# RESOLUTION OENO 7/2007

# OENOLOGICAL CARBON

THE GENERAL ASSEMBLY,

IN VIEW of Article 2 paragraph iv of the Agreement of 3 April 2001 establishing the International Organisation of Vine and Wine,

UPON THE PROPOSAL of the Sub-commission of the methods of analysis and appraisal of wines and the "Specifications of oenological products" expert group,

DECIDES to replace the existing monograph in the International Oenological Codex by the following monograph:

# OENOLOGICAL CARBON

**INS N°: 153**

## 1. OBJECT, ORIGIN AND FIELD OF APPLICATION

Oenological carbon are of plant (generally wood) origin. In order to increase their adsorption properties, the carbon is subjected to activation either at a high temperature or a lower temperature in the presence of an acid, (generally phosphoric acid). Oenological carbon must not be activated with a metal-based catalyser like zinc chloride.

It is in the form of very fine and light black powder, or in granulated form.

There are humid preparations which can reduce dust accumulation. In this case, weight loss as described in 3.1 can reach 60%.

Carbon can be agglomerated with bentonite; in which case, the ashes in 3.2 are more than 20%.

Carbon for oenological purposes is used to correct alterations due to fungus in addition to the colour of white musts from purple, spotted or oxidised grapes. The carbon can eliminate anthocyanins and oxidised or non-oxidised polyphenols in addition to polysaccharides. The carbon are used to correct the organoleptic characteristics of musts made from grapes altered by fungus.

Oenological carbon can also be used to reduce the presence of Ochratoxin A in musts, the musts during fermentation and in white wine.

Decolourising carbon has a relatively weak deodorising effect.



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Absorption by carbon is not very selective and depends on its structure, porosity and specific surface area.

The limit concerning the use of carbons should be compliant with the prescriptions of the OIV International Code of Oenological Practices (expressed by weight of dry carbon).

# 2. LABELLING

The label should indicate the storage conditions, the expiration date for humid solutions, and a mention of whether there are existing regulations regarding the usage of the product and specify if it concerns decolourising or deodorising carbon.

# 3. TEST TRIALS

## 3.1. Loss with dessication

Put 5g of carbon in a silica capsule and heat to 100°C in an incubator. After 3 hours of dessicating, weight loss should not be more than 20%.

**All limits set for carbon refer to dry carbon weight.**

## 3.2. Ashes

Incinerate the previously obtained dry residue at 550°C-600°C. These ashes should not be more than 10%.

## 3.3. Soluble matter in acids

Boil 5 g of dried carbon with 20 ml of concentrated hydrochloric acid (R) and 100 ml of water. Once cooled, filter using a fine filter or membrane.

Evaporate the filtrate and dry at 100°C–105°C. The soluble matter content in acids should not be more than 5%.

## 3.4. Chlorides

Shake 0.067g of dried carbon and 20 ml of distilled water. Filter. Add 5 ml of diluted nitric acid (R) to 5 ml of filtrate. Fill up to 20 ml and add 0.5 ml of silver nitrate solution at 5% (R).

Compare any opalescence or cloudiness to a prepared control sample as indicated in the annex. Other methods such as ionic chromatography can be used.

Chloride content should not be more than 3g/kg.



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## 3.5. Cyanides

Put a quantity of carbon containing 1 g of dried carbon with 10 ml of diluted sulphuric acid (R) in a 100 ml conical flask. Adapt to the conical flask a pressure relief tube plunged into approximately 2 ml of saturated borax solution (R) in a test tube. Distil and gather 2 to 3 ml of distillate. Add 5 drops of potassium anhydrosulphite solution at 2% (R) and leave for 5 minutes. Add 1 ml of iron sulphate solution (II) at 5% (R) and leave for 15 minutes. Then add 2 drops of phenolphthalein (R). Use a saturated borax solution (R) to make the solution a little more alkaline. Leave for 5 minutes. Add 2 drops of iron sulphate (III) and ammonia solution at 10% (R) and 1 ml of concentrated hydrochloric acid (R). No colouration nor blue precipitate should form.

## 3.6. Polycyclic aromatic hydrocarbons

Polycyclic aromatic hydrocarbons including benzo[a]pyrene are extracted by hexane; the solvent is evaporated and the residue is taken by the methanoltetrahydrofuran mixture for HPLC analysis following the method described in chapter II.

NOTE: It is also possible to determine benzo[a]pyrene by gas chromatography by using an apolar capillary column with detection by mass spectrometry following the method described in chapter II of the International Oenological Codex.

Benzo[a]pyrene content should not be more than 10 µg/kg.

## 3.7. Sulphides

Put a quantity of carbon containing 1 g of dried carbon with 10 ml of diluted hydrochloric acid and 10 ml of water in a 50 ml flask.

Distil and collect 5 ml of distillate in a test tube containing 5 ml of 1 M sodium hydroxide solution.

0.5 ml of lead nitrate solution at 1 g per litre (R) is added to 1 ml of test trial solution. There should be no brown colouring or black precipitate. Sulphide content expressed in sulphur should not be more than 20 mg/kg.

## 3.8. Preparation of test trial solution

Put a quantity of carbon corresponding to 2.5 g of dried carbon with 50 ml of a citric acid solution at 5 g a litre with a pH of 3 (R), in a conical flask with a wide opening that can be sealed. Shake vigorously for 5 minutes and allow to stand at least 12 hours. Filter through a fine filter or a membrane in order to obtain a clear solution.



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## 3.9. Iron

Add 5 ml of water, 1 ml of concentrated hydrochloric acid, 2 ml of 5% potassium thiocyanate solution (R) to 5 ml of test trial solution as prepared in point 3.8. The colouration obtained should be lighter than the control sample prepared with 10 ml of iron salt solution (II) at 0.010 g of iron per litre (R), and 1 ml of concentrated hydrochloric acid (R), 2 ml of 5% potassium thiocyanate solution (R). Atomic absorption spectrophotometry can also be used.

Iron content should not be more than 200 mg/kg.

## 3.10. Lead

Determine the lead according to the method described in chapter II on the test trial solution prepared according to point 3.8.

Lead content should not be more than 2 mg/kg.

## 3.11. Mercury

Determine the mercury according to the method described in chapter II on the test trial solution prepared according to point 3.8.

Mercury content should not be more than 1 mg/kg.

## 3.12. Arsenic

Determine the arsenic according to the method described in chapter II on the test trial solution prepared according to point 3.8.

Arsenic content should not be more than 3 mg/kg.

## 3.13. Calcium

Determine the calcium according to the method described in chapter II on the test trial solution prepared according to point 3.8.

Calcium content should not be more than 10 g/kg.

## 3.14. Cadmium

Determine cadmium according to the method described in chapter II on the test trial solution prepared according to point 3.8.

Cadmium content should not be more than 1 mg/kg.



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### 3.15. Zinc

Determine the zinc according to the method described in chapter II on the test trial solution prepared according to point 3.8.

Zinc content should not be more than 25 mg/kg.

### 3.16. Specific surface area

The specific surface area of a decolourising carbon must be between 600 and 2000 $m^2/g$.

Methylene blue decolourisation is the method used. (Methylene blue indicator).

### 3.17. Methylene blue indicator

Prepare 4 conical flasks and place 0.1 g of carbon.

Add 10, 15, 17 and 20 ml of methylene blue solution at 1.2 g/l (absorbance at 620 nm is between 0.830 and 0.850).

After shaking for 5 minutes, filter through a slow filter and note the volume of the solution in the conical flask which underwent decolourisation.

Depending on the results, repeat this experiment with different volumes of solution.

Put the solution in a spectrophotometer at 664 nm with the absorbance value of 0.08 with an optical path of 1 cm.

The volume of the methylene blue test solution in ml just discoloured, represents the methylene blue indicator.

## 4. Phenol index

### 4.1. Introduction

hen activated carbon is applied in the treatment of wine, the phenol index can be used to define a limit value over which the carbon is considered as a decolouriser and under which it is regarded as a deodoriser. The phenol index selected is the AWWA B600-90 index

### 4.2. Principle:

AWWA phenol index: this index, expressed in g of carbon scaled to the dry weight per l ofsolution represents the carbon powder concentration required to decrease the phenol concentration of a solution from 200 mg/l to 20 mg/l.



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## 4.3. Description of the AWWA method:

This index is determined using an adsorption isotherm based on at least 4 different weights of carbon put in contact with a phenol solution.

This isotherm represents the weight of phenol adsorbed in mg/l/g carbon, in relation to the residual phenol concentration in the solution, expressed in mg/l.

## 4.4. Reagents

4.4.1. Pure disodic hydrogenophosphate $Na_2HPO_4$ for analysis

4.4.2. Distilled water

4.4.3. Pure phosphoric Acid ($H_3PO_4$)

4.4.4. Pure phenol

4.4.5. Buffer solution A of disodic hydrogenophosphate with a pH of 6.5 at 104 g/l

In a 1-litre graduated flask, dissolve 104 g of disodic hydrogenophosphate (4.4.1) in 300 ml of hot water (4.4.2), add 14 ml of phosphoric acid (4.4.3) and make up to one litre. Homogenise. Check that the pH is 6.5 ± 0.1

4.4.6. Buffer solution B of disodic hydrogenophosphate with a pH of 6.5 at 10.4 g/l

In a 1-litre graduated flask, place 100 ml of buffer solution A at 104 g/l (4.4.5) and make up with water (4.4.2). Homogenise.

4.4.7. Phenol solution with 1 g/l

In a 100-ml graduated flask, place 100 mg of phenol (4.4.4) and make up to 100 ml with water (4.4.2). Obtain complete dissolution by stirring.

4.4.8. Calibration solutions of phenol with 20, 40, 60, 80, 100, and 120 mg/l

In a series of 100-ml graduated flasks, respectively place 2 ml, 4 ml, 6 ml, 8 ml, 10 ml, and 12 ml of the phenol solution with 1 g/l (4.4.7). Make up to 100 ml using buffer solution B (4.4.6).

4.4.9. Phenol solutions with 200 mg/l

In a 1-litre flask, place 200 ml of the phenol solution at 1 g/l (4.4.7), add 100 ml of buffer solution A (4.4.5), make up to 1 l with water (4.4.2). Homogenise.

4.4.10. Measuring the phenol index of oenological carbon powder

**Note:** The water content of the carbon must be known in order to scale the index to the dry carbon weight.

## 4.5. Apparatus

4.5.1. Laboratory glassware i.e.: graduated precision pipettes to measure small



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volumes, 100-ml and 1-l graduated flasks, funnels, and 300-ml conical bottles

4.5.2. Filter paper

4.5.3. Laboratory balance, precision to within 0.10 mg

4.5.4. Spectrometer capable of operating in the ultraviolet spectrum and housing quartz tanks with an optical thickness of 1 cm.

4.5.5. Laboratory shaker (it is not recommended to use a magnetic bar)

## 4.6. Procedure

4.6.1. Phenol calibration curve.

Measure the absorbance at 270 nm in tanks with an optical thickness of 1 cm (4.5.4) of each phenol solution with 20, 40, 60, 80, 100, and 120 mg/l (4.4.8). Calculate the straight regression line of the absorbance in relation to the phenol concentration.

Note The blank is based on buffer solution B (4.4.6).

4.6.2. Determine the residual phenol for each carbon (4.4.10)

In a series of 300-ml conical flasks, place 200 ml of phenol solution at 200 mg/l (4.4.9), then respectively 0.4, 0.5, 0.6 and 0.7 g of carbon; close the bottle.

For these 4 preparations, stir for 30 minutes (4.5.5) so that the carbon remains in suspension.

Filter on paper (4.5.2) the 4 samples containing the carbon and a blank (phenol solution with 200 mg/l (4.4.8) without carbon).

Measure the absorbance at 270 nm in tanks with an optical thickness of 1 cm (4.5.4) of each one of the filtered solutions.

Note 1 The blank is based on buffer solution B (4.4.6).

Note 2 At least one of the quantities of carbon must adsorb 90% of the phenol in the solution; if not, widen the carbon weight range.

## 4.7. Calculations

4.7.1. Determine the percentage of residual phenol in each filtrate for each activated carbon: residual % = milligram per litre of residual phenol filtrate * 100/200 (milligram per litre of phenol in the test solution). **i.e. a = % residual phenol**

4.7.2. Determine the percentage of X (adsorbed phenol)

- % of X = 100 -% residual in the filtrate. **i.e. X** = 100 – a

4.7.3. The quantities of activated carbon for 200 ml of phenol solution are multiplied



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by 5 to obtain the quantities of activated carbon, **i.e. M** in grams per litre.

4.7.4. Calculate the percentage of the value of **X/M** for each activated carbon.

4.7.5. Plot the isotherm: percentage of residual normality of the filtrate on the X-axis (a) and the percentage of X/M on the Y-axis using 2x2 logarithmic paper; establish the straight regression line and determine the regression equation. It is also possible to calculate the regression using the logarithm for the values of a and X/M.

4.7.6. Determine X/M at 10%; **i.e. C** (when the residual phenol concentration of the filtrate is 10%).

4.7.7. **Phenol index** in grams per litre = 90/C * (100 -% of humidity/100); **i.e. P**

This formula refers to activated carbon without humidity.

4.7.8. **Limit values**

A carbon is regarded as a deodouriser if its phenol index is lower than 3.5

4.7.9. Examples

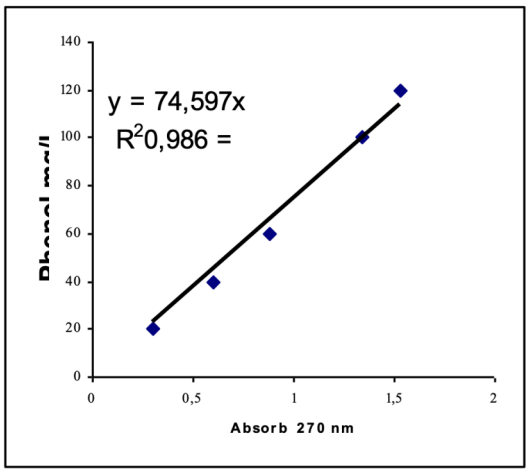
Standard curve

| Abs at 270 nm | Phenol mg/l |
|---|---|
| 0.303 | 20 |
| 0.603 | 40 |
| 0.8777 | 60 |
| 1.3443 | 100 |
| 1.53 | 120 |



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Calibration straight line for phenol titration

| A1 | g of carbon | % humidity | Dry weight | Abs | C phenol |
|---|---|---|---|---|---|
| | | 1.31 | 0.2467 | 0.309 | 23 |
| | | 1.31 | 0.6173 | 0.065 | 5 |
| | 0.25<br>0.6255<br>0.7136<br>0.7829 | 1.31 | 0.7043 | 0.0454 | 3 |
| | | 1.31 | 0.7726 | 0.0367 | 3 |

A1
Calculations

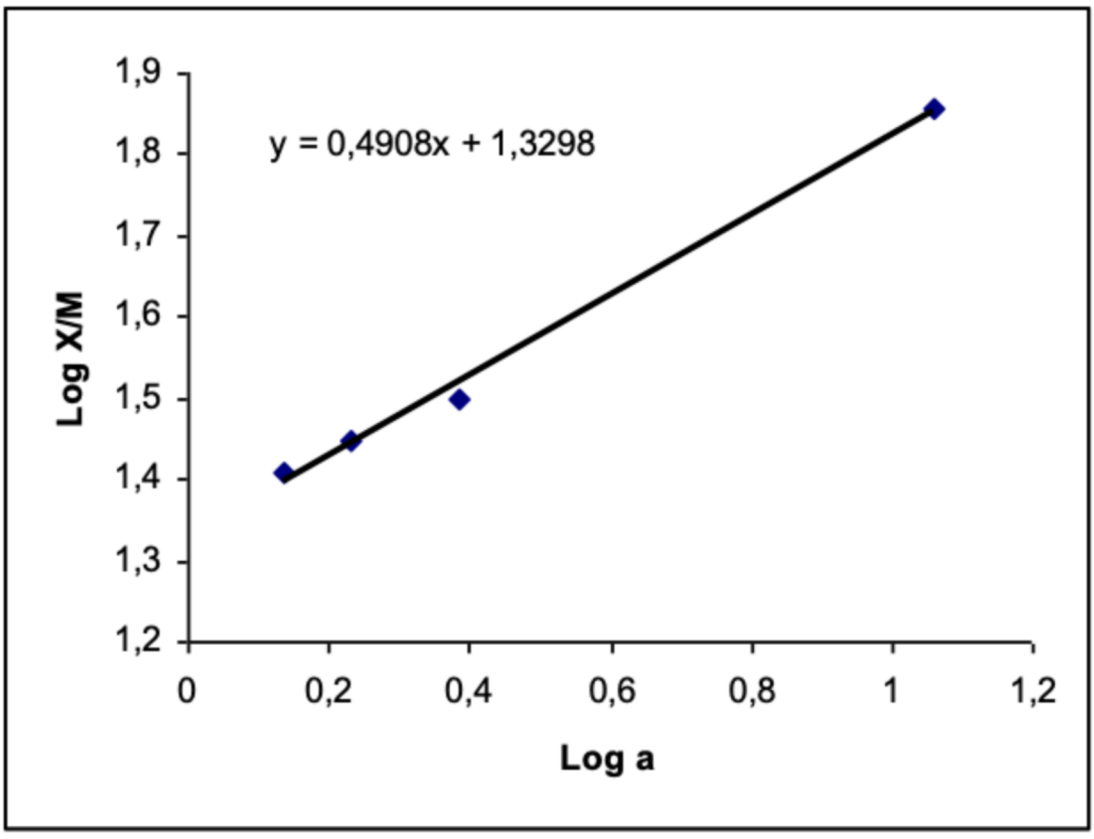
| a | X | M | a | X/M | Log a | Log X/M |
|---|---|---|---|---|---|---|
| 11.53 | 88.47 | 1.23 | 11.53 | 71.72 | 1.061829 | 1.855636 |
| 2.42 | 97.58 | 3.09 | 2.42 | 31.61 | 0.384605 | 1.499871 |
| 1.69 | 98.31 | 3.52 | 1.69 | 27.92 | 0.228747 | 1.445885 |



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| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1.37 | 98.63 | 3.86 | 1.37 | 25.53 | 0.136357 | 1.407065 | |
| | | | | c | | | P |
| | | | 10.00 | 66.16 | 1 | 1.8206 | 1.3 |




Adsorption isotherm of carbon A1

| A2 | g of carbon | % Humidity | Dry weight | Abs | C phenol |
|---|---|---|---|---|---|



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| | | | | | |
|---|---|---|---|---|---|
| | | 1.60 | 0.3989 | 0.9969 | 74 |
| | | 1.60 | 0.5916 | 0.679 | 51 |
| 0.4054 | | 1.60 | 0.7787 | 0.4972 | 37 |
| 0.6012<br>0.7914 | | 1.60 | 0.8887 | 0.4126 | 31 |
| 0.9032<br>1.4040 | | 1.60 | 1.3815 | 0.2654 | 20 |

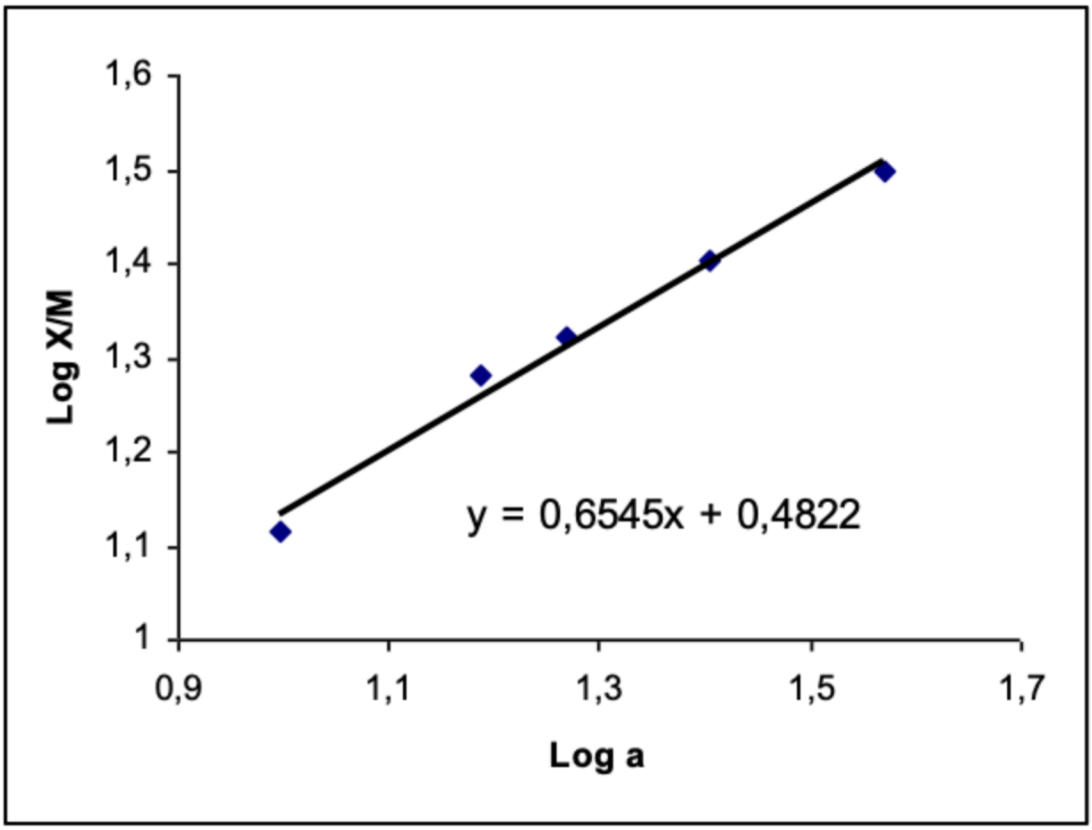
A2
Calculations

| a | X | M | a | X/M | Log a | Log X/M | |
|---|---|---|---|---|---|---|---|
| 37.18 | 62.82 | 1.99 | 37.18 | 31.49 | 1.570343 | 1.498241 | |
| 25.33 | 74.67 | 2.96 | 25.33 | 25.25 | 1.403561 | 1.402199 | |
| 18.54 | 81.46 | 3.89 | 18.54 | 20.92 | 1.268222 | 1.320569 | |
| 15.39 | 84.61 | 4.44 | 15.39 | 19.04 | 1.187221 | 1.279687 | |
| 9.90 | 90.10 | 6.91 | 9.90 | 13.04 | 0.995635 | 1.115409 | |
| | | | | c | | | P |
| | | | 10 | 13.70 | 1 | 1.1367 | 6.5 |



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Adsorption isotherm of carbon A2

4.7.10. Collaborative analysis: AWWA phenol indices in g/l

| | Lab 1 | Lab 2 | Lab 3 | Lab 4 |
|---|---|---|---|---|
| A1 | 1.7 | 1.53 | 1.8 | 1.3 |
| A2 | 5.1 | 4.56 | 6.2 | 6.5 |
| A3 | 1.3 | 1.29 | 1.8 | 1.4 |
| A4 | 5.8 | 4.95 | 10.0 | 7 |
| B1 | 11.4 | 7.18 | 10.6 | 7.6 |



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| | | | | |
|---|---|---|---|---|
| B2 | 1.8 | 1.47 | 2.3 | 1.4 |
| B3 | 49.4 | 21.97 | 18.0 | 17.5 |
| B4 | 2.9 | 2.80 | 3.6 | 2.6 |
| C1 | 1.9 | 1.69 | 2.3 | 1.8 |
| C2 | 1.7 | 1.56 | 2.0 | 1.5 |
| C3 | 5.4 | 4.71 | 6.2 | 4.9 |
| C4 | 5.4 | 4.55 | 6.0 | 4.7 |

Reproducibility: 2.88 for the 5.86 general average SR = 1,03

# 5. DETERMINATION OF THE DECOLOURISATION CAPACITY OF CARBON

## 5.1. Principle

Measuring the decolourisation of an oenocyanin solution with a precise amount of carbon under defined conditions.

## 5.2. Apparatus:

Equipment:

5.2.1. Precision balance in mg

5.2.2. Magnetic stirrer

5.2.3. Absorption spectrophotometer for OD to 420, 520 and 620 nm measures

Glassware:

5.2.4. 250 ml cylindrical flask

5.2.5. 250 ml conical flask

5.2.6. 200 ml volumetric flask

5.2.7. Chamber with a 1 mm optical path for an absorption spectrophotometer.



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## 5.3. Reagents

5.3.1. Very pure demineralised water

5.3.2. Crystallised acetic acid

5.3.3. Tartaric acid

5.3.4. Crystallised sodium acetate

5.3.5. 96% volume ethanol

5.3.6. Oenocyanin powder

## 5.4. Preparation of oenocyanin solution

5.4.1. Pour approximately 150 ml of demineralised water (5.3.1) in a 250 ml cylindrical flask (5.2.4).

5.4.2. Shake (5.2.2).

5.4.3. Weigh 0.900 g ± 0.001 g of oenocynanin (5.3.6) and dissolve by adding small amounts while stirring in a vortex mixer.

5.4.4. Weigh 1.400 g ± 0.01 g of tartaric acid (5.3.3) and pour into the cylindrical flask (5.2.4).

5.4.5. Pour 0.8 ml of crystallised acetic acid (5.3.2) and 1.4 g of crystallised sodium acetate (5.3.4).

5.4.6. Shake continuously until completely dissolved (5.2.2)

5.4.7. Transfer to a 200 ml volumetric flask (5.2.6).

5.4.8. Adjust to 200 ml with the rinsing water from the cylindrical flask of the 5.4 preparation.

5.4.9. Transfer again into a 250 ml cylindrical flask (5.2.4).

5.4.10. Shake (5.2.2).

5.4.11. Centrifuge 150 ml of the solution for 10 minutes at 10,000 g place the supernatant in a cuvette with 1 mm optical path

5.4.12. Measure the colour intensity of the spectrometer (5.2.3)

- CI1 = OD 420 + OD 520 + OD 620
- CI1 = 4 ± 0.3

## 5.5. Decolourisation by carbon

5.5.1. Weigh 100 mg of dried carbon.



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(Measure the humidity in order to define the exact dose of humid carbon to be used).

5.5.2. Put the carbon in 100 ml of oenocyanin solution with colour intensity

- CI 1 = 4 ± 0.3

5.5.3. Shake for 30 minutes (5.2.2).

5.5.4. Allow to stand for 10 minutes and centrifuge 10 ml of this mixture for 10 minutes at 10,000 g.

5.5.5. Measure the colour intensity with a spectrometer (5.2.3) under 1 mm of optical path:

- CI 2 = OD 420 + OD 520+ OD 620

### 5.6. Calculation of decolourisation capacity

The decolourisation capacity (DC): DC = 100 (CI1 – CI2) / CI1

Carbon is considered as a 'decolourising agent' when DC is more than or equal to 40.

## 6. STORAGE

Carbon cannot be stored in open bags because of its adsorption capacities. Oenological carbon must be stored in sealed packages away from volatile substances that it could adsorb.



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