VA Application Note No. V - 103

Title:	Chromium in lime ($CaCO_3$)

Summary:	Cr(VI) is determined at the HMDE in an electrolyte containing ethylenediamine and acetate. Because Cr(III) is electrochemically inactive, all Cr has to be oxidised prior to analysis.

Sample:	lime ($CaCO_3$)
Sample preparation:	2 g lime are dissolved in 7 mL of w(HNO_3) = 65%, suprapur and filled up to 100 mL with ultrapure water. 1 mL lime solution + 10 mL water + 1 drop c($KMnO_4$) = 0.02 mol/L. The mixture is boiled for 10 min. $KMnO_4$ solution has to be added to keep the pink color. The volume has also kept constant at approx. 10 mL by adding water. Before cooling to room temperature the pH is adjusted to 7-9 with NaOH.

Analysis of Cr(VI)

Electrolyte	
Measuring solution	10 mL oxidised sample solution + 10 µL ethylene diamine + 150 µL w(acetic acid) = 100%, suprapur + 200 µL w(NH_3) = 25%, suprapur adjust pH to 6.8 with acetic acid
Auxiliary electrode (AE)	Pt
Reference electrode (RE)	Ag/AgCl/KCl (3 mol/L)

Parameters		
	Working electrode	HMDE
	Stirrer speed	2000 rpm
	Mode	DP
	Purge time	600 s
	Deposition potential	no deposition
	Deposition time	0 s
	Equilibration time	10 s
	Pulse amplitude	50 mV
	Start potential	70 mV
	End potential	-170 mV
	Voltage step	6 mV
	Voltage step time	0.2 mV
	Sweep rate	30 mV/s
	Peak potential Cr(VI)	-30 mV

Results:	Cr(VI)
	5.5 µg/g

Determination of Cr(VI)