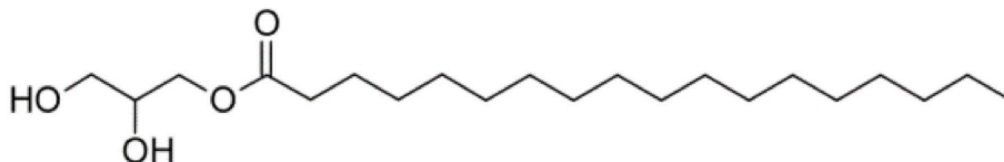




Water determination in Glycerol Monostearate

HYDRANAL™ Laboratory Report L 705

(2,3-Dihydroxypropyl Octadecanoate)



This long-chained stearate does not dissolve in the alcoholic Karl Fischer working media. Thus it is necessary to use a solubilizing agent.

In the European Pharmacopoeia (Ph. Eur.) pyridine is used as a solubilizing agent, at elevated temperature. However, the endpoint detection in pure pyridine is not ideal, as the system tends to over-titrate. For good results, we recommend using a mixture of methanol and chloroform.

Procedure for volumetric one-component titration:

Add 40 mL Hydranal™-LipoSolver CM, or a mixture of 20 mL Hydranal-Methanol dry or Hydranal-Methanol Rapid and 20 mL Hydranal-Chloroform to the titration vessel. Titrate to dryness with Hydranal-Composite 5.

Precisely weigh-in a 1 g sample using differential weighing and titrate the water content with Hydranal-Composite 5.

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Procedure for volumetric two-component titration:

Add 40 mL Hydranal-Solvent CM or a mixture of 20 mL Hydranal-Solvent and 20 mL Hydranal-Chloroform to the titration vessel. Titrate to dryness with Hydranal-Titrant 5. Precisely weigh-in a 1 g sample using differential weighing and titrate the water content with Hydranal-Titrant 5.

Hydranal-Water Standard 10.0, Hydranal-Water Standard 1.0, and Hydranal-Standard Sodium Tartrate Dihydrate are suitable for determination of the titer or control of the volumetric determination.

VOLUMETRIC REAGENTS

34805 HYDRANAL-Composite 5	37863 HYDRANAL-Chloroform
37855 HYDRANAL-LipoSolver CM	34801 HYDRANAL-Titrant 5
34741 HYDRANAL-Methanol dry	34812 HYDRANAL-Solvent CM
37817 HYDRANAL-Methanol Rapid	34800 HYDRANAL-Solvent

WATER STANDARDS

34849 HYDRANAL-Water Standard 10.0	34696 HYDRANAL-Standard Sodium Tartrate Dihydrate
34425 HYDRANAL-CRM Water Standard 10.0	34424 HYDRANAL-CRM Sodium Tartrate Dihydrate
34828 HYDRANAL-Water Standard 1.0	
34426 HYDRANAL-CRM Water Standard 1.0	

AUXILIARIES

34788 HYDRANAL-Humidity Absorber	34241 HYDRANAL-Molecular Sieve 0.3 nm
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Enclosure:

Suitability Test according to Ph. Eur. (also available separately, on request).

Suitability test according to Ph. Eur., method 2.5.12 Water semi-micro determination

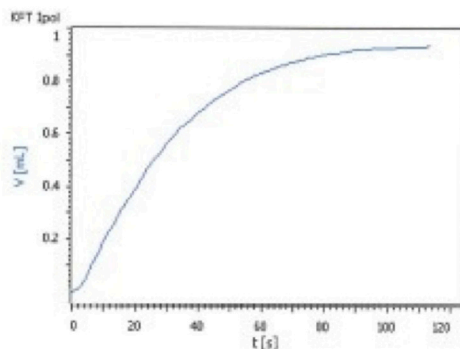
Water determination by Karl Fischer titration using HYDRANAL-Composite

Product	Glycerol monostearate 40-55	
Titrant	HYDRANAL-Composite 5	Titer 5.295 mg/mL
Working medium	40 ml Pyridine	
Sample handling	By means of a powder funnel. Weigh by difference.	
Procedure	The working medium is placed in the titration vessel, heated to 40°C and titrated to dryness with the titrant. Then the sample is added and titrated in the same way to a stable end point.	
Recovery of water added	After achieving the end point, sequential known amounts of water are added and titrated in the same way.	

	Sample	Water added				
		1	2	3	4	5
Sample size (g)	0.9900					
Water added (mg)		3.68	3.82	3.71	3.91	3.78
Water found (mg)	4.9610	3.70	3.85	3.71	3.93	3.79
Water content %	0.5011					
Recovery %		100.56	100.86	99.89	100.59	100.29

The reagent/solvent system is considered to be acceptable if:

The mean recovery is between 97.5% and 102.5%	Mean recovery (%)	100.44
The slope b is between 0.975 and 1.025 (deviation +/-2.5%)	Slope	1.004
The error e1 and e2 are not greater than 2.5%	Error 1 (%)	0.26
	Error 2 (%)	0.12



Test results fulfil the requirements according to Ph. Eur.

This approach, using pyridine, is recommended by Ph. Eur. 7.6. But please note, that end point detection in pure pyridine is not perfect.

Attached you will find the procedure that is favored by the HYDRANAL-laboratory, using a mixture of methanol and chloroform as working medium.

Seelze, 10.09.13



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Product **Glycerol monostearate 40-55**

Titrant HYDRANAL-Composite 5 **Titer** 5.295 mg/mL

Working medium 20 ml HYDRANAL-Methanol dry + 20 ml HYDRANAL-Chloroform

Sample handling By means of a powder funnel. Weigh by difference.

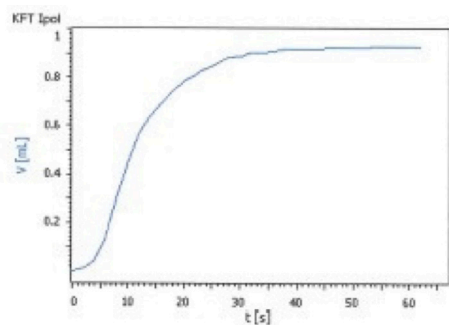
Procedure The working medium is placed in the titration vessel and titrated to dryness with the titrant. Then the sample is added and titrated in the same way to a stable end point.

Recovery of water added After achieving the end point, sequential known amounts of water are added and titrated in the same way.

	Sample	Water added				
		1	2	3	4	5
Sample size (g)	0.9911					
Water added (mg)		3.75	3.48	3.78	3.69	4.00
Water found (mg)	4.8022	3.78	3.49	3.85	3.72	4.01
Water content %	0.4845					
Recovery %		100.65	100.40	101.93	100.95	100.08

The reagent/solvent system is considered to be acceptable if:

The mean recovery is between 97.5% and 102.5%	Mean recovery (%)	100.80
The slope b is between 0.975 and 1.025 (deviation +/-2.5%)	Slope	1.010
The error e1 and e2 are not greater than 2.5%	Error 1 (%)	0.25
	Error 2 (%)	1.19



Test results fulfil the requirements according to Ph. Eur.

Seelze, 05.09.13 WSK



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