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

<table>
<thead>
<tr>
<th>Titrator:</th>
<th>TitroLine 7500 KF trace M1 - 4</th>
</tr>
</thead>
</table>

## Electrodes

<table>
<thead>
<tr>
<th>Generating electrode:</th>
<th>with diaphragm (TZ 1753) or without diaphragm (TZ 1752)</th>
</tr>
</thead>
</table>

## Reagents

**Use with diaphragm (TZ 1753):**

<table>
<thead>
<tr>
<th>Anolyt:</th>
<th>Recommended from Sigma Aldrich: 70 ml HYDRANAL-Coulomat A + 30 ml Chloroform or 70 ml HYDRANAL-Coulomat Oil. Merck: CombiCoulomat frit + additional solvent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Catholyt</td>
<td>Hydranal Coulomat CG for Hydranal reagents; CombiCoulomat frit for Merck</td>
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</table>

| Additional solvent | The addition of up to 30 % to the anolyt of a long chain alcohol such as decanol or octanol or chloroform is recommended for Merck combicoulomat. |

**Use without diaphragm (TZ 1752):**

<table>
<thead>
<tr>
<th>Reagent:</th>
<th>Recommended is from Sigma Aldrich: Hydranal Coulomat AG-H, from Merck CombiCoulomat fritless</th>
</tr>
</thead>
</table>

| 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. |
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 µg/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 certificat 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.

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**GLP-Dokumentation**

![Titrationsdiagramm](image)

**Methodendaten**
- Methodenname: Water in ppm
- Enddatum: 21.03.13
- Titrationsdauer: 1 m 42 s
- Endzeit: 11:24:16

**Titrationsdaten**
- Proben ID: Standard 0.1
- Einwaage: 3.51320 g
- Startdrift: 2.8 µg/min
- Wasser: 343.899 µg
- Result: 97.9 ppm
- Mittelwert: 98.1 ppm
- rel. STABW: 0.9 %
Sample:
- Open the sample container
- Use a suitable plastic or glass syringe. Depending 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 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

Titration diagram

<table>
<thead>
<tr>
<th>ppm</th>
<th>Oil</th>
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<tbody>
<tr>
<td>0</td>
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</tr>
<tr>
<td>2.0</td>
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<td>3.0</td>
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<td>29.0</td>
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<tr>
<td>30.0</td>
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</table>

Methodendaten
- Methodenname: Oil VWR Belgium
- Titrationsdauer: 2 m 15 s
- Enddatum: 21.03.13
- Endzeit: 18:32:47

Titrationstable
- 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 data

Method name: Water in ppm
Method type: Automatic titration
Created at: 04/29/13 8:35:39
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 µg/min²
Stop delay time: 5 s

Min. titration time: 60 s
Max. titration time: 500 s

Working point: 300 mV
Control factor: 4

Calculation formula

Water: µg*M*F1/(F2*M)
Unit: ppm
Mol (M): 1.00000
Decimal places: 1

Factor 1 (F1): 1.00000
Weight (W): mg
Factor 2 (F2): 1.00000
Statistics: Off

Device information

Device: TitroLine 7500 KF trace
Serial number: 10047004
Software version: 13124

Date: 06.05.2013
Hints

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

Literature

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