VA Application Note No. V - 123

Title:	**Determination of total iron in ethylene glycol (2,3-dihydroxy-naphthalene method)**

Summary:	The concentration of Fe(total) is determined in monoethylene glycol by adsorptive stripping voltammetry with 2,3-dihydroxy-naphthalene as complexing agent. The detection limit of the method is approx. 0.1 µg/L with respect to the content in the measuring vessel. If no bromate is added to the supporting electrolyte the sensitivity of the method is about 10 times lower. All reagents have to be added in the order as listed below. Fe(II) and Fe(III) give signals with the same sensitivity. All reagents typically contain iron impurities, especially the 2,3-dihydroxy-naphthalene. Therefore a subtraction of the reagent blank is recommended.

Sample:	Monoethylene glycol (content approx. 75%)
Sample preparation:	none

Analysis of total Fe

HEPPS buffer	c(HEPPS) = 0.5 mol/L c(NaOH) = 0.25 mol/L *HEPPS: 4-(2-Hydroxyethyl)-piperazine-1-propane-sulfonic acid*
DHN solution	c(DHN) = 0.02 mol/L *DHN: 2,3-Dihydroxy-naphthalene*
Bromate solution	c($KBrO_3$) = 0.4 mol/L
Measuring solution	10 mL H_2O + 10 µL DHN solution + 200 µL HEPPS buffer + 0.5 mL bromate solution + 10 µL ethylene glycol sample → pH adjusted to 8.0 ± 0.1
Working electrode (WE)	**MME** (Multi Mode Electrode) 6.1246.020
Auxiliary electrode (AE)	**Pt** 6.0343.000
Reference electrode (RE)	Ag/AgCl/KCl (3 mol/L): 6.0728.020 + 6.1245.010

Parameters

Working electrode	HMDE
Stirrer speed	2000 rpm
Mode	DP

	Purge time	300 s
	Deposition potential	-0.1
	Deposition time	30 s
	Equilibration time	10 s
	Pulse amplitude	50 mV
	Start potential	-0.1 V
	End potential	-0.8 V
	Voltage step	4 mV
	Voltage step time	0.1 s
	Sweep rate	40 mV/s
	Peak potential Fe	-530 mV

Results:	Fe(total) (blank subtracted)
	3.4 mg/L

Determination of total Fe