



LABORATORY PRACTICES IN WINE ANALYSIS

Dpto. Nutrición y Bromatología II. Facultad de Farmacia. UCM



ANALYTICAL DETERMINATIONS IN WINE

▶ pH

▶ TOTAL ACIDITY

▶ VOLATILE ACIDITY

▶ ALCOHOLIC STRENGTH

▶ SULPHUR DIOXIDE

▶ FOLIN-CIOCALTEU INDEX



DETERMINATION OF pH

Potentiometric method

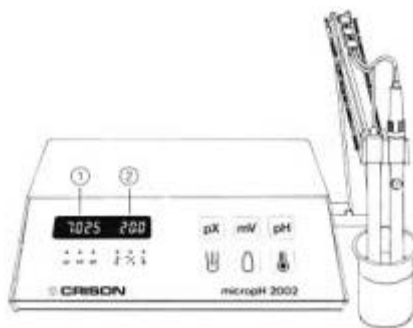
PURPOSE.- Potentiometric measurement of pH in wine

PRINCIPLE.- The difference in potential between an electrode of pH and the reference electrode both immersed in the sample is measured with a pH-meter.

Procedure

1. Calibration

- Connect the pH-metro
Screen: 0000 20°C
- Adjust T
- Calibration with standard sol.
pH 7.02 y pH4.00



- Dip the electrode until the diafrgm is immersed
Magnetic stirring
- Wash with distilled water an
dry between
measurements.

2. Measurement

- Dip the electrode into the wine: 15 s. Read the pH
- Carry out two determinations.
- Note down the results



DETERMINATION OF TOTAL ACIDITY

Volumetric method

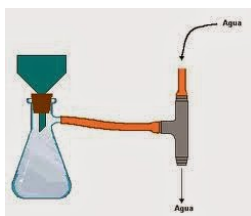
Principle.- Titration with NaOH N/10 and bromothymol blue as indicator and comparison with an end-point colour standard or potentiometric titration with a pH-meter to $\text{pH} = 7,00 \pm 0,5$.

Procedure

1. Preliminary operations

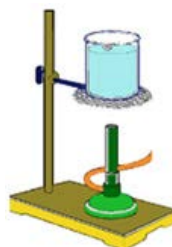
Elimination of CO₂ in wine

- 50mL wine
- Kitasato 500mL
- Connection to vacuum pump
- Agitation 1-2'



Elimination of CO₂ in H₂O

- 100 mL H₂O
- Beaker
- Boiling
- Cooling at room T



Calibration of pH-meter

Sol. standard
pH 7.02 and pH 4.00



DETERMINATION OF TOTAL ACIDITY

Volumetric method

Titration with bromothymol blue as indicator

Obtention of colour standard

NaOH N/10



25 ml Boiled distilled H₂O
1 ml bromothymol blue
10 ml white wine



Yellow

Green

+ 5ml buffer sol. pH 7



Blue-green

Titration of wine

NaOH N/10

30 ml Boiled distilled H₂O
1 ml bromothymol blue a
10 ml white wine



Yellow

Blue-green

Comparison of colour

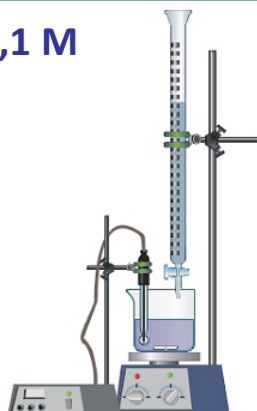
Potentiometric titration with a pH-meter

Expressed in: g tartaric acid/L Wine

NaOH 0,1 M



25 ml Boiled distilled H₂O
10 ml white wine CO₂ free



- Place it in magnetic stirrer with stirring bar
- Place the calibrated pH electrode
- Magnetic stirring at low speed

Titration **End point** pH = $7 \pm 0,5$



DETERMINATION OF VOLATILE ACIDITY

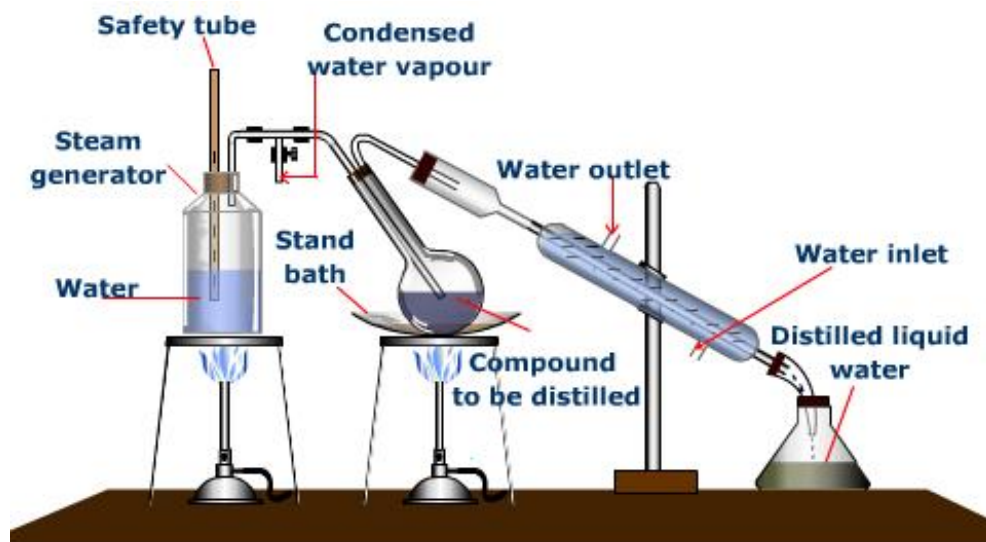
Volumetric method

PRINCIPLE.- Volatile acids (acetic, formic, propionic and butiric) are separated from the wine by steam distillation and titrated using NaOH with phenolphthalein as indicator. The acidity of free and combined sulphur dioxide distilled should be subtracted from the acidity of the distillate.

Procedure

Steam distillation

Conventional distillator



Automatic distillator



Distillation tube:
20 mL wine
+ tartaric acid

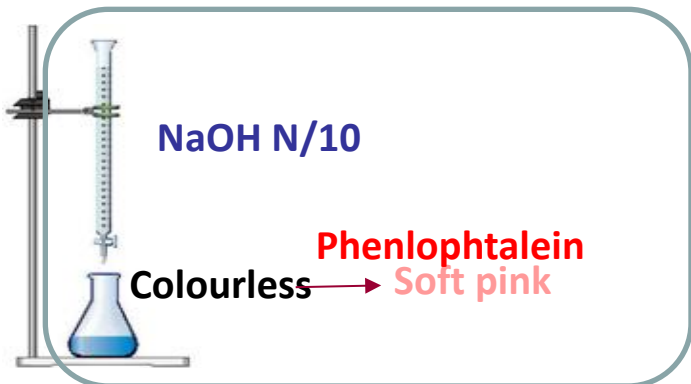
Collecting
flask
250 mL

DETERMINATION OF VOLATILE ACIDITY

Volumetric method

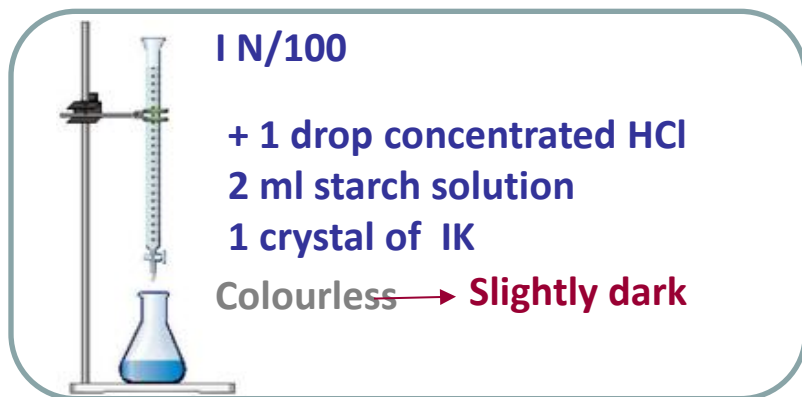
Expressed in:
g acetic acid/L Wine

1. Titration of volatile acidity + free and combined SO_2

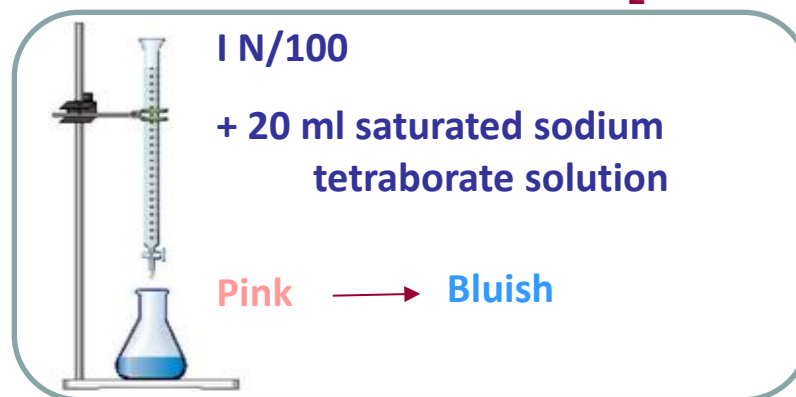


Redox titration

2. Titrate the free SO_2



3. Titrate the combined SO_2



Redox titration



Redox titration



ALCOHOLIC STRENGTH BY VOLUME

Measurement using a pycnometer

PURPOSE.- Measurement of alcoholic strength by volume (%) in wines

Principle.- Distillation of wine made alkaline. Measurement of the alcoholic strength of the distillate with a pycnometer.

PROCEDURE

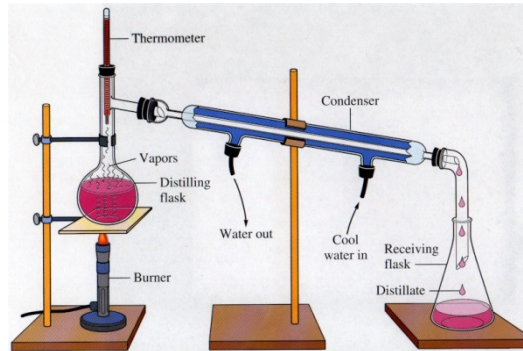
1. Collect the alcohol by distillation

100 mL wine (remove CO_2)

+ calcium hydroxide
+ pieces of inert porous material



Apparatus



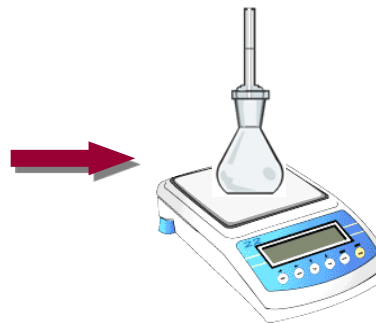
Collect the distillate



2. Measurement of the alcohol in the distillate with a pycnometer at 20°C

Weighing of :

- 1) Pycnometer (p)
- 2) Pycnometer + distilled water (p')
- 3) Pycnometer + distillate (p'')



Calculate
Density

$$D = \frac{p'' - p}{p' - p}$$

WINDISCH Table
% of alcohol

ALCOHOLIC STRENGTH BY VOLUME

Measurement using a pycnometer

WINDISCH TABLES

% alcoholic strength by volume respect to specific gravity at 20° C

Specific gravity	Alcohol % (v/v)	Specific gravity	Alcohol % (v/v)
1,00000	0,00	0,98530	11,00
0,99851	1,00	0,98471	11,50
0,99704	2,00	0,98412	12,00
0,99560	3,00	0,98297	13,00
0,99419	4,00	0,98182	14,00
0,99281	5,00	0,98071	15,00
0,99149	6,00	0,97960	16,00
0,99020	7,00	0,97850	17,00
0,98894	8,00	0,97743	18,00
0,98771	9,00	0,97638	19,00
0,98711	9,50	0,97532	20,00
0,98650	10,00	0,97425	21,00
0,98590	10,50	0,97318	22,00



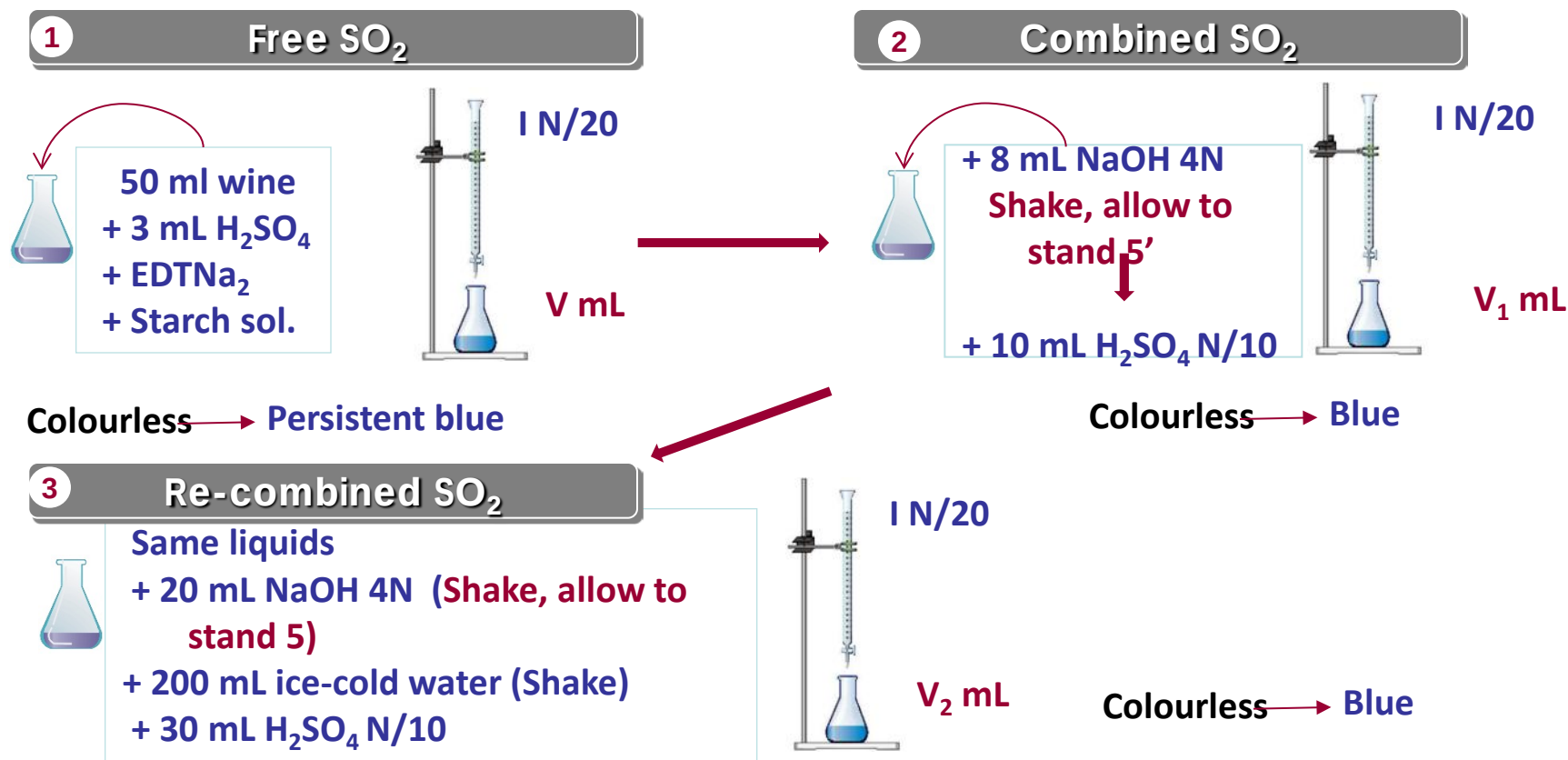
DETERMINATION OF SULPHUR DIOXIDE

Double Ripper titrimetric method

PRINCIPLE Free sulphur dioxide is determined by direct titration with iodine. The combined sulphur dioxide is subsequently determined by iodometric titration after alkaline hydrolysis. When added to the free sulphur dioxide, it gives the total sulphur dioxide.

PROCEDURE

1. Titration of free and combined SO_2



DETERMINACIÓN DE ANHIDRIDO SULFUROSO

Double Ripper titrimetric method

Procedure

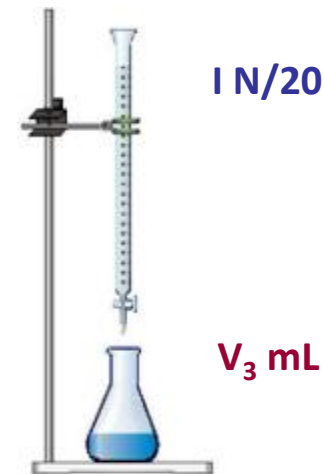
2. Titration of other reducing substances



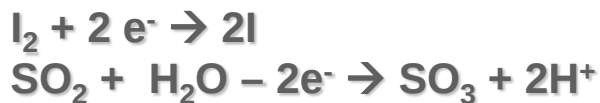
50 mL wine
+ 5 mL ethanal sol.
+ 30 mL H₂SO₄ N/10

Stopper the flask
Allow to stand 30'

+ 30 mL H₂SO₄ N/10
+ Starch solution



Redox reaction



Colourless → Blue

Expression of results

Free SO₂ : V - V₃

Combined SO₂ : V₁ + V₂

Total SO₂ : (V + V₁ + V₂) - V₃

Free sulphur dioxide (mg/L) = 32 (V - V₃)

Total sulphur dioxide (mg/L) = 32 ((V + V₁ + V₂)) - V₃

Expressed in:
Free or combined mg SO₂ /L Wine



DETERMINATION OF TOTAL POLIPHENOLS

Folin-Ciocalteu Index

PRINCIPLE.- All phenolic compounds contained in wine are oxidized by Folin-Ciocalteu reagent (phosphotungstic and phosphomolybdic acids) which is reduced to a mixture of blue oxides of tungsten and molybdenum. The blue colouration produced has a maximum absorption in the region of 750nm and is proportional to the total quantity of phenolic compounds.

Procedure

Sample

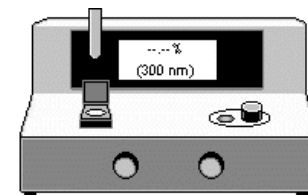


1 mL Diluted red wine 1/5
+ 5 mL Folin reagent
+ 20 mL CaCO_3

*Bring to 100
mL dist. H_2O .*



*Shake
Allow to
stand 30'*



**Absorbance at
750 nm**

Blank



1 mL Distilled H_2O
+ 5 mL Folin reagent
+ 20 mL CaCO_3

*Bring to 100
mL dist H_2O .*



*Shake
Allow to
stand 30'*



Expression of results

$$F I = (\text{Abs sample} - \text{Abs blank}) \times 100$$



LINKS TO VIDEOS OF INTERES IN THE PHYSICO-CHEMICAL ANALYSIS OF WINES

- Measuring pH for Wines
<https://www.youtube.com/watch?v=Uspu7qqT5dw>
- Measuring Brix for Wines
<https://www.youtube.com/watch?v=ZnB35bks4o8>
- Measuring Titratable Acidity in Wine
<https://www.youtube.com/watch?v=rRLvOvih2-8>
- Measuring Volatile acidity in wines
https://www.youtube.com/watch?v=eJdq9jkdZ_0
- Measuring alcoholic Strength in wines
<https://www.youtube.com/watch?v=upBaDKAzXJg>
- Measuring of Dioxide sulphur in wines
<https://www.youtube.com/watch?v=vzrnwPYjYzg>

