VA Application Note No. V - 54

Title:	**Palladium in pharmaceutical products**

Summary:	The concentration of Pd in pharmaceutical products is determined by polarography after wet digestion.

Sample:	Drug against high blood pressure
Sample preparation:	**Wet digestion** 1 g sample and 7 mL concentrated H_2SO_4 are heated in Kjeldahl flask over a Bunsen burner flame until the solution turns black. In total 17 mL H_2O_2 are added in portions of approx. 1 mL. After each addition it is heated until the solution turns brown again. The digestion is complete when the solution stays clear and colorless when heated until acid vapors are formed. At the end the H_2SO_4 is evaporated nearly to dryness. The residue is diluted with 10 mL H_2O and rinsed quantitatively in the measuring vessel with another 10 mL H_2O.

Analysis of Pd

Ammonia buffer pH 9.6	$c(NH_3)$ = 2 mol/L $c(NH_4Cl)$ = 1 mol/L	
NaOH solution	c(NaOH) = 2 mol/L	
Measuring solution	10 mL digested sample solution + 10 mL H2O + 2 mL ammonia buffer pH 9.6 pH 7 adjusted with NaOH solution	
Working electrode (WE)	**MME** (Multi Mode Electrode)	6.1246.020
Auxiliary electrode (AE)	**Pt**	6.0343.000
Reference electrode (RE)	Reference system: Ag/AgCl/KCl (3 mol/L) Intermediate electrolyte: c(KCl) = 3 mol/L	6.0728.020 6.1245.010

Parameters

Working electrode	DME
Stirrer speed	2000 rpm
Mode	DP
Purge time	300 s
Equilibration time	10 s
Pulse amplitude	0.05 V
Start potential	-0.4 V
End potential	-1.0 V

	Voltage step	0.006 V
	Voltage step time	0.6 s
	Sweep rate	0.01 V/s
	Peak potential Pd	-0.77 V

Results:	Pd
	50 µg/g

Determination of Pd