Application

SI Analytics

a **xylem** brand

**Water determination
according to Karl Fischer in
organic liquids**

Application

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Use

This method is a general description how to determine the water content in organic liquids such as methanol, isopropyl alcohol, glycol and other similar liquids.

Appliances

- Titrator: TL 7500 KF (TL 7500 KF 05/10/20) or TL 7750 with KF accessories
- µl syringe or one-way insulin syringe with needle (> 0.8 mm)
- 4 digit balance

Electrodes

- KF 1100 with fixed cable

Reagents

- Titration agent: 1 component titrant or 2 component Titrant with 2 or 5 mg/ml concentration
- Solvent: dry methanol, combi solvent or a two component solvent/solvent E

Description

The setup of the instrument is described in the operating manual.

Determination of the titrant concentration

The determination of the titrant concentration (titer determination, standardization) is necessary before the sample titration and we recommend a weekly titer determination.

Fill the titrant in the exchange unit and the solvent in the titration vessel as described in the manual. Do not forget to enter the concentration of the titrant (e.g. 2 or 5) into the WA unit. Otherwise the drift determination is wrong. Load the standard method (default method) Titer 1-Comp. (liq. std.) or Titer 2-Comp. (liq. std.) as user method and start the method.

The KF titrator starts automatically the conditioning routine. The solvent and also the titration vessel contents also water which has to be titrated before. When the conditioning is ready you see "Conditioning ready" on the screen.

The conditioning remains active until the actual titration is started by pressing <F1/START>.

- Open the glass ampoule with the water standard (10 mg/g) and rinse the 10 ml syringe 1-2 times with 0.5 - 1 ml of the standard.
- Fill the complete content of the liquid standard then into the syringe and wipe then the needle with a tissue.
- Place the syringe with needle on a 4 digit balance. Maybe use a beaker, place this beaker on the balance and put the syringe then into the beaker during the weighing process.
- Press the tara button.
- Press Start/F1 to interrupt the conditioning. You are prompted immediately to add the sample.
- Please add between 0.5 and 1 g standard for a 2 mg/ml titrant and 1.5 – 2 g for a 5 mg/ml titrant through the septum,
- Place the syringe again on the balance and read the absolute weight form the balance (ignore the minus)
- Enter the sample ID (optional)
- Enter the sample weight and confirm with ENTER/F1

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The titrator starts then automatically the titration. The result will appear on the screen at the end of the titration. Repeat the titer determination two times (total 3 times). The average value of the titrant concentration in mg/ml will be stored automatically in the exchange unit and used for the calculation of the sample water concentration.

GLP documentation

Titration graph
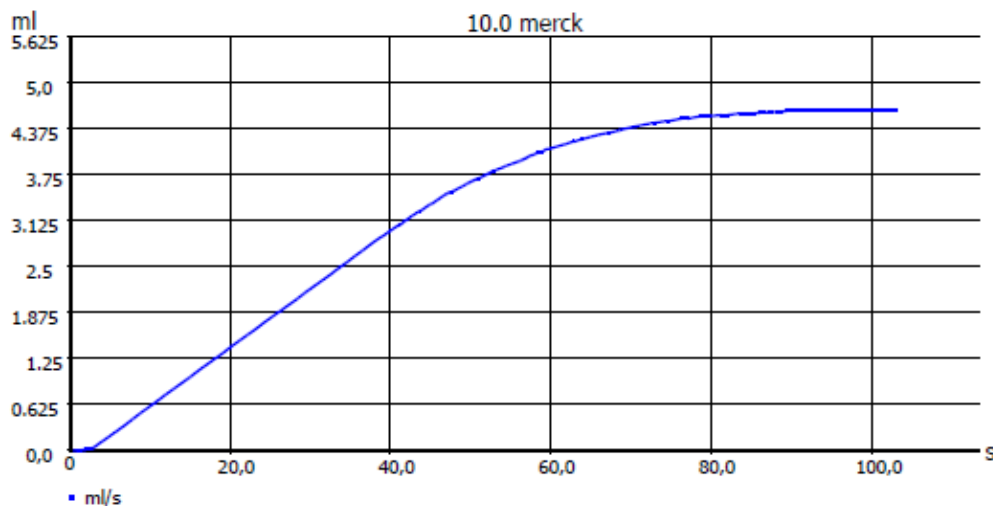
Method data

Method name:	Titer 2-Comp. (liq. st.)	Titration duration:	1 m 43 s
End date:	27.05.13	End time:	16:03:32

Titration data

Sample ID:	10.0 merck	Weight:	0.90070 g
Start µA:	-0.052 µA	End µA:	22.873 µA
Start drift:	59 µg/min	End drift:	43 µg/min
Consumption:	4.624 ml	Drift correction:	Off
Titer:	1.9479 mg/ml		
Mean value:	---	RSD:	---

Calculation formula

Titer:	$(W*F2)/((EP-B)*M*F1) \to WA$	Mol (M):	1.00000
Weight (W):	0.90070 g (m)	Factor 2 (F2):	10.0000
Blank value (B):	0.0000 ml	Factor 1 (F1):	1.0000
Statistics:	1 from 3		

Method data overall view

Method name:	Titer 2-Comp. (liq. st.)	Created at:	05/27/13 13:26:56
Method type:	Automatic titration	Last modification:	05/27/13 15:57:51
Titration mode:	KF	Documentation:	GLP
Linear steps:	0.004 ml		
Fixed delay time:	0 s		

Extraction time:	0 s
Pretitration:	Off
Endpoint:	20.0 µA
delta endpoint:	15.0 µA

Endpoint delay:	10 s
Drift:	50 µg/min
Drift correction:	Off

Min. titration time:	60 s
Max. titration time:	600 s

Polarization voltage:	100 mV

Dosing parameter

Dosing speed:	60.00 %	Filling speed:	30 s
Maximum dosing volume:	50.00 ml		

Unit values

Unit size:	10ml
Unit ID:	10035454
Reagent:	Composite 2
Batch ID:	no entry
Concentration [mg/ml]:	1.94360
Determined at:	05/14/13 23:46:49
Expire date:	--
Opened/compounded:	--
Test according ISO 8655:	08/21/12
Last modification:	05/27/13 15:56:13

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Sample Titration

The procedure for organic liquid samples is the sample as for the liquid standard. The only difference is the used method. Please load the default method "Sample 1 Comp." or "Sample 2 Comp." for the sample titration. Use also a suitable syringe (1, 2, 5 or 10 ml) with needle for the liquid sample.

- Rinse syringe 1-2 times with 0.5 - 1 ml of the sample. Use between 0.2 – 10 g of the sample depending on the expected water concentration (Please refer to the enclosed table)
- Place the syringe with needle on a 4 digit balance. Maybe use a beaker, place this beaker on the balance and put the syringe then into the beaker during the weighing process.
- Press the tara button.
- Press Start/F1 to interrupt the conditioning. You are prompted immediately to add the sample.
- Add the sample trough the septum
- Place the syringe again on the balance and read the absolute weight form the balance (ignore the minus)
- Enter the sample ID (optional)
- Enter the sample weight and confirm with ENTER/F1

The titrator starts then automatically the titration. The result will appear on the screen at the end of the titration.

Recommended sample amounts:

expected water amount	Titre 2 mg/ml	Titre 5 mg/ml
10 ppm – 100 ppm	> 10 g	-----
100 – 500 ppm	2- 10 g	-----
500 ppm – 1000 ppm (0.1 %)	1- 5g	5 – 10 g
0.1 % - 10 %	0.5 – 1 g	1 – 2 g
1 % - 10 %	-----	0.1 – 2 g
10 % - 100 %	-----	max. 0.1 g

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Methods

Sample titration (page 1)

GLP documentation

Titration graph

ml/s
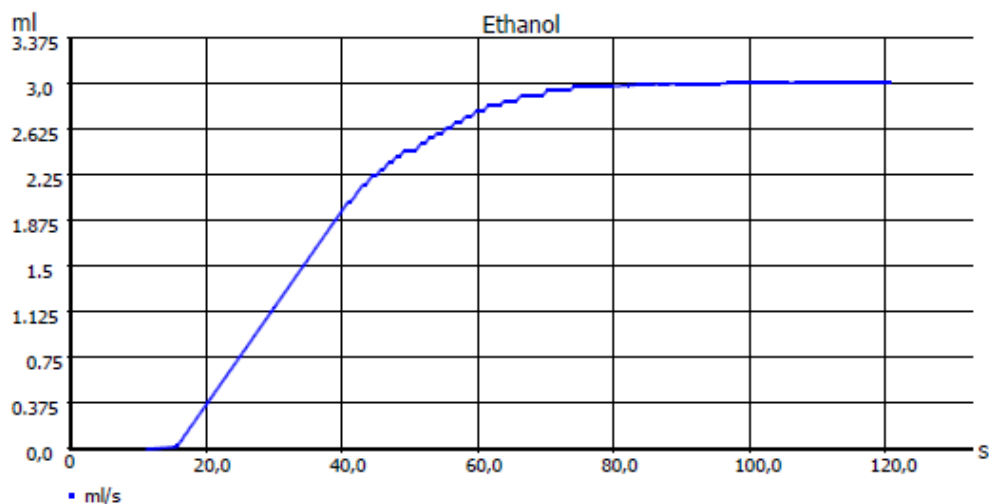
Method data

Method name:	CombiSolvent 1	Titration duration:	2 m 1 s
End date:	06.09.12	End time:	16:34:09

Titration data

Sample ID:	Ethanol	Weight:	0.65630 g
Start µA:	20.565 µA	End µA:	23.324 µA
Start drift:	45 µg/min	End drift:	99 µg/min
Consumption:	3.015 ml	Drift correction:	Off

result:	2.379 %

Calculation formula

result:	(EP-B)*T*M*F1/(W*F2)
Mol (M):	1.00000

Blank value (B):	0.0000 ml	Titre (T):	5.17870000 (a)
Factor 1 (F1):	0.1000	Weight (W):	man
Factor 2 (F2):	1.0000	Statistics:	Off

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sample titration (page 2):

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Method data overall view

Method name:	CombiSolvent 1	Created at:	09/06/12 16:25:47
Method type:	Automatic titration	Last modification:	09/06/12 16:32:18
Titration mode:	KF	Documentation:	GLP
Linear steps:	0.005 ml		
Fixed delay time:	1 s		

Extraction time:	10 s
Pretitration:	Off
Endpoint:	20.0 µA
delta endpoint:	4.0 µA

Endpoint delay:	10 s
Drift:	100 µg/min
Drift correction:	Off

Min. titration time:	30 s
Max. titration time:	600 s

Polarization voltage:	100 mV

Dosing parameter

Dosing speed:	60 %	Filling speed:	30 s
Maximum dosing volume:	50.00 ml		

Unit values

Unit size:	10ml
Unit ID:	10035463
Reagent:	
Batch ID:	no entry
Concentration [mg/ml]:	5.17870
Determined at:	--
Expire date:	--
Opened/compounded:	--
Test according ISO 8655:	08/27/12
Last modification:	09/06/12 16:15:24

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Application

Hints

If you have any questions concerning the application, you are welcome to contact us.

Literature

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