

Analysis of wine

Traces of copper, lead, iron and zinc could be present in wine as pollutants originated from usual treatments of grapes and wine. I. e. sulphur dioxide is added in order to control fermentation.

Although official methods for the trace analysis are based on Atomic spectroscopy, Voltammetry allows an equivalent and low cost trace analysis of metals in wine, with the same performances.

Voltammetry necessitate only a little nitrogen gas cylinder for the deaeration of the solutions and usual glassware and reagents; free metals can be analysed on the plain sample, while total metals have to be analysed after a simple acidic treatment.

All the procedures are simple and are reported on the operator manual. In the next pages the sheets of the following analysis of wine are reported:

- Cu, Pb, Cd e Zn by means of DPASV
- Fe by means of DPV
- Sulphur dioxide by means of DPV

Analysis of copper, lead, zinc and cadmium

Method for total metals.

Analyse free metals directly on untreated wine.

Accurate method

Pour 10 - 25 ml of wine in a 50 ml volumetric flask, add 0.8 ml of 65% HNO₃. Heat at 50°C for 2 hour. Cool and bring to volume with 0.2 M HCl.

Rapid method

Pour 10 - 25 ml of wine in a 50 ml volumetric flask and bring to volume with 2 M HCl.

Voltammetric analysis

Pour 10 ml of solution in the cell and analyse, in sequence Pb and Cd and after Cu. Add NH₃ until pH between 3 and 6 and, finally analyse zinc.

Blank analysis

Measure the concentration of Pb, Cd and Zn in the reagent used for he sample treatment. Use the direct subtraction method software. Copper is usually absent in the above reagents.

Analysis of Iron

Analyse iron directly, on the plain sample, without any treatment. The solution has to be well deaerate (at least for 10 minutes) in order to eliminate completely the oxygen because the discharge potential of oxygen coincides with the iron one.

Voltammetric analysis

Pour 10 ml of supporting electrolyte in the cell, add 2 – 5 ml of sample. Deaerate accurately. Repeat scanning until the peaks are reproducible (meaning the oxygen is completely eliminated).

Blank analysis

Measure the concentration of Fe in the supporting electrolyte. Use the direct subtraction method software..

Analysis of sulphur dioxide

Analyse free sulphur dioxide on the plain sample or the total amount on the sample treated with KOH. Anyway, analyse the sample as soon as possible, after the opening of the bottle of sample.

Total sulphur dioxide

Pour 10 ml of sample in 2 ml of 3 M KOH (or NaOH), gently, while the tip of the pipette is dipped in KOH solution.

Voltammetric analysis

Pour 10 ml of supporting electrolyte in the cell. Deaerate accurately (because oxygen could oxidise sulphur dioxide). Add 0.1 – 5 ml of plain wine (for free SO₂ analysis) or treated wine (for total SO₂ analysis).

Blank analysis

Blank analysis is not necessary.

Analysis of lead

Analysis: Barbera

Sample Concentration = 141 $\mu\text{g/l}$

Method: 5 additions

Volumes Table

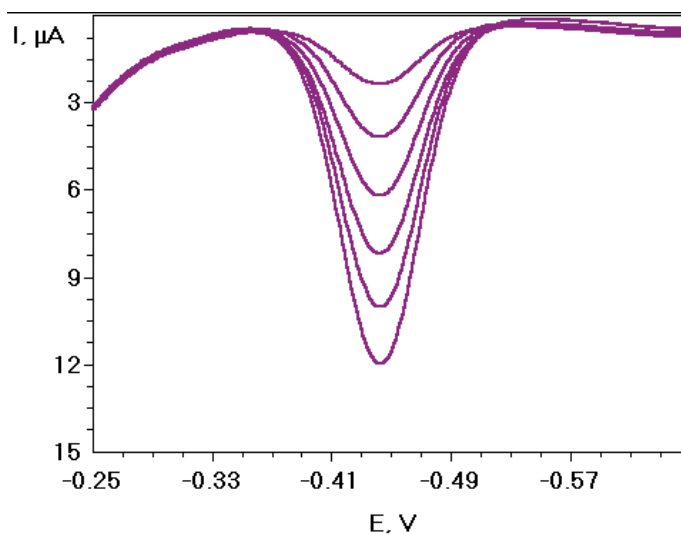
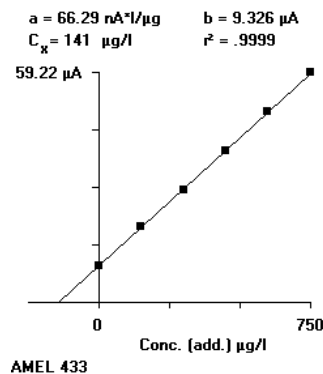
Solvent Volume	0 (ml)
Supporting Sol.	8 (ml)
Sample Volume	2 (ml)
Standard Conc.	10000 ($\mu\text{g/l}$)

Height Table

#	Peak Pot.	Height
0	-442.8	1.884 μA
1	-442	3.859 μA
2	-442.8	5.779 μA
3	-442.8	7.726 μA
4	-443.5	9.697 μA
5	-442.8	11.66 μA

Regression Data

#	Add.Conc.	Height x dilution	
0	0 $\mu\text{g/l}$	9.420 μA	$y = ax + b$
1	150 "	19.35 μA	$a = 66.29 \text{ nA}^*/\mu\text{g}$
2	300 "	29.07 μA	$b = 9.326 \mu\text{A}$
3	450 "	38.98 μA	$r^2 = .9999$
4	600 "	49.07 μA	
5	750 "	59.22 μA	



Analysis of copper

Sample Concentration = 584 $\mu\text{g/l}$

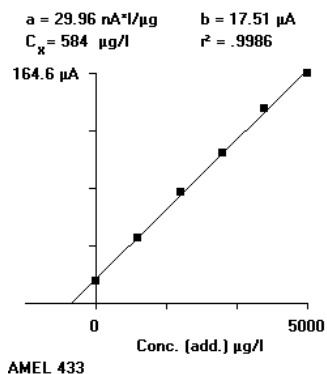
Method: 5 additions

Volumes Table

Solvent Volume	0.15 (ml)
Supporting Sol.	8 (ml)
Sample Volume	2 (ml)
Standard Conc.	10000 ($\mu\text{g/l}$)

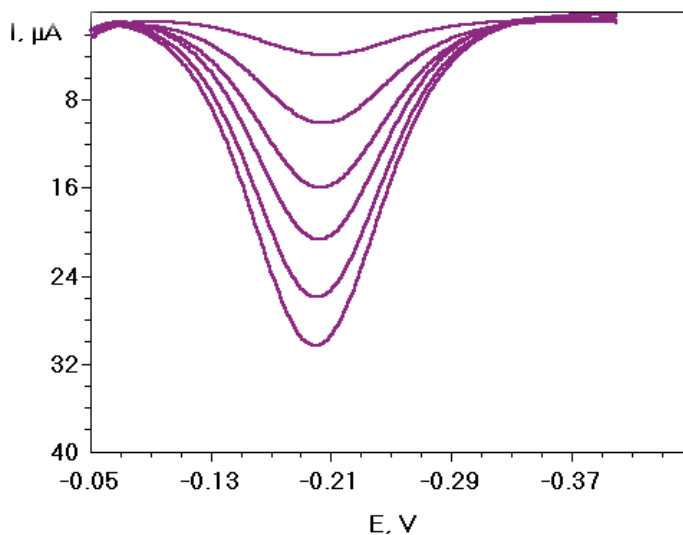
Height Table

#	Peak Pot.	Height
0	-207.3	3.142 μA
1	-205.8	9.061 μA
2	-202.8	15.15 μA
3	-201.9	20.02 μA
4	-200.5	25.46 μA
5	-199.8	29.52 μA



Regression Data

#	Add.Conc.	Height x dilution	
0	0 $\mu\text{g/l}$	15.95 μA	$y = ax + b$
1	1000 "	46.90 μA	$a = 29.96 \text{ nA}^*/\mu\text{g}$
2	2000 "	79.92 μA	$b = 17.51 \mu\text{A}$
3	3000 "	107.6 μA	$r^2 = .9986$
4	4000 "	139.4 μA	
5	5000 "	164.6 μA	



Analysis of zinc

Analysis: Barbera

Sample Concentration = 1 mg/l

Method: 5 additions

Volumes Table

Solvent Volume	2.15 (ml)
Supporting Sol.	8 (ml)
Sample Volume	2 (ml)
Standard Conc.	10 (mg/l)

Height Table

#	Peak Pot.	Height
0	-966	1.952 μA
1	-966	3.663 μA
2	-963.8	5.264 μA
3	-965.3	7.034 μA
4	-963.8	8.709 μA
5	-965.3	10.36 μA

Regression Data

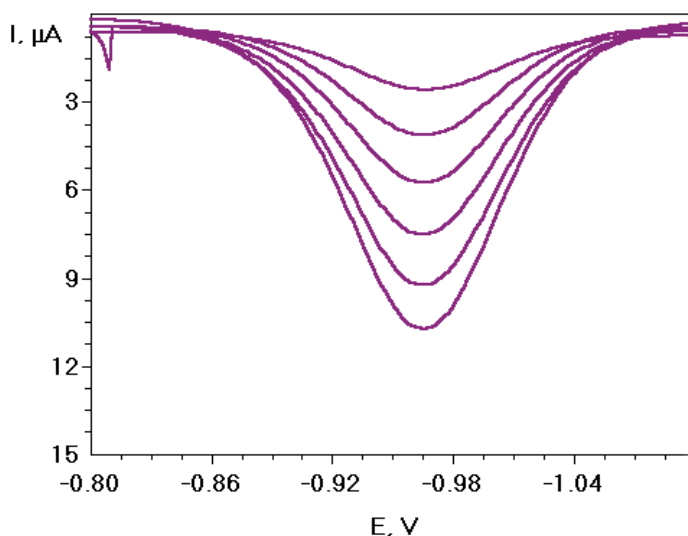
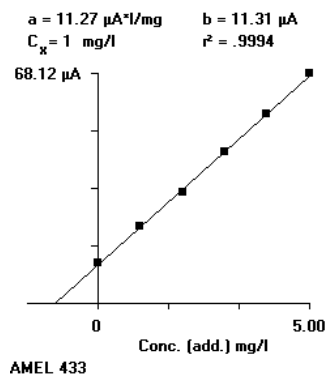
#	Add. Conc.	Height x dilution
0	0 mg/l	11.86 μA
1	1.00 "	22.62 μA
2	2.00 "	33.04 μA
3	3.00 "	44.84 μA
4	4.00 "	56.39 μA
5	5.00 "	68.12 μA

$$y = ax + b$$

$$a = 11.27 \mu\text{A} \cdot \text{l}/\text{mg}$$

$$b = 11.31 \mu\text{A}$$

$$r^2 = .9994$$



Analysis of iron

Analysis: Barbera

Sample Concentration = 1.79 mg/l

Method: 5 additions

Volumes Table

Solvent Volume	0 (ml)
Supporting Sol.	5 (ml)
Sample Volume	5 (ml)
Standard Conc.	10 (mg/l)

Height Table

#	Peak Pot.	Height
0	-928.6	930.0 nA
1	-928.6	1.624 μ A
2	-927.1	2.231 μ A
3	-924.1	2.926 μ A
4	-922	3.379 μ A
5	-922.6	3.783 μ A

Regression Data

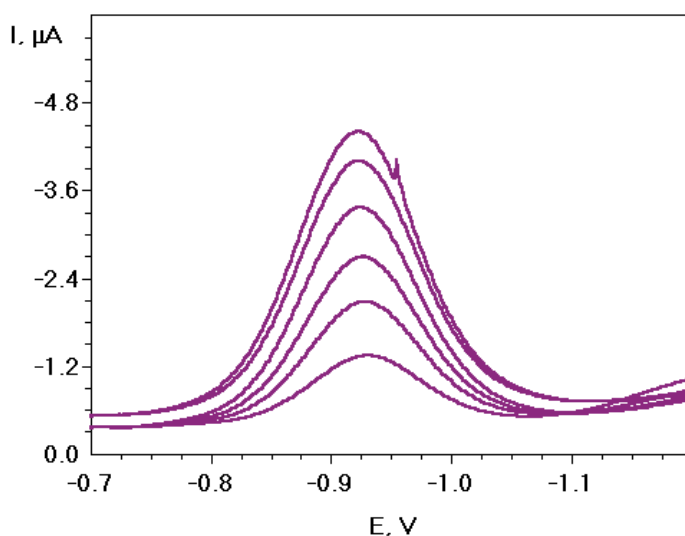
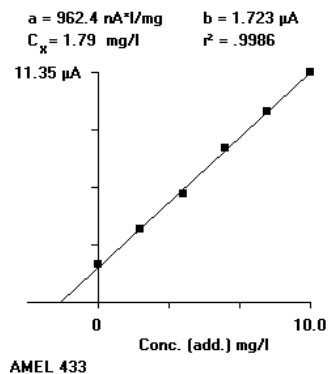
#	Add.Conc.	Height x dilution
0	0 mg/l	1.860 μ A
1	2.00 "	3.573 μ A
2	4.00 "	5.356 μ A
3	6.00 "	7.610 μ A
4	8.00 "	9.462 μ A
5	10.0 "	11.35 μ A

$$y = ax + b$$

$$a = 962.4 \text{ nA} \cdot \text{l/mg}$$

$$b = 1.723 \text{ } \mu\text{A}$$

$$r^2 = .9986$$



Analysis of sulphur dioxide

Analysis: Barbera

Sample Concentration = 164 mg/l

Method: 5 additions

Volumes Table

Solvent Volume	2.6 (ml)
Supporting Sol.	10 (ml)
Sample Volume	0.5 (ml)
Standard Conc.	100 (mg/l)

Height Table

#	Peak Pot.	Height
0	-597	1.100 μA
1	-594	1.612 μA
2	-592.5	2.185 μA
3	-592.5	2.684 μA
4	-589.5	3.119 μA
5	-588.6	3.643 μA

Regression Data

#	Add. Conc.	Height x dilution
0	0 mg/l	28.83 μA
1	100 "	43.86 μA
2	200 "	61.62 μA
3	300 "	78.38 μA
4	400 "	94.22 μA
5	500 "	113.7 μA

$$y = ax + b$$

$$a = 169.2 \text{ nA} \cdot \text{l/mg}$$

$$b = 27.81 \mu\text{A}$$

$$r^2 = .9989$$

