Water determination in petrochemical products using coulometric KF-Titrator TitroLine® 7500 KF trace

Description

This application note describes the general determination of water in liquid petrochemical samples using the coulometric KF titrator TitroLine 7500 KF trace. The application note includes samples such as transformer, engine oils, diesel and other fuels or similar samples which needs the use of a generator electrode with diaphragm. The application note describes only the direct titration and not the use of the headspace oven TO 7280 oven.

The coulometric method is recommended for water concentrations from 1 ppm to 5 %.

Instruments

Titrator	TL 7500 KF trace
Indicator Electrode	KF 1150
Generator Electrode	TZ 1753
Cable	LB 04 NN
Stirrer	TM 235, TM 235 KF
Titration vessel	TZ 1751, TZ 1754
Other items	Desiccant (molecular sieve 4 nm). 100 g are included in the scope of delivery of the accessory kit TZ 1789 (scope of delivery of M3 and M4) as well as 1 g glass wool and syringes with cannula.

Reagents

1	Anolyt: 70 ml HYDRANAL-Coulomat A + 30 ml Xylol or 100 ml HYDRANAL-Coulomat Oil. Merck: CombibiCoulomat frit + additional solvent
2	Catholyt: Hydranal Coulomat CG for Hydranal reagents; CombiCoulomat frit for Merck
3	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.
4	Water standards: 1 mg/g, 0.1 mg/g certified standard in ampoules or specila standard for Oil with low water concentration

Reagents

It is recommended to use only ready-to-use coulometric KF reagents. See possible reagents and mixtures above.

Cleaning of the electrode

The indicator electrode KF 1150 and the generator electrode can be cleaned with all kinds of solvents. Heavy contamination can be removed with 65 % nitric acid. The electrode must be immersed in nitric acid for at least 1-2 hours. Then rinse the electrode successively with deionized water and alcohol. Allow the electrode to dry either in the air or at max. 45-50 ° C in the drying cabinet. An der Luft

Sample preparation

Coconut oil like virgin coconut oil is solid at room temperature up to 25 °C. Take 10 or more g of the pre-mixed waxy sample and place it in a water bath at 30–40 °C. After a couple of minutes, the sample becomes liquid.

General procedure

Assemble the titrator according to the instructions and connect the two electrodes. Fill the molecular sieve into the glass drying tube TZ 1505.

Fill approx. 100 ml of anolyte into the titration vessel via a glass funnel or by operating the pump of the stirrer TM 235 KF. When the mark (TZ 1751 / TZ 1754) is reached, there is about 100 ml inside the vessel.

Fill about 3 ml of the catholyte into the cathode chamber of the generator electrode and put it into the titration vessel.

Set the stirring speed to an appropriate level. Air bubbles can occur as this does not disturb the titration.

Switch the titrator on. When the titrator is fully powered up, it will immediately start to generate iodine at the generator electrode. Select the standard method ‘Water in ppm’ or ‘Water in %’.

The titrator continues to condition until the starting conditions (drift < 10 µg/min) are not reached. When the starting conditions are reached, the method can be started. If the start conditions have not been reached, the method is greyed out. The conditioning with a generator electrode with diaphragm takes longer than with a generator electrode without. It can take a couple of hours.

Standard:

- Open the ampoule
- Use a suitable plastic or glass syringe. Use a needle with a diameter between 0.6 – 0.8 mm and a length of minimum 70 mm.
- First rinse the syringe with 1 ml 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 + needle and cap inside and weigh it.
- Zero the balance
- Press the start button on the TL 7500 KF trace
- "Please add the sample" appears on the display
- Inject about 0.75 – 1.5 ml of the standard into the titration vessel after piercing the septum. The needle should be inside the anolyte. Then remove the syringe a little bit and draw a few ml back
- Place the syringe back to the glass beaker on the balance and read the exact weight from the display or press the print button for automatic transfer in case the balance is connected to the titrator.
- Enter sample ID and sample weight. The titration starts automatically.
- Repeat the determination 1-2 times.

Sample:

- Sample preparation of the coconut sample see above.
- Use a suitable plastic or glass syringe. Use a needle with a diameter between 1.2- 1.5 mm and a length of minimum 70 mm.
- First rinse the syringe 1-2 times with 1 ml each of the sample. Then fill 1 – 5 ml sample into the syringe.
- Place a 100 ml glass beaker (tall form) on a balance, put the syringe + needle and cap inside and weigh it.
- Zero the balance
- Press the start button on the TL 7500 KF trace
- "Please add the sample" appears on the display
- Inject about 0.5 – 2 ml of the sample into the titration vessel after piercing the septum. The needle should be inside the anolyte. Then remove the syringe a little bit and draw a few ml back.
- Place the syringe back to the glass beaker on the balance and read the exact weight from the display or press the print button for automatic transfer in case the balance is connected to the titrator.
- Enter sample ID and sample weight. The titration starts automatically.

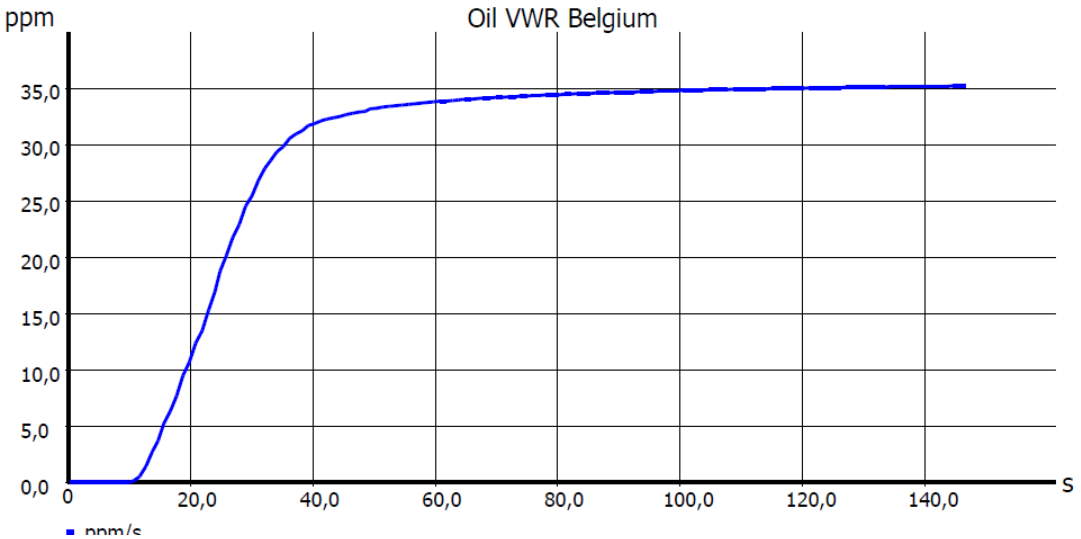
Recommended sample amounts

Water concentrations	Sample amount
1 – 10 ppm	5 – 10g
10 – 100 ppm	2 – 5 g
0.01 – 0.1 %	0.5 – 2 g
0.1 – 1 %	0.1 – 0.5
1 – 5 %	0.05 – 0.1 g

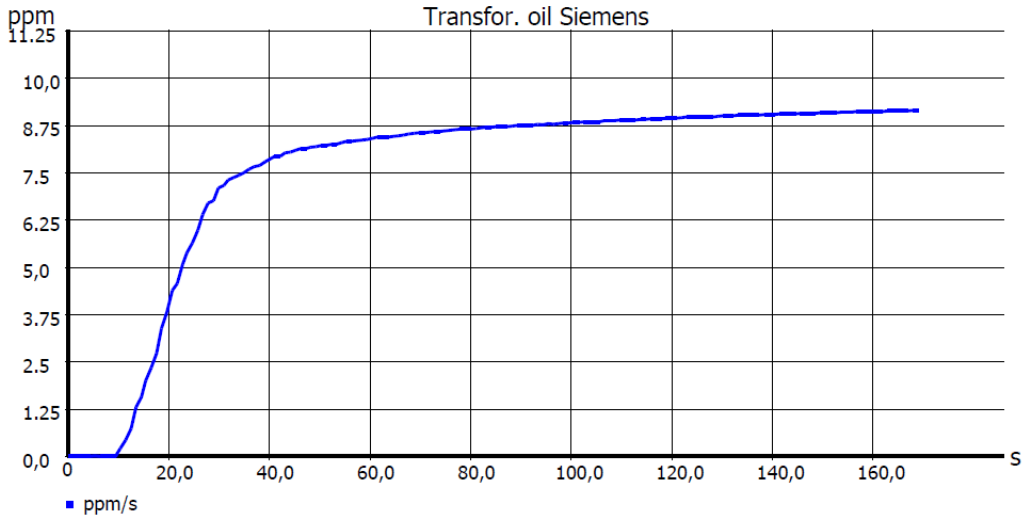
Titration parameter

Sample titration examples:

Titration graph



Titration graph



Default method	Water in %/ppm
Start drift	10.0 µg/min
Interval	30 seconds
Stability:	0.5 µg/min
Stop drift (delta):	2.0 µg/min
Stop drift tolerance:	0.02 µg/min²
Stirrer control	free
Min. titration time:	60 seconds
Max. titration time:	600 seconds
Working point:	300 m
Control factor	4

Calculation:

$$Result\ [ppm] = \mu g * M * F1/(F2 * W)$$

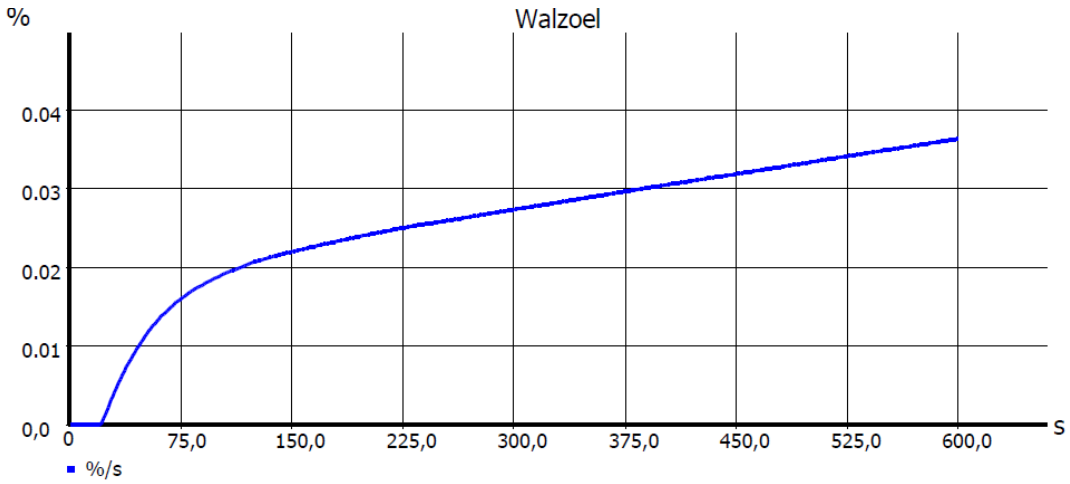
µg		Titrated water amount absolute
M	1	Molecular weight
W	man	sample weight in g
F1	1	Conversion factor
F2	1	Conversion factor

Calculation:

$$Result\ [\%] = \mu g * M * F1/(F2 * W)$$

µg		Titrated water amount absolute
M	1	Molecular weight
W	man	sample weight in g
F1	0.0001	Conversion factor
F2	1	Conversion factor

If the titration curves look like below then there is a side reaction and it is recommended to use the headspace oven TO 7280:



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Let's Solve Water