Chromium - Hexavalent and total Chromium

Method: DTPA in acetate buffer, pH 6.2
Function: Differential Pulse Adsorptive Stripping Voltammetry (DPS/a)

Start Potential (mV)	-1000
End Potential (mV)	-1400
Current range	20,48 μA
Scan Speed (mV/s)	10
Deposition time (s)	30
Deposition Pot. (mV)	-900
Number of cycles	3
Delay before sweep (s)	5
Purge and stir time (s)	300
Stirring speed (rpm)	300
Drop Size (a.u.)	60

Chromium (VI) concentrate standard solution (1 g/l)
Dissolve 2.828g of pure and dried $K_2Cr_2O_7$ in 1 l of 1% HNO_3, in a volumetric flask.
($MM_{K_2Cr_2O_7} = 294.19$; $MM_{Cr} = 52.00$)

Supporting electrolyte
Solution of 0.05 M DTPA (diethylentriammino pentaacetic acid), 0.2 M CH_3COONa, 2.5 M $NaNO_3$ at pH 6.2.
Dissolve 1.96 g of DTPA, 1.64 g of CH_3COONa and 21.3 g of $NaNO_3$ in 80 ml of distilled water, adjust the pH to 6.2 using 30% NaOH solution.
Bring to volume in a 100ml volumetric flask with distilled water.

Procedure
Analysis of Cr (VI).
Add 2 ml of supporting electrolyte to 10 ml of neutralised sample. Deaerate for 10 minutes and start the analysis.

Analysis of total Cr
Deaerate 10 ml of neutralised sample for 10 minutes, then add 2 ml of deaerated supporting electrolyte. Start the analysis immediately.

Treatment for the removal of low level of organic substances in natural water
Add 20 µl of 30% H_2O_2 to 10 ml of neutral sample irradiate for 1 hour with a 150 W UV lamp, in a closed system to avoid sample evaporation.
Then add 0.1 ml of 0.5 M NaOH and 0.5 ml of bromine water diluted 1+100. Heat at 60°C for15 minutes.
Cool and add 0.1 ml of 0.5M H_2SO_4.
Finally, add 2 ml of supporting electrolyte.

Waste water, biological samples and solid samples
Opportunely digest or dissolve the sample with an oxidising treatment and dissolve the residue with distilled water. Neutralise, bring to a volume of 10 ml with distilled water and add 2 ml of supporting electrolyte.

Working standard solution (0.1 mg/l)
Dilute 0.1 ml of Cr (VI) concentrated standard solution in 1 l of distilled water, in a volumetric flask. Prepare this solution at the moment of the analysis.

Warnings

Also Cr (III) give a peak in the analysis conditions, but its height decrease in few minutes, after the addition of the supporting electrolyte.

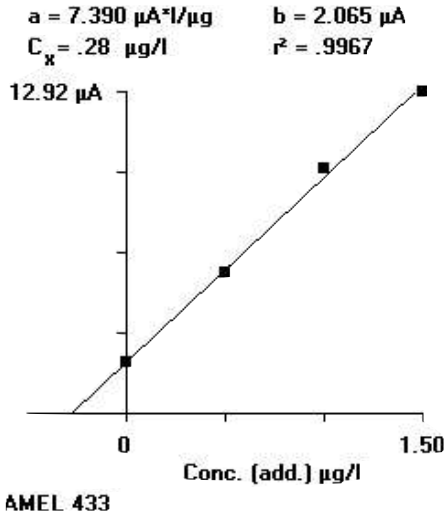
Surfactants and organic substances reduce the peak heights, than the latter must be removed using a treatment with H_2O_2. In this way only total Cr can be analysed.

The oxidation with H_2O_2 has to be made at neutral pH to avoid loss of volatile forms of chromium.

Bromine water is used for the oxidation of Cr(III) to Cr(VI) because this substance give no interference with the analysis.

Analytical report

Analysis: tap water - Chromium VI
Sample Concentration = 0.28 µg/l
Method: 3 additions



Volumes Table

Solvent Volume	0 (ml)
Supporting Sol.	2 (ml)
Sample Volume	10 (ml)
Standard Conc.	100 (µg/l)

Heights Table

#	Peak Pot.	Height
0	-1208.5	1.675 µA
1	-1219.5	4.683 µA
2	-1230.5	8.141 µA
3	-1245.6	10.63 µA

Regression Data

#	Add.Conc.	Height x dilution
0	0 µg/l	2.010 µA
1	.5	5.643 µA
2	1	9.852 µA
3	1.50 "	12.92 µA

y = ax + b
a = 7.390 µA*l/µg
b = 2.065 µA
r^2 = .9967