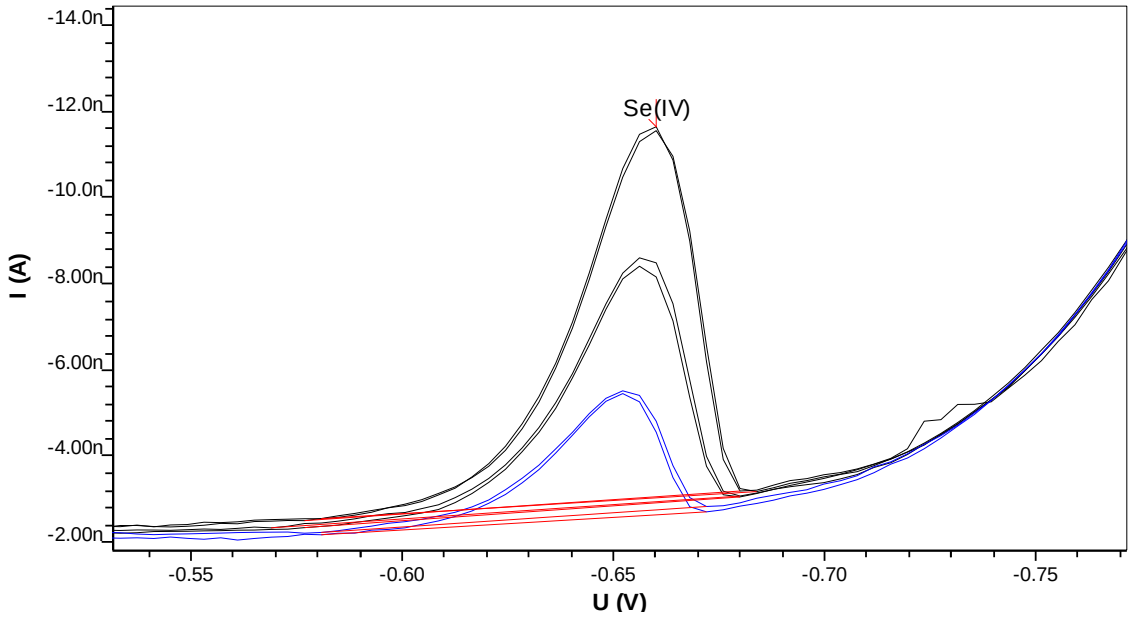
VA Application Note V–109

Selenium in waste water after UV digestion



Selenium is determined by cathodic stripping voltammetry (CSV) at the hanging mercury drop electrode (HMDE). Se(IV) is deposited on the surface of the mercury drop in sulfuric acid electrolyte under addition of copper ions as Cu_xSe_y.

Waste water samples containing organic contaminants have to be digested by UV irradiation before analysis. In addition, the sample has to undergo a second irradiation step at pH 7–9 to reduce Se(VI) to Se(IV), since only Se(IV) is electrochemically active.

Results

Se in waste water
9.8 µg/L

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Method description

Sample

Waste water

Instruments

797 VA Computrace & 909 UV Digester

Sample preparation

For UV digestion 10 mL waste water sample, 10 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 reduce Se(VI) to Se(IV) the pH of the solution is adjusted to 7–9 with NaOH. The samples are irradiated at 90 °C for 60 min.

Parameters 909 UV Digester

Temperature	90 °C
Irradiation time (UV digestion)	60 min
Irradiation time (reduction)	60 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%
$(NH_4)_2SO_4$	Ammonium sulfate, 99.9999%
Cu standard stock solution	ß(Cu(II)) = 1 g/L
H_2SO_4	Sulfuric acid, for trace analysis*, ≥ 95%
EDTA	Ethylenediaminetetraacetic acid disodium salt dihydrate, 99%

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

Solutions

EDTA solution	c(EDTA) = 0.1 mol/L

Analysis

Measuring solution	1 mL digested sample + 9 mL ultrapure water + 3.3 g $(NH_4)_2SO_4$ + 0.1 mL Cu standard stock solution + 1 mL EDTA solution → pH adjusted to 2.2 with H_2SO_4

Parameters 797 VA Computrace

Working electrode	HMDE
Stirrer speed	2000 rpm
Mode	DP
Purge time	300 s
Deposition potential	-0.4 V
Deposition time	30 s
Equilibration time	10 s
Start potential	-0.45 V
End potential	-0.85 V
Pulse amplitude	0.05 V
Pulse time	0.04 s
Voltage step	0.004 V
Voltage step time	0.1 s
Sweep rate	0.04 V/s
Peak potential Se(IV)	-0.65 V

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