The results of laboratory 1 for samples C/J were not kept in the following tables.

B.3.1. Repeatability of results for measuring glucose and fructose

The measurements of repeatability were performed according to 2 methods:

1 - repetitions of results of 4 laboratories out of 12 samples (test trials duplicated-TD). The data taken into account are the 2 first series of results given by these laboratories.

$$S_r ED = \sqrt{(\sum dt^2/2n}$$

Where di is the difference obtained from the 2 first repetitions of the analyses from the same sample by each laboratory and n the number of laboratories whose results are taken into account.

RSDr **ED** (%); it is the coefficient of variation of the standard deviation in relation to the mean value in %

repeatability limit r = 2.8 S_r **ED**

2 – repetitions of analyses on the same material analysed blindly (Youden pair -**YP**)

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$$S_r = \sqrt{\sum dt^2/2(n-1)}$$

Where di is the difference of the results obtained of the 2 samples blind duplicated by the same laboratory (example results of samples A and E by laboratory 3).

The results are in table III and are represented in figure 4.

Table III – RESULTS OF REPEATABILITY VALUES

Sample	F	L	A	E	B	G	D	H	I	K	C	J
Average content in G+F (g/l)	4.9	4.6	8.2	7.9	22.3	23.0	25.3	25.5	107	107	141	142
Sr ED (g/l)	0.04	0.05	0.08	0.05	0.24	0.21	0.14	0.17	0.43	0.27	1.56	1.05
r ED (g/l)	0.11	0.14	0.22	0.14	0.67	0.59	0.39	0.48	1.20	0.76	4.4	2.9
RSDrED (%)	0.8	1.1	0.9	0.7	1.1	0.9	0.6	0.6	0.4	0.3	1.7	0.7
SrPY (g/l)	0.14		0.10		0.24		0.12		0.50		0.80	
r PY (g/l)	0.39		0.28		0.67		0.34		1.4		2.2	
RSDrPY (%)	2.5		1.3		1.0		0.5		0.5		0.6	

The repeatability values are low and coherent according to the two estimation methods.

B.3.2 Reproducibility for determining glucose and fructose

Sugars in wine have been analysed for many years according to this method.

Routinely, an internal quality control is carried out using a reference material (for example: TITRIVINS – DUJARDIN SALLERON). The analyses of results has enabled to estimate the standard deviation of reproducibility over a long period (estimation renewed each year).

S_R intralaboratory = 0.5 g/l for glucose + fructose content equal to 12 g/l

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Table IV contains the reproducibility results obtained during this inter-comparison test trial. The laboratory effect was deduced S_L as indicated in the OIV resolution.

Sample	F	L	A	E	B	G	D	H	I	K	C	J
Average content in G+F (g/l)	4.9	4.6	8.2	7.9	22.3	23.0	25.3	25.5	107	107	141	142
SR inter(g/l)	0.5	0.4	0.4	0.4	1.0	1.0	0.6	0.6	3.2	2.9	2.2	3.2
SL (g/l)	0.43		0.39		0.97		0.59		3.0		2.6	
R inter (g/l)	1.3		1.12		2.8		1.7		8.5		7.6	
RSD R inter (%)	9.5		5		4.4		2.4		2.9		1.9	

Table IV – REPRODUCIBILITY RESULTS AND LABORATORY EFFECT

Figure 4 recapitulates the results of the standard deviation of reproducibility.

It appears that the repeatability values being low, it is the laboratory effect which is the origin of most of the dispersion of results.

Over a long period, the intralaboratory reproducibility has the same value as the interlaboratory reproducibility is observed as expected.

In conclusion, one can consider taking into account the trend curves that are represented in figure 4, that repeatability is relatively stable in the whole study zone (5 to 150 g/l of glucose + fructose), but that reproducibility is higher for lower concentrations.

On average, the repeatability (r repeatability limit) is relatively constant around 1% and reproducibility (R reproducibility limit) is between 2% and 5%; about 10% for glucose + fructose contents of 5 g/l.

So conventionally the repeatability and reproducibility values are:

For G+F >5 g/l

- RSDr = 1%
- RSDR = 4%
- Repeatability limit r = 3% (2.8 RSDr)

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- Reproducibility limit R= 10% (2.8 RSDr)

For G+F between 2 and 5 g/l

- RSDr = 3%
- RSDR = 10%
- Repeatability limit r = 8% (2.8 RSDr)
- Reproducibility limit R = 30% (2.8 RSDr)

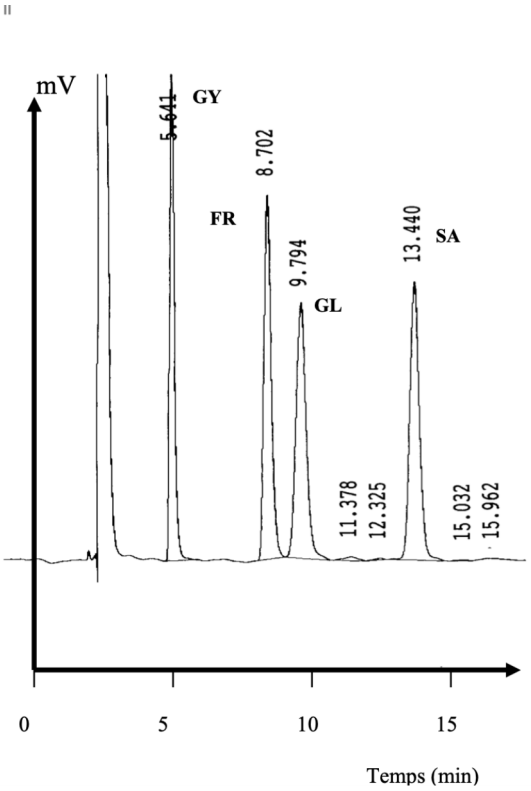


Figure 1 - Chromatogram of a calibration solution

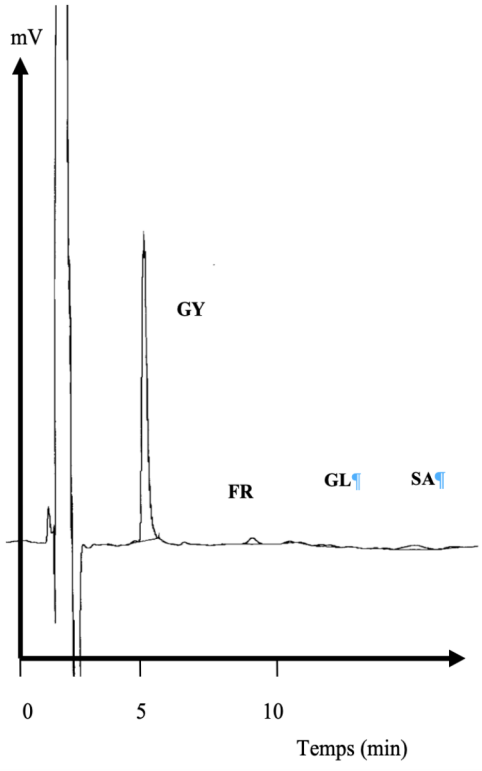
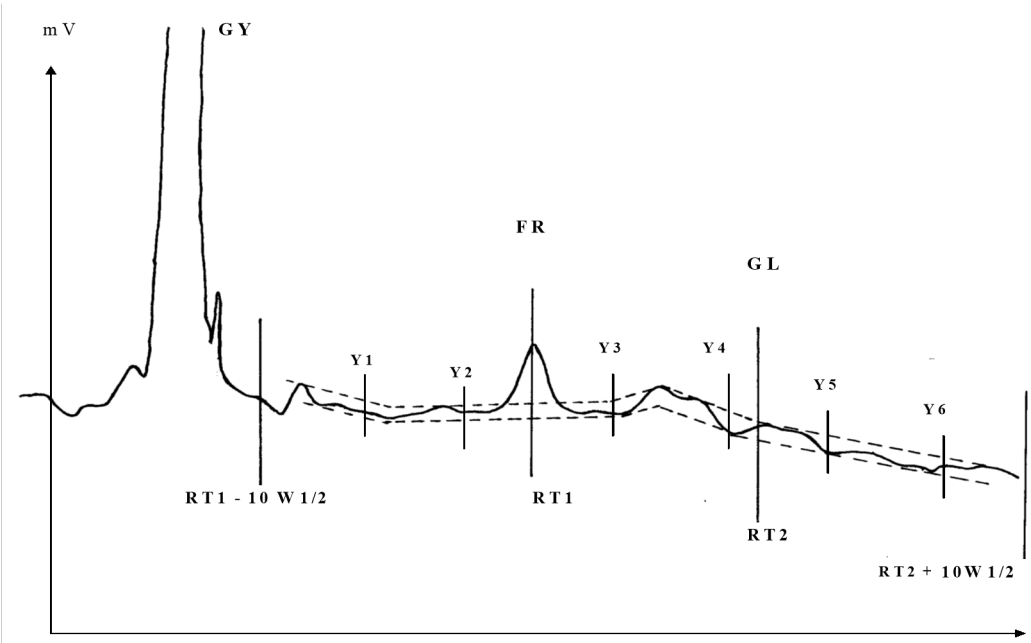


Figure 2 - Chromatogram of a rosé wine (sugars and glycerol at 10 g/l.)

Glycerol (GY), fructose (FR), glucose (GL), saccharose (SA)

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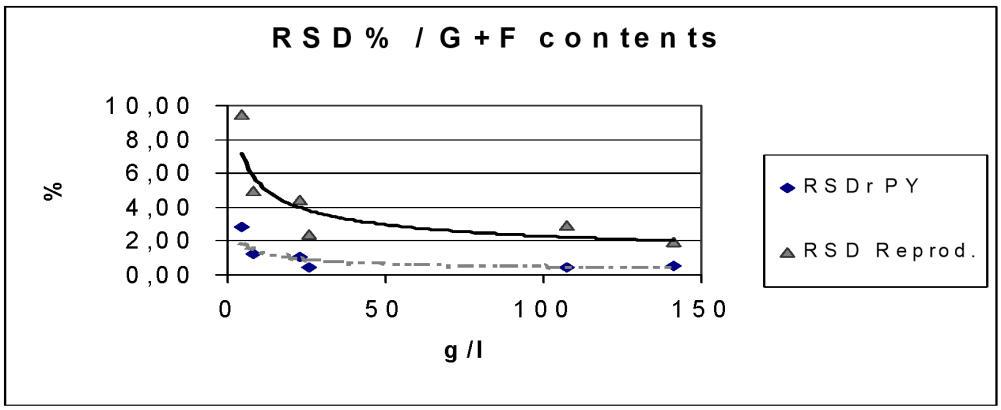


fructose (FR), glucose (GL), saccharose (SA) Glycerol (GY),

Figure 3 - Measure of pitches of noise after enlargement of chromatogram

RT1: retention time of fructose; RT2: retention time of glucose

W1/2: width of peak at mid-height; Yi: pitch of noise at point i



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Figure 4 – Representation of variation coefficients of standard deviations according to glucose + fructose contents.

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