

**Determination of water in
petrochemistry products
using Titrator TitroLine 7500
KF trace**

Use

The application describes the procedure of the coulometric water determination in petrochemistry products such as mineral oil and similar products. The application note describes only the direct titration and not the use of evaporation oven.

Appliances

Titration: TitroLine 7500 KF trace M1 - 4

Electrodes

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

Reagents

Use with diaphragm (TZ 1753):

Anolyte: Recommended from Sigma Aldrich: 70 ml HYDRANAL-Coulomat A + 30 ml Chloroform or 70 ml HYDRANAL-Coulomat Oil. Merck: CombiCoulomat frit + additional solvent

Catholyte: Hydranal Coulomat CG for Hydranal reagents; CombiCoulomat frit for Merck

Additional solvent: The addition of up to 30 % to the anolyte 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 a long chain alcohol such as decanol or octanol is recommended (not needed for Hydranal AG-H).

Standard: Standards are available from Merck and Sigma Aldrich. Recommended are the standards with lower concentration of 0.01 %. Available are also special oil standards with very low water concentrations between 15-30 ppm.

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 $< 10 \mu\text{g}/\text{min}$ 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

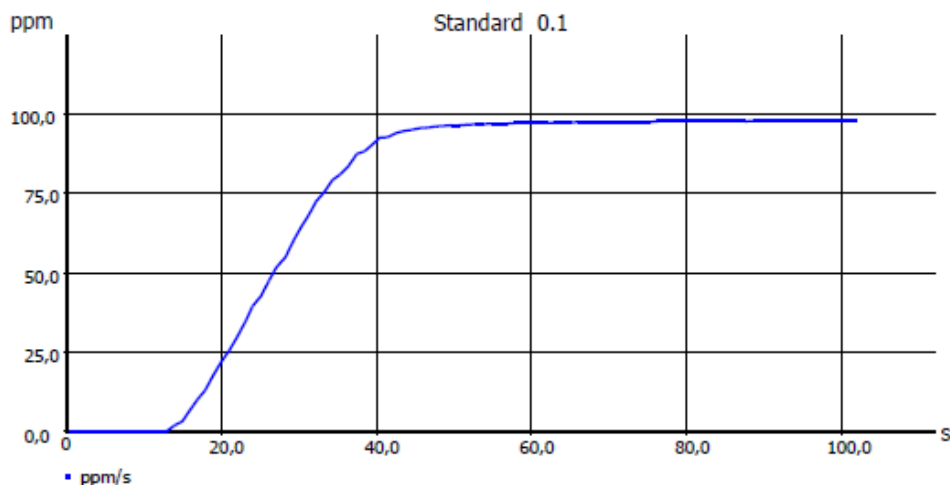
Before you start the sample titration it is recommended to run some tests with a water standard. Standards with certificate in ampoules are recommended instead of pure water.

Standard:

- Open the ampoule
- Use a suitable plastic or glass syringe. Depending on the standard use a needle with a diameter between 0.8 mm and 1.0 mm (oil standard) 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 beaker 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

Titrationdiagramm



Methodendaten

Methodenname: Water in ppm
Enddatum: 21.03.13

Titrationdauer: 1 m 42 s
Endzeit: 11:24:16

Titrationdaten

Proben ID: Standard 0.1

Einwaage: 3.51320 g

Startdrift: 2.8 $\mu\text{g}/\text{min}$

Enddrift: 4.5 $\mu\text{g}/\text{min}$

Wasser: 343.899 μg

Result: 97.9 ppm

Mittelwert: 98.1 ppm

rel. STABW: 0.9 %

Application

Sample:

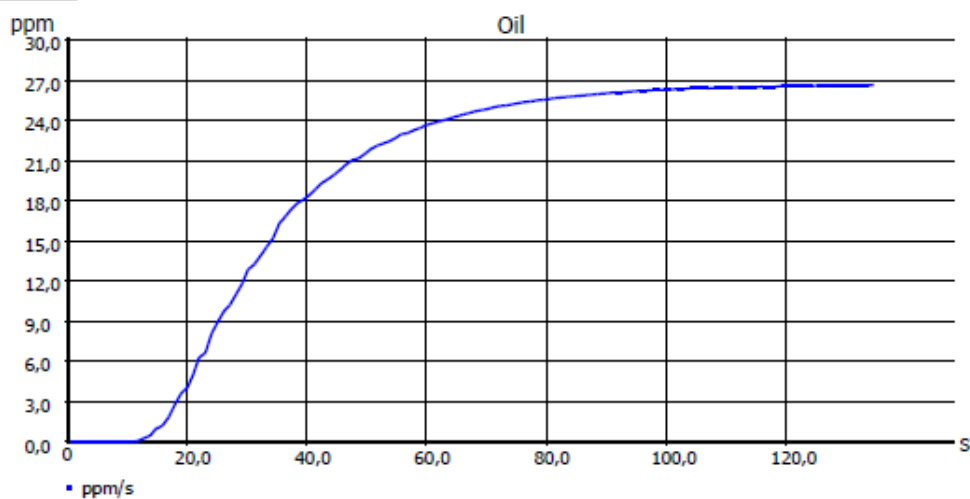
- Open the sample container
- Use a suitable plastic or glass syringe. Depended on the sample use a needle with a diameter between 0.9 mm and 1.5 mm.
- First rinse the syringe 1-2 times with the sample and then then draw up slowly the sample in the syringe without air-bubbles.
- Place a 100 ml glass 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.

GLP-Dokumentation

Titrationdiagramm



Methodendaten

Methodenname:	Oil VWR Belgium	Titrationdauer:	2 m 15 s
Enddatum:	21.03.13	Endzeit:	18:32:47

Titrationdaten

Proben ID:	Oil	Einwaage:	3.93360 g
Startdrift:	1.7 µg/min	Enddrift:	3.1 µg/min
Wasser:	104.787 µg		
Water:	26.6 ppm		

Method

Method data

Method name:	Water in ppm	Created at:	04/29/13 8:35:39
Method type:	Automatic titration	Last modification:	05/03/13 14:15:33
		Documentation:	GLP

Start drift:	10.0 µg/min
Stop drift (delta):	2.0 µg/min
Stop drift tolerance:	0.02 ug/min ²
Stop delay time:	5 s

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

Working point:	300 mV
Control factor:	4

Calculation formula

Water:	$\mu\text{g} \cdot \text{M} \cdot \text{F1} / (\text{F2} \cdot \text{W})$	Mol (M):	1.00000
Unit:	ppm	Decimal places:	1

Factor 1 (F1):	1.0000	Factor 2 (F2):	1.0000
Weight (W):	man	Statistics:	Off

Device information

Device: TitroLine 7500 KF trace
Serial number: 10047704
Software version: 1312t

mth_Water_in_ppm_06_05_13-14_29_29.pdf

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Hints

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

Literature

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