
Application Bulletin

Of interest to: General analytical laboratories; Pharmaceutical industry; Food

B 1, 4, 7

Voltammetric determination of tocopherols (vitamin E) in edible oils and fats

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 (GCE). The limit of quantitation is approximately 5 ppm (mg/kg) tocopherol.

Instruments and accessories

- 746 VA Trace Analyzer with 747 VA Stand or
- 757 VA Computrace

Electrodes:

- Working electrode (WE): rotating disk electrode with glassy carbon electrode tip (GC-RDE):
6.1246.000 drive shaft + 6.1204.110 GC electrode tip + 6.2709.040 stopper
 - Reference electrode (RE): Ag/AgCl/LiCl sat. in ethanol:
6.0728.010 Ag/AgCl + 6.1245.010 electrolyte vessel + 6.2312.000 electrolyte solution LiCl sat. in ethanol
 - Auxiliary electrode (AE): platinum:
6.0343.000 platinum rod electrode
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Reagents

All reagents used should be of the highest purity (p.a. or «suprapur»). Only ultra-pure water should be used.

- Sulfuric acid, $w(\text{H}_2\text{SO}_4) = 96\%$
- DL- α -Tocopherol, CAS 10191-41-0
- Ethanol, abs.
- Toluene, puriss. p.a.

Ready-to-use solutions

- Supporting electrolyte: Mix 5.9 g w(H₂SO₄) = 96% with 500 mL ethanol/toluene 2 : 1.
- Standard solution: $\rho(\text{tocopherol}) = 1 \text{ g/L}$:
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

10 mL sample solution is pipetted into the measuring vessel.

The voltammogram is recorded at the GC-RDE using the following parameters:

working electrode	RDE
reference electrode	Ag/AgCl/LiCl sat. in ethanol
auxiliary electrode	Pt
stirrer speed	2000 rpm
mode	DP
purge time	60 s
deposition time	15 s
deposition potential	550 mV
equilibration time	30 s
pulse amplitude	50 mV
start potential	550 mV
end potential	900 mV
voltage step	6 mV
pulse time	40 ms
voltage step time	0.6 s
sweep rate	10 mV/s
peak potential	700 mV

The concentration is determined by standard addition.

Remark

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

Literature

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

Figures

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===== METROHM 746 VA TRACE ANALYZER (5.746.0101) =====
Method: AB097 .mth OPERATION SEQUENCE
Title : Determination of vitamine E. AB97
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	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

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	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					

Fig. 1: Method for the determination of vitamin E with the 746 VA Trace Analyzer.

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===== 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: -
    
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Pos.  Ident.1/S1  Ident.2/S2  Ident.3/S3  Method.call  Sample size/S0
Vite  VitE        1.0
    
```

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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
    
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Substance : Tocopher
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
Comments  : -----
    
```

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

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Final results          +/- Res.dev.  %          Comments
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Tocopher = 15.866 ug/g          0.670    4.22
    
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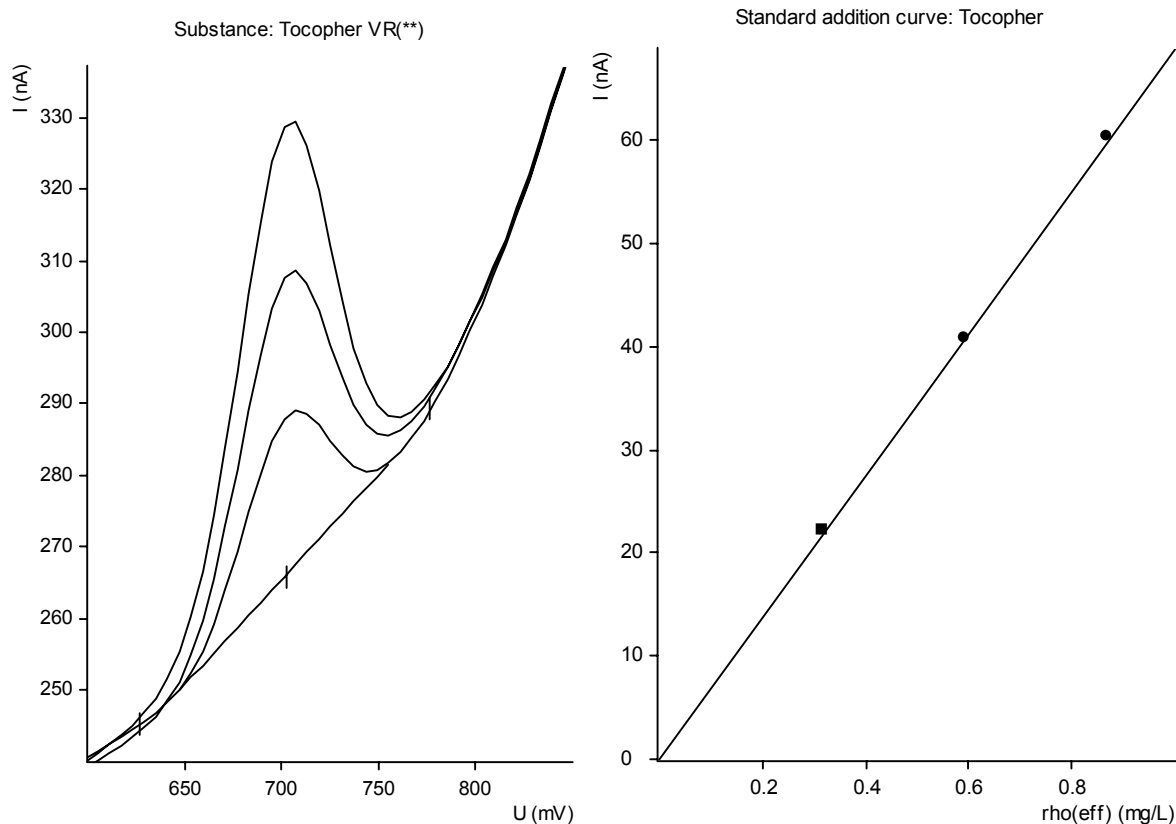


Fig. 2: Example of a determination of vitamin E with the 746 VA Trace Analyzer.