

Determination of water in vegetable oil using Titrator TitroLine® 7500 KF trace

Date: 19.12.2013 Page 1 of 7



Use

The application describes the procedure of the coulometric water determination in vegetable oil products such as olive-, sunflower or similar vegetable oil.

Appliances

Titrator: TitroLine 7500 KF trace M1 - 4

Electrodes

Generating electrode: with diaphragm (TZ 1753) or without diaphragm (TZ 1752)

Reagents

Use with diaphragm (TZ 1753):

Anolyt: Recommended from Sigma Aldrich: 80 ml HYDRANAL-Coulomat

AG-H + 20 ml Chloroform. Merck: CombibiCoulomat frit + additional

solvent

Catholyt Hydranal Coulomat CG for Hydranal reagents; CombiCoulomat frit

for Merck

Additional solvent The addition of up to 20 % to the analyt of a long chain alcohol such

as decanol or octanol or chloroform is recommended for Merck

combicoulomat.

Use without diaphragm (TZ 1752):

Reagent: Recommended is from Sigma Aldrich: Hydranal Coulomat AG-H,

from Merck CombiCoulomat fritless

Additional solvent The addition of up to 20 % to the reagent of chloroform or a long

chain alcohol such as decanol or octanol is recommended. Standards are available from Merck and Sigma Aldrich.

Standard Standards are available from Merck and Sigma Aldrich.

Recommended are the standards with concentration of 0.1 %.

Date: 19.12.2013 Page 2 of 7



Description

Set up the unit and fill the reagents as described in the operating manual. Switch on the instrument and wait until the drift is < 10 μ g/min and stable. For M3 and M4 (generator electrode with diaphragm) it takes sometimes several hours to get a low drift value.

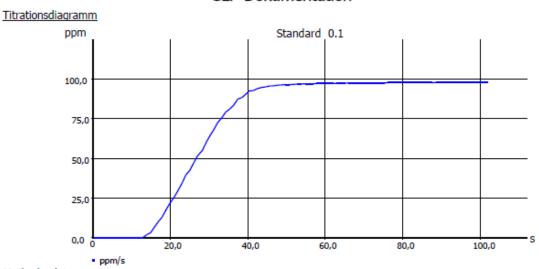
Standard and sample Titration

Before you start the sample titration the first time it is recommended to run some tests with a water standard. Standards with certificat in ampoules are recommended instead of pure water.

Standard:

- Open the ampoule
- Use a suitable plastic or glass syringe. Depended on the standard use a needle with a diameter between 0.8 mm and 1.2 mm and a length of minimum 70 mm.
- First rinse the syringe 1-2 times with 1 ml each of the standard then draw up slowly the entire ampoule content in the syringe without air-bubbles.
- Place a 100 ml glass beaker (tall form) on a balance, put the syringe inside and weigh it.
- Press tara
- Press the start button on the TL 7500 KF trace
- Inject about 0,75 1,5 ml of the standard into the titration vessel
- Place the syringe inside the glass baker on the balance and read the exact weight from the display/or press the print button for automatic transfer.
- Enter sample ID and sample weight. The titration starts automatically.
- Repeat the determination 2 -3 times.

GLP-Dokumentation



Methodendaten

Methodenname:	water in ppm	litrationsdauer:	1 m 42 s
Enddatum:	21.03.13	Endzeit:	11:24:16

<u>Titrationsdaten</u>

Proben ID:	Standard 0.1	Einwaage:	3.51320 g
Startdrift:	2.8 µg/min	Enddrift:	4.5 μg/min
Wasser:	343.899 µg		
Result:	97.9 ppm		
Mittelwert:	98.1 ppm	rel. STABW:	0.9 %

Date: 19.12.2013 Page 3 of 7



Sample:

- Open the sample container
- Use a suitable plastic or glass syringe. Depended on the sample use a needle with a diameter between 0.9 mm and 1.5 mm.
- First rinse the syringe 1-2 times with the sample and then then draw up slowly the sample in the syringe without air-bubbles.
- Place a 100 ml glass beaker (tall form) on a balance, put the syringe inside and weigh it.
- Press tara
- Press the start button on the TL 7500 KF trace
- Inject about 1-2 ml of the sample into the titration vessel
- Place the syringe inside the glass baker on the balance and read the exact weight from the display/or press the print button for automatic transfer.
- Enter sample ID and sample weight. The titration starts automatically.

Note:

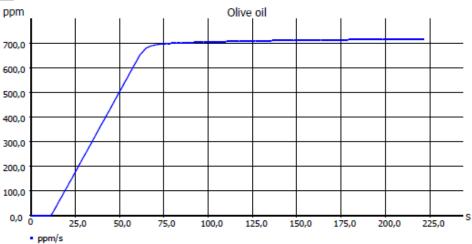
If the sample is not homogenous it has to be homogenised before.

Date: 19.12.2013 Page 4 of 7

a **xylem** brand

GLP documentation

Titration graph



Method data

Method name: Olive oil Titration duration: 3 m 42 s
End date: 16.05.13 End time: 10:32:59

Titration data

Sample ID: Olive oil Weight: 0.68190 g

Start drift: $64.7~\mu g/min$ End drift: $7.0~\mu g/min$

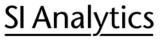
Water: 488.529 μg Water: 716.4 ppm

Calculation formula

Water: $\mu g^*M^*F1/(F2^*W)$ Mol (M): 1.00000

Factor 1 (F1): 1.0000 Factor 2 (F2): 1.0000 Weight (W): 0.68190 g (m) Statistics: Off

Date: 19.12.2013 Page 5 of 7



a **xylem** brand

Method

Method data overall view

Method name: Olive oil Created at: 05/16/13 9:59:24

Method type: Automatic titration Last modification: 05/16/13 9:59:24

Documentation: GLP

 $\begin{array}{ll} \mbox{Start drift:} & 10.0 \ \mu \mbox{g/min} \\ \mbox{Stop drift (delta):} & 2.0 \ \mu \mbox{g/min} \\ \mbox{Stop drift tolerance:} & 0.02 \ \mbox{ug/min}^2 \end{array}$

Stop delay time: 5 s

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

Working point: 300 mV Control factor: 4

Date: 19.12.2013 Page 6 of 7



Hints

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

Literature

SI Analytics GmbH Hattenbergstr. 10 55122 Mainz Germany

Phone: +49 (0) 6131 / 66 - 5118

+49 (0) 6131 / 66 - 5118 +49 (0) 6131 / 66 - 5001 Fax: E-Mail: titration@si-analytics.com

Homepage: www.si-analytics.com

Page 7 of 7 Date: 19.12.2013