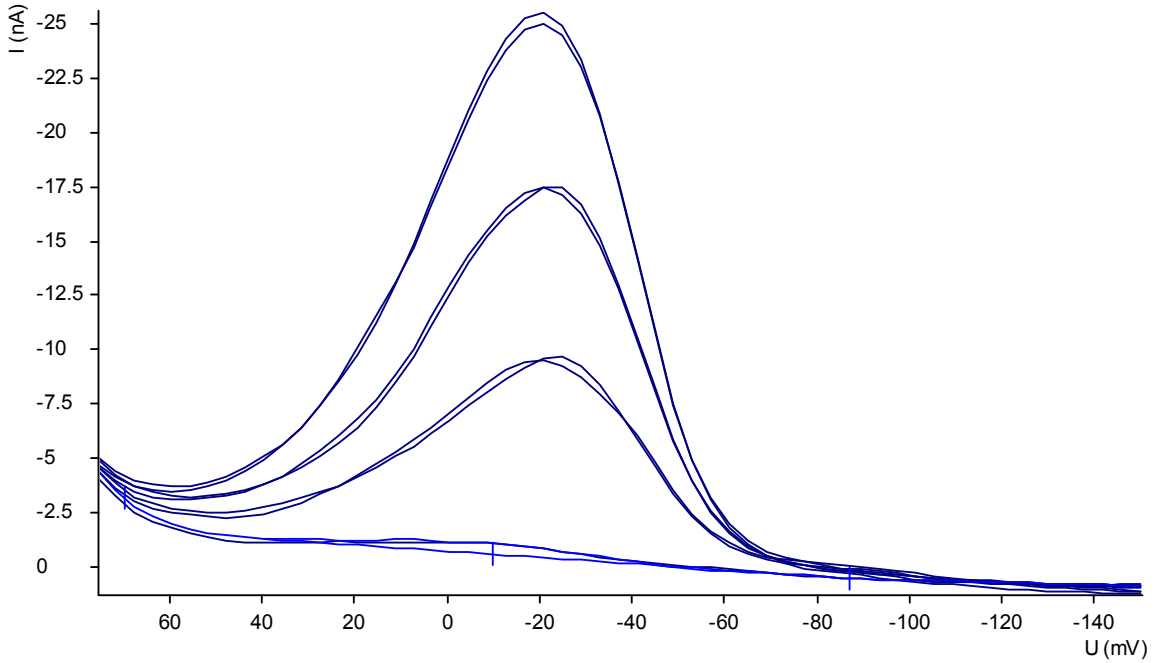
VA Application Note V–110

Total chromium in waste water after UV digestion (polarographic method with ethylene diamine)



Cr(VI) is determined by polarography at the SMDE in acetate solution containing ethylene diamine to mask interfering copper ions.

Only Cr(VI) is electrochemically active. Therefore, all chromium has to be oxidized to Cr(VI) prior to analysis. UV irradiation at pH > 4 is used to oxidize chromium.

Results

Cr(total) in waste water
8.2 µg/L

Metrohm

Method description

Sample

Waste water

Instruments

797 VA Computrace & 909 UV Digester

Sample preparation

For UV digestion 10 mL waste water, 20 µL HCl, and 50 µL H_2O_2 are pipetted into the 12 mL quartz sample vessels. The sample holder with the 12 quartz sample vessels is placed in the 909 UV Digester. The samples are irradiated at 90 °C for 60 min. To oxidize Cr(III) to Cr(VI), the pH of the solutions is adjusted to 4–6 with NaOH and 50 µL H_2O_2 are added. The samples are irradiated at 90 °C for 20 min.

Parameters 909 UV Digester

Temperature	90 °C
Irradiation time (UV digestion)	60 min
Irradiation time (Oxidation)	20 min

Electrodes

Multi-Mode Electrode pro Silanized capillaries	6.1246.120 6.1226.050
Ag/AgCl/KCl (3 mol/L) reference electrode. Bridge electrolyte c(KCl) = 3 mol/L	6.0728.020 6.1245.010
Separate Pt rod electrode	6.0343.000

Reagents

HCl	Hydrochloric acid, for trace analysis*, w(HCl) = 30%
H_2O_2	Hydrogen peroxide solution, for trace analysis*, w(H_2O_2) = 30%
NaOH	Sodium hydroxide solution, for trace analysis*, w(NaOH) = 30%
$H_2N(CH_2)_2NH_2$	Ethylene diamine, 99.5%
NH_3	Ammonia solution, for trace analysis*, w(NH_3) = 25%
CH_3COOH	Acetic acid, for trace analysis*

*e.g., Merck suprapur®, Sigma-Aldrich TraceSelect® or equivalent.

Analysis

Measuring solution	5 mL digested, oxidized waste water + 15 mL ultrapure water + 10 µL ethylene diamine + 200 µL NH_3 + 150 µL acetic acid → pH adjusted to 6.8 ± 0.1 with NaOH

Parameters 797 VA Computrace

Working electrode	SMDE
Stirrer speed	2000 rpm
Mode	DP
Purge time	600 s
Equilibration time	10 s
Start potential	0.1 V
End potential	-0.15 V
Pulse amplitude	0.05 V
Voltage step	0.004 V
Voltage step time	0.4 s
Sweep rate	0.01 V/s
Peak potential Cr(VI)	-0.02 V