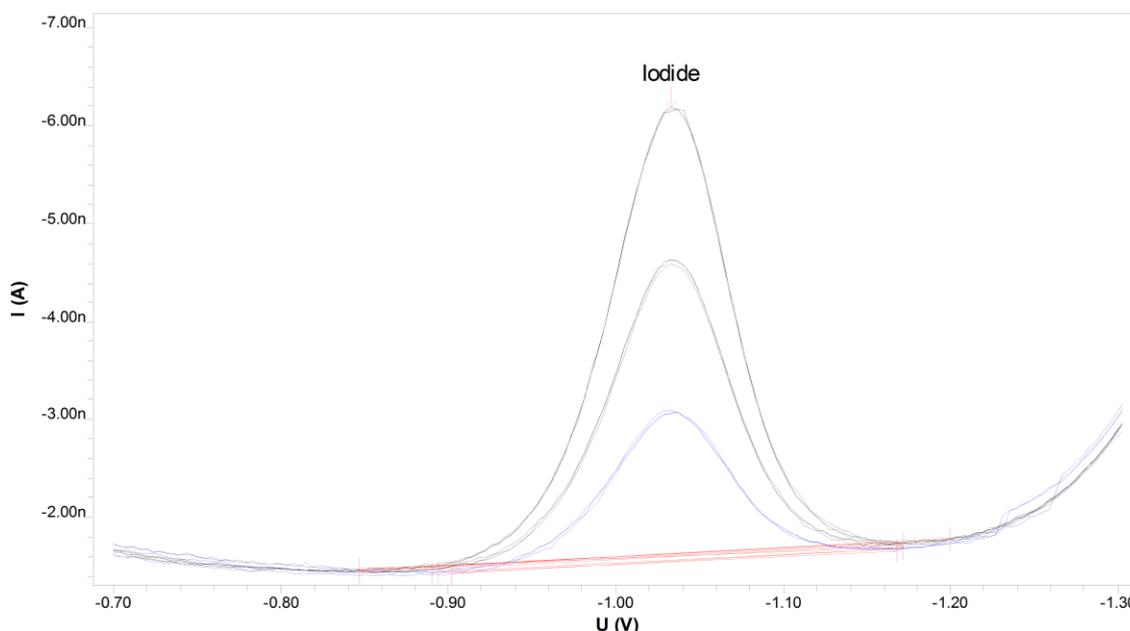


Iodide in NaCl brine electrolytes in chlor-alkali electrolysis plants



The membrane process, the best available technology in chlor-alkali electrolysis, requires highly purified sodium chloride brine electrolytes. One of the most important parameters to be monitored is the concentration of iodide. In the electrolysis cell, iodide is oxidized to iodate, which precipitates in the ion-exchange membrane and decreases the membrane's useful lifetime. The described voltammetric method is straightforward and sensitive for the trace determination of iodide in brine. The method involves the oxidation of iodide to iodate and the subsequent determination of the iodate by differential pulse voltammetry. The limit of detection is approximately 2.5 µg/L iodide.

Results

Iodide in brine

40.1 µg/L

Method description

Sample

- Sodium chloride brine $\beta(\text{NaCl}) = 300 \text{ g/L}$

Instrument

797 VA Computrace



Electrodes

Working electrode (WE)	6.1246.120 MME (Multi-Mode Electrode pro) with 6.1226.030 glass capillary
Reference electrode (RE)	6.0728.020 reference electrode (Ag/AgCl/ c(KCl) = 3 mol/L) with 6.1245.010 electrolyte vessel filled with intermediate electrolyte c(KCl) = 3 mol/L
Auxiliary electrode (AE)	6.0343.000 platinum electrode

Reagents

NaOH	Sodium hydroxide solution, suprapur, $w(\text{NaOH}) = 30 \%$
NaOCl	Sodium hypochlorite solution, $w(\text{NaOCl}) = \sim 10 \%$
KIO ₃	Potassium iodate, analytical grade
Iodate standard stock solution	$\beta(\text{I}^{+V}) = 1 \text{ g/L}$ in water
Na ₂ SO ₃	Sodium sulfite anhydrous
EDTA	Ethylenediaminetetraacetic acid (EDTA)

Solutions

Na ₂ SO ₃ solution	$w(\text{Na}_2\text{SO}_3) = 10\%$
EDTA solution	$c(\text{EDTA}) = 0.5 \text{ mol/L}$, adjusted to pH 11 with NaOH
Iodate standard stock solution	$\beta(\text{KIO}_3) = 1686 \text{ mg/L}$ in water ($\approx 1000 \text{ mg/L}$ iodide)
Iodate standard addition solution	$\beta(\text{KIO}_3) = 84.3 \text{ mg/L}$ in water ($\approx 50 \text{ mg/L}$ iodide)

Analysis

Measuring solution	10 mL sample + 20 μL NaOH to adjust pH 11.0 ... 11.5 + 50 μL NaOCl
	Mix, wait 20 min
	+ 1 mL Na ₂ SO ₃ solution + 0.1 mL EDTA solution

Parameters

Working electrode	HMDE
Drop size	9
Stirrer speed	2000 rpm
Mode	DP
Initial purge time	300 s
Addition purge time	10 s
Deposition potential	0 V
Deposition time	0 s
Equilibration time	5 s
Pulse amplitude	0.05 V
Start potential	-0.7 V
End potential	-1.3 V
Voltage step	0.004 V
Voltage step time	0.3 s
Sweep rate	0.13 V/s
Peak potential iodate	-1.03 V

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