Formaldehyde

Method: Hydrazine in 0.1 M citrate buffer, pH 6.5
Technique: Differential Pulse Voltammetry (DPV/a)

Start Potential (mV)	-600
End Potential (mV)	-1300
Current range	2.048 µA
Scan Speed (mV/s)	3
Number of cycles	5
Delay before sweep (s)	10
Purge and stir time (s)	300
Stirring speed (rpm)	60
Drop Size (a.u.)	60

Formaldehyde concentrated standard solution (1 g/l)
Dilute 2.74 ml of 36.5 % Formaldehyde in 1 l of distilled water in a volumetric flask.
Prepare fresh solution at the moment of analysis. Titrate the solution as described in the next page.

Supporting Electrolyte
1- 1 M ammonium citrate buffer at pH 6.5. Dissolve 21 g of mono hydrate citric acid in 80 of distilled water. Adjust pH to 6.5 by using conc. NH_3. Bring to the mark in a 100 ml volumetric flask with distilled water.
2- 1% Hydrazine sulphate aqueous solution. Prepare fresh solution at the moment of analysis

Procedure
Pour 10 ml of sample in the cell, add 1 ml of ammonium citrate buffer. Deaerate for 10 min. Add 1 ml of hydrazine solution.

Working standard solution (10 mg/l)
Dilute the concentrated standard solution 1+99 in, at the moment of the analysis.

Interference
High concentration of other aldehydes can overlap the peak of the formaldehyde.
Zinc can overlap the formaldehyde peak: use clean reagents.

Alternative supporting electrolytes
0.1 M ammonium phosphate buffer, pH 6.5 – pay attention to the presence f zinc in this reagent !
0.36 M sulphuric acid – lower sensitivity and worse base line can be performed with this reagent.

Analysis of formaldehyde in the air

Sample the air in a bubbler filled with in 25 ml of 0.1 M citrate buffer solution for 1 – 2 hour using a pump, with a flow of 1 ml/ min.
Pour the solution in a 25 ml volumetric flask and bring to volume with a 0.1 M citrate buffer solution.
Use 10 ml of his solution for the voltammetric analysis.

Titration of the concentrated standard solution of formaldehyde

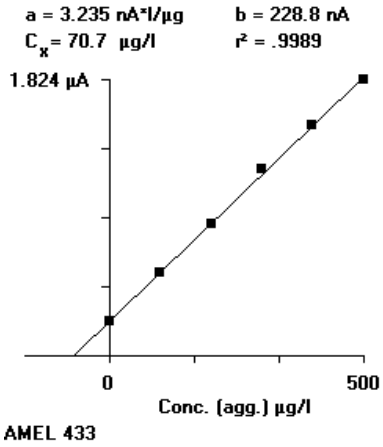
Pour 5 ml of fresh 1.13 M Na_2SO_3 solution (142.3 g/l), in a 50 ml beaker.
Adjust pH between 8.5 and 10 with NaOH or HCl.
Record the final pH.
Add 10 ml of concentrated standard solution of formaldehyde. The pH should be greater than 11.
Titrate the solution back to the pH recorded before with 0.02 N H_2SO_4 (about 17 ml are needed).
If pH is overrun, back titrate again with 0.01 M NaOH.
Calculate the concentration of the solution:

$$C\ (mg/ml) = \frac{30 \cdot (N_{H_2SO_4} \cdot V_{H_2SO_4} - N_{NaOH} \cdot V_{NaOH})}{V_{conc.std.sol.}}$$

$$= 3 \cdot (N_{H_2SO_4} \cdot V_{H_2SO_4} - N_{NaOH} \cdot V_{NaOH})$$

Analytical report

Analysis: Air of the lab
Sample (solution) Concentration = 71 µg/l
Sample (air) Concentration = 33 µg/m^3
Method: 5 addition and blank subtraction



Volumes Table

Solvent Volume	0 (ml)
Supporting Sol.	1 (ml)
Sample Volume	10 (ml)
Standard Conc.	10000 (µg/l)

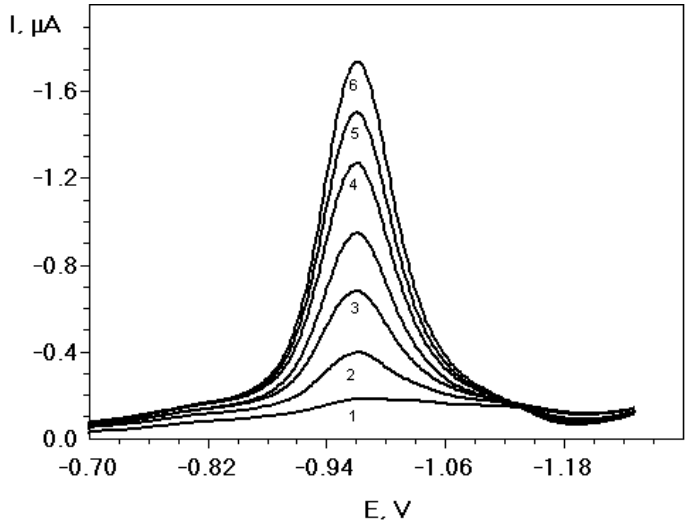
Height Table

#	Peak Pot.	Height
0	-968.6	204.1 nA
1	-968.6	489.5 nA
2	-970	776.3 nA
3	-968.6	1.091 µA
4	-968.6	1.342 µA
5	-970.9	1.586 µA

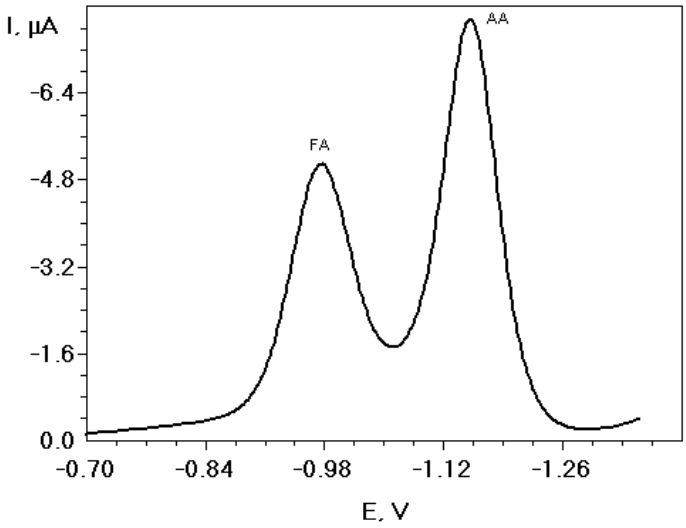
Regression Data

#	Add.Conc.	Height x dilution
0	0 µg/l	224.5 nA
1	100 "	543.4 nA
2	200 "	869.5 nA
3	300 "	1.234 µA
4	400 "	1.530 µA
5	500 "	1.824 µA

y = ax + b
a = 3.235 nA*l/µg
b = 228.8 nA
r^2= .9989



1= Blank; 2= Sample; 3, 4, 5, 6, 7 = standard sol. additions



The procedure can be used also for the analysis of the other total aldehydes, even if formaldehyde has to be present at low concentration.
FA = formaldehyde; AA= Acetaldehyde (total aldehydes)