Product Information
Specifications & Tests

DL-alpha-Tocopherol

Product code: 0410276

1.	**Appearance:** Visual evaluation	**clear, viscous oil**
2.	**Colour** Visual evaluation	**colourless to yellowish-brown**
3.	**Identity**	**corresponds**

- Identification Test A. (Optical rotation) as described in the Ph. Eur. monograph 0692 of all-*rac*-α-Tocopherol.
- Gas chromatography method as described under **Assay (Ph. Eur.)**: Identity corresponds when the retention time of the main peak from the sample chromatogram matches the retention time of main peak from the reference solution.

4.	**Optical Rotation**	**-0.01° to + 0.01°**

Proceed according to Ph. Eur. method Optical Rotation (*2.2.7*) and Identification test A. as described in the Ph. Eur. Monograph 0692 of all-*rac*-α-Tocopherol.

5.	**Refractive Index 589 nm, 20°C**	**1.503 - 1.507**

Proceed according to Ph. Eur. method Refractive Index (*2.2.6)*.

6.	**Absorbance in Ethanol at about 292 nm (Max.)**	**71.0 - 76.0**

Proceed according to Ph. Eur. method Absorption Spectrophotometry, Ultraviolet and Visible (*2.2.25*).

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7. Acidity **max. 1.0 mL 0.10 N NaOH**

Proceed according to the specific test for Acidity as described in the USP monograph of Vitamin E or with the in-house as described, below:

Reagents, solvents, standard materials

Ethanol, abs.	96 % Ph. Eur. *R*
Diethyl ether	≥99.7 % Ph. Eur. *R*
1 N NaOH	Ph. Eur. *R*

The described system or a system with equivalent performance may be used:

Titration System

Titration unit for potentiometric titration, *e.g.*, 904 Titrando (Metrohm) with 5 mL dosing unit and stirrer, or equiv.

Electrode: pH electrode for non-aqueous acid/base titrations, *e.g.*, Solvotrode (Metrohm), or equiv.

Analysis of Sample

Dissolve 2–3 g of the sample in 50 mL of a mixture of equal volumes of ethanol (96 per cent) *R* and diethyl ether *R*. Titrate while stirring with 0.02 mol/L NaOH, using the Solvotrode. If necessary, heat to about 40 °C to dissolve the sample. When heating has been applied to aid dissolution, maintain the temperature at about 40 °C during the titration.

Analysis of Blank Sample

Proceed as described above with a blank sample containing 50 mL of a mixture of equal volumes of ethanol (96 per cent) *R* and diethyl ether *R*.

Calculation

$$\text{Acidity} = \frac{(M - B) \times 0.2}{E}$$

M: Consumption of 0.02 N NaOH for the sample solution [mL]
B: Consumption of 0.02 N NaOH for the blank solution [mL]
0.2: Conversion from 0.02 N NaOH to 0.1 N NaOH
E: Sample Weight [g]

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8. Sulfated Ash (Residue on Ignition) max. 0.1 %

Proceed according to Ph. Eur. method Sulfated Ash (*2.4.14*).

9. Residual Solvents

Toluene max. 80 ppm

Determine according to Ph. Eur. method Identification and control of residual solvents (*2.4.24*) or with in-house gas chromatographic method as described, below:

Reagents, solvents, standard materials

DMAC	*N*,*N*-Dimethylacetamide ≥99.9 %
Toluene	≥99.7 %

Preparation of test solution, blank solution, and calibration solution

Stock solution	In a 50 mL volumetric flask, add approx. 20 mL DMAC, then add 890 mg toluene. Fill up to volume with dimethylacetamide.
Blank solution	DMAC
SST solution	In a 50 mL volumetric flask, add approx. 20 mL DMAC, then add 250 µL stock solution. Fill up to volume with dimethylacetamide and homogenize. Transfer 2 mL into a 20 mL headspace vial and seal hermetically.
Test solution	Transfer approx. 200 mg of the test substance, accurately weighted, into a 20 mL Headspace-Vial, add 2 mL of DMAC and seal hermetically.

SST Requirements

Blank	No interfering peak at toluene retention time.
SST	The user laboratory should have procedures in place for adequate system suitability testing.

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Equipment

The described system or a system with equivalent performance may be used

GC system	gas chromatography with static head-space injection
Stationary phase	Agilent DB-1 GC (dimethylpolysiloxane): length 60 m, ID 0.32 mm, Film thickness 3.0 µm or equivalent

GC & HS parameters

Static head space (HS) sample preparation	Vial equilibration temperature		90 °C (HS oven temperature)
	Vial equilibration time		15 min
	Vial Pressurization equilibration time		0.1 min
	Vial shaking		Low
	Vial pressure		1.5 bar
	Loop temperature		110 °C
	Transfer line temperature		130 °C
Other HS parameters (for information)	Vial pressurization time		0.02 min
	Loop fill time		0.05 min
	Loop fill equilibration time		0.05 min
	Temperature equilibration time		0.2 min
	Sampler inject time		2 min
	Sequence valve purge time		2 min
Oven temperature	Rate (°C/min)	Temperature (°C)	Isotime (min)
	---	60	5
	30	120	6
	22.5	210	2

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GC parameters	Carrier gas	Helium for chromatography
	Carrier flow	Constant flow, 1.5 ml/min
	Split	1:10
	Injection volume	1 ml
	Injector temperature	150 °C
	Detector temperature	250°C
	Chromatogram run time	19 min

Calculation

According to the determination by area using an external standard

$$\text{Content [ppm]} = \frac{A_S \times W_{Std} \times CF}{A_{Std} \times W_S} \times 200$$

A_S = area of the corresponding residual solvent in the sample solution [pA*min]
W_{Std} = amount of the corresponding residual solvent in the reference solution [mg]
A_{Std} = area of the corresponding residual solvent in the reference solution [pA*min]
W_S = amount of the test substance [mg]
CF = correction factor
200 = calculation factor in ppm (10^6) with dilution factor (2×10^{-4})

Retention time (for information)

Toluene: 15.3 min

Limit of quantification (LOQ): 20 ppm

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10. Related Substances (Ph. Eur.)

• **Impurity A**	**max. 0.5 %**
• **Impurity B**	**max. 1.5 %**
• **Impurities C and D**	**max. 1.0 %**
• **Any other impurity**	**each max. 0.25 %**
• **Total**	**max. 2.5 %**

Proceed according to test "Related substances" as described in the Ph. Eur. monograph 0692 of all-*rac*-α-Tocopherol or to the in-house gas chromatographic method as described under **Assay (Ph. Eur.)**.

11. Assay (Ph. Eur.) **97.0 – 102.0 %**

Proceed according to test "Assay" as described in the Ph. Eur. monograph 0692 of all-*rac*-α–Tocopherol or the the gas chromatographic in-house method as described, below.

The described system or a system with equivalent performance may be used:

Gas chromatograph	Agilent, Model 6850 with FID detector
Column	Fused silica (crosslinked 5 % Phenylsilanol, 95 % Methylsilanol)
Dimensions:	25 m x 0.32 mm
Film thickness:	0.52 µm
Carrier gas:	H_2
Total flow rate:	2.5 mL/min.
Split ratio:	1:5
Column temperature:	Start 180 °C (2 min.), increase 20.0 °C/min. to 280 °C (12 min.)
Injector temperature:	280 °C
Detector temperature:	300 °C
Injection volume:	1.0 µL
Duration:	19 min.

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Solutions

Internal standard solution

Weigh in 33.9 to 34.1 g C_{28} alkane (= internal standard) and dissolve with 10 L heptane.

Sample solution

Weigh in 170.0 to 210.0 mg of sample (accurately weighed) into a 50 mL volumetric flask and make up to volume with the internal standard solution. Mix for 4 min. (injection solution).

Reference solution

Weigh in 170.0 to 210.0 mg (accurately weighed) of the current all-*rac*-α-tocopherol reference standard into a 50 mL volumetric flask, make up to volume with internal standard solution. Mix for 4 min. (injection solution).

Identity

Identity corresponds when the retention time of the main peak from the sample chromatogram matches the retention time of main peak from the reference solution.

Retention time [min.]	Compound	Relative retention time
approx. 9.0	Internal standard	0.71
approx. 10.5	Impurity A	0.83
approx. 11.4	Impurity B	0.90
approx. 12.7	all-*rac*-α-tocopherol	1.00
approx. 13.0	Impurity C + D	1.02

Calculation

For calculating the amount of impurity A, B, C, and D, the response factor of *all-rac*-α-tocopherol is used.

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Calculation of the assay:

$$\text{Assay (\%,w/w)} = \frac{A_S \times RF \times W_{iStd(S)} \times 100}{A_{iStd(S)} \times W_S}$$

With RF:

$$RF = \frac{W_{Ref} \times A_{Ph.Eur.} \times A_{iStd(St)}}{W_{iStd(St)} \times A_{St} \times 100\%}$$

A_S	peak area of all-*rac*-α-tocopherol in sample solution chromatogram
A_{St}	peak area of all-*rac*-α-tocopherol in standard solution chromatogram
$A_{iStd(S)}$	peak area of internal standard (Octacosan) in sample chromatogram
$A_{iStd(St)}$	peak area of internal standard (Octacosan) in standard solution chromatogram
$A_{Ph.Eur.}$	assay of Ph. Eur. reference standard [%]
$W_{iStd(S)}$	amount of internal standard in sample solution [mg]
$W_{iStd(St)}$	amount of internal standard in standard solution [mg]
W_S	sample weight [mg]
W_{Ref}	weight of all-*rac*-α-tocopherol reference standard [mg]
RF	response factor all-*rac*-α-tocopherol
100%	calculation factor to percent

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