

Application Bulletin 97/3 e

Determination of tocopherols (vitamin E) in edible oils and fats by anodic stripping voltammetry at a glassy carbon RDE

Summary

Edible oils and fats contain natural tocopherols and, in some cases, also synthetic tocopherols added as antioxidants. The method described below allows the simple and rapid determination of the tocopherol content by voltammetry. The tocopherols are oxidized electrochemically at the glassy carbon electrode. The limit of quantitation is approximately 5 ppm (mg/kg) tocopherol.

Instruments

VA instrument
capable of operating a rotating disk electrode and supporting differential pulse (DP) measuring mode

Electrodes

WE	Glassy carbon electrode tip	6.1204.600
	Driving axle for RDE	6.1204.x10
RE	Ag/AgCl reference electrode	6.0728.x20
	Ag/AgCl/LiCl (sat. in ethanol)	
	Electrolyte vessel	6.1245.010
	Filled with c(LiCl) = sat. in ethanol	
AE	Glassy Carbon rod	6.1247.000
	Electrode holder	6.1241.x20

Reagents

All of the used reagents must be of purest quality possible (for analysis or for trace analysis*).

- Sulfuric acid, $w(\text{H}_2\text{SO}_4) = 96\%$, for analysis, 7664-93-9
- DL- α -Tocopherol, (Vitamin E), for analysis, CAS 10191-41-0
- Ethanol absolute, for analysis, CAS 64-17-5
- Toluene, for analysis, CAS 108-88-3
- Ultrapure water, resistivity $>18 \text{ M}\Omega\cdot\text{cm}$ (25 °C), type I grade (ASTM D1193)

Solutions

Electrolyte	$c(\text{H}_2\text{SO}_4) = 0.1 \text{ mol/L}$ in ethanol/toluen
	Mix 5.9 g $w(\text{H}_2\text{SO}_4) = 96\%$ with
	500 mL ethanol/toluene 2:1.

Standard solution

Tocopherol standard stock solution	$\beta(\text{tocopherol}) = 1 \text{ g/L}$
	Approx. 100 mg tocopherol is dissolved in supporting electrolyte and made up to 100 mL. (Due to its high viscosity, it is very difficult to weigh in exactly 100 mg tocopherol. For this reason, the actual quantity taken is determined accurately and the resulting concentration is then used for the calculations.)
	Tocopherol standard solutions with lower concentrations are prepared from the stock solution by dilution with supporting electrolyte.

Sample preparation

In a 50 mL volumetric flask, 1 ... 5 g oil or fat sample is dissolved in supporting electrolyte. The solution is then filled to the mark with supporting electrolyte.

Analysis

Measuring solution

10 mL sample solution

The concentration is determined by standard addition.

Parameters

Voltammetric	
Measuring mode	DP – Differential pulse
Stirring rate	2000 min^{-1}
Potentiostatic pretreatment	

Potential 1	0.55 V
Waiting time 1	15 s
Equilibration time	30 s
Sweep	
Start potential	0.55 V
End potential	0.9 V
Potential step	0.006 V
Potential step time	0.6 s
Sweep rate	0.01 V/s
Pulse amplitude	0.05 V
Substance	
Name	Vitamin E
Characteristic potential	0.7 V

Sample size	1.010 g
β (Tocopherol)	15.9 $\mu\text{g/g}$

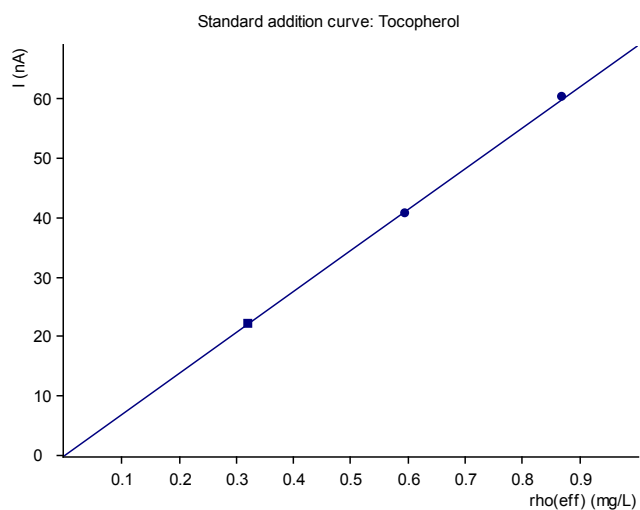
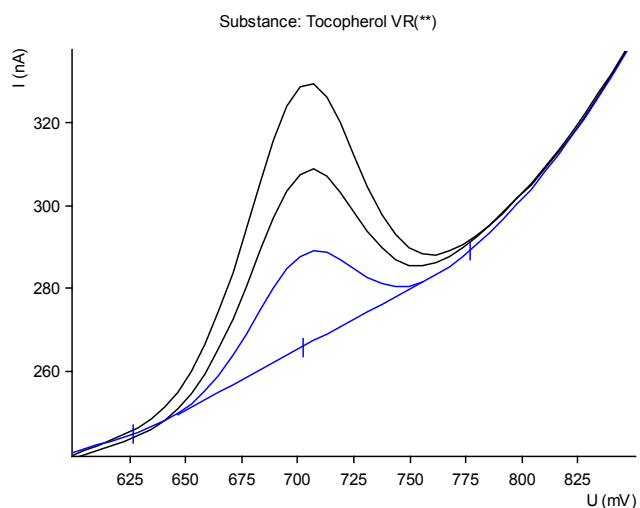
Comments

- After each series of measurements, the electrode is cleaned with aluminum oxide powder.

References

- H. McBride, D. Evans
Rapid voltammetric method for the estimation of tocopherols and antioxidants in oils and fats
Anal. Chem. 45 (1973) 446–449.

Example



Result

Sample	Edible oil
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Appendix

Report of the example determination of tocopherol in edible oil

===== METROHM 746 VA TRACE ANALYZER (5.746.0101) =====							
Determ.	: 06071647	User:		Date:	1999-06-07		
Modified	: 2000-08-23 17:29:34	Run:	0	Time:	16:47:16		
Sample table:	-						
Pos.	Ident.1/S1	Ident.2/S2	Ident.3/S3	Method.call	Sample size/S0		
	VitE	1.0			1.010 g		
Method	: AB097						
Title	: Determination of vitamine E. AB97						
Remark1	: 1 g oil dissolved in electrolyte						
Remark2	: WE:GC-RDE, AE:Pt, RE:LiCl sat. in EtOH						
Substance	: Tocopher					Comments	
Mass conc.	: 320.5 ug/L	Mass	: 3.205 ug			-----	
MC.dev.	: 13.5 ug/L (4.22%)	Add.mass	: 2.75 ug				
Cal.dev.	: -	V0.sample	: 10 mL				
	VR	U/mV	I/nA	I.mean	Std.dev.	I.delta	Comments
	00	703	22.11	22.11			
	10	703	40.55	40.55		18.44	
	20	703	59.98	59.98		19.44	
Substance	Techn.	Y.reg/offset	Slope	Nonlin.	Mean deviat.		
Tocopher	std.add.	2.206e-08	6.883e-05		3.392e-10		
Final results			+/- Res.dev.	%	Comments		
Tocopher =	15.866 ug/g		0.670	4.22			

Method print for the determination of tocopherol

===== METROHM 746 VA TRACE ANALYZER (5.746.0101) =====							
Method:	AB097	.meth	OPERATION SEQUENCE				
Title:	Determination of	vitamine E. AB97					
	Instructions	t/s	Main parameters		Auxiliary parameters		
1	SMPL/M		V.fraction	10.000 mL	V.total	50.0 mL	
2	STIR		Rot.speed	2000 /min			
3	PURGE	60.0					
4	OSTIR						
5	OPURGE	2.0					
6	(ADD						
7	STIR	10.0	Rot.speed	2000 /min			
8	(REP						
9	SEGMENT		Segm.name	asv			
10	REP)0						
11	ADD>M		Soln.name	std	V.add	0.025 mL	
12	ADD)2						
13	END						
Method:	AB097		SEGMENT				
			asv				
	Instructions	t/s	Main parameters		Auxiliary parameters		
1	RDE	2.0	Rot.speed	2000 /min			
2	DPMODE		U.ampl	50 mV	t.meas	20.0 ms	
			t.step	0.60 s	t.pulse	40.0 ms	
3	MEAS	15.0	U.meas	550 mV			
4	OSTIR	30.0					
5	SWEEP	37.2	U.start	550 mV	U.step	6 mV	
			U.end	900 mV	Sweep rate	10 mV/s	
6	OMEAS		U.standby	mV			
7	STIR		Rot.speed	2000 /min			
8	END						