DETERMINATION OF WATER IN PETROCHEMICAL PRODUCTS USING COULOMETRIC KARL FISCHER

SI Analytics
a xylem brand

Titration Application

Use

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

Required Equipment

Apparatus

- ☐ TitroLine 7500 KF trace M1 - 4

Electrode and Electrolyte

- ☐ Generating Electrode: With diaphragm (TZ 1753) or without diaphragm (TZ 1752)

Reagents

Use with diaphragm (TZ 1753):

- ☐ Anolyt: Recommended from Honeywell: 70 ml HYDRANAL-Coulomat A + 30 ml Xylol or 100 ml HYDRANAL-Coulomat Oil. Merck: CombiCoulomat frit + additional solvent
- ☐ Catholyt: Hydranal Coulomat CG for Hydranal reagents; CombiCoulomat frit for Merck
- ☐ Additional solvent: The addition of up to 30 % to the anolyt of a long chain alcohol such as decanol, octanol or chloroform is recommended for Merck combicoulomat.

Use without diaphragm (TZ 1753):

- ☐ Reagent: Recommended from Honeywell: 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 for Merck CombiCoulomat (not needed for Hydranal AG-H).
- ☐ Standard: Standards are available from Merck and Honeywell. Standards with lower concentration (0.01 %) are recommended. Special oil standards are also available with very low water concentrations between 15-30 ppm.

Procedure

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 sometimes takes several hours to get a low drift value.

Standard and Sample Titration

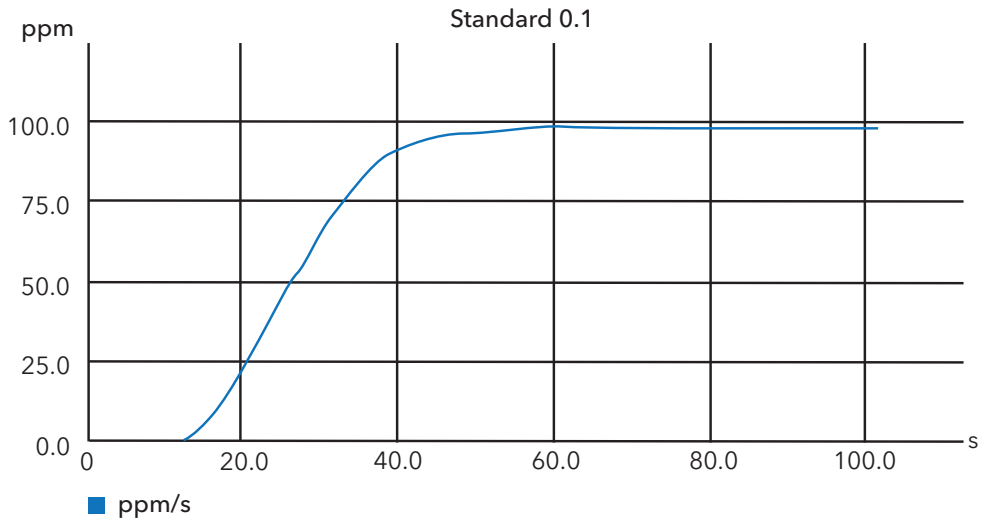
Before you start the sample titration it is recommended to run some tests with a water standard. Standards with certificate in ampoules are recommended instead of pure water.

Standard:

- Open the ampoule carefully.
- 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 minimum length of 70 mm.
- First, rinse the syringe 1-2 times with 1 ml of the standard then slowly draw up the entire ampoule content in the syringe without air-bubbles.
- Place a 100 ml glass beaker (tall form) on a balance and put the syringe inside.
- Press **Tare**.
- 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 absolute 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 documentation

Titration graph



Method data

Method name:	Water in ppm	Titration duration	1 m 42 s
End date:	21.03.13	End time:	11:24:16

Titration data

Sample ID:	Standard 0.1	Weight	3.51320 g
Start drift:	2.8 µg/min	End drift:	4.5 µg/min
Water:	343.899 µG		
Result:	97.9		
Average:	98.1 ppm	rel. STDEV:	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, slowly draw up the sample in the syringe without air bubbles.
- Place a 100 ml glass beaker (tall form) on a balance and put the syringe inside.
- Press **Tare**
- 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 beaker on the balance and read the absolute weight from the display or press the **Print** button for automatic transfer.
- Enter sample ID and sample weight. The titration starts automatically.

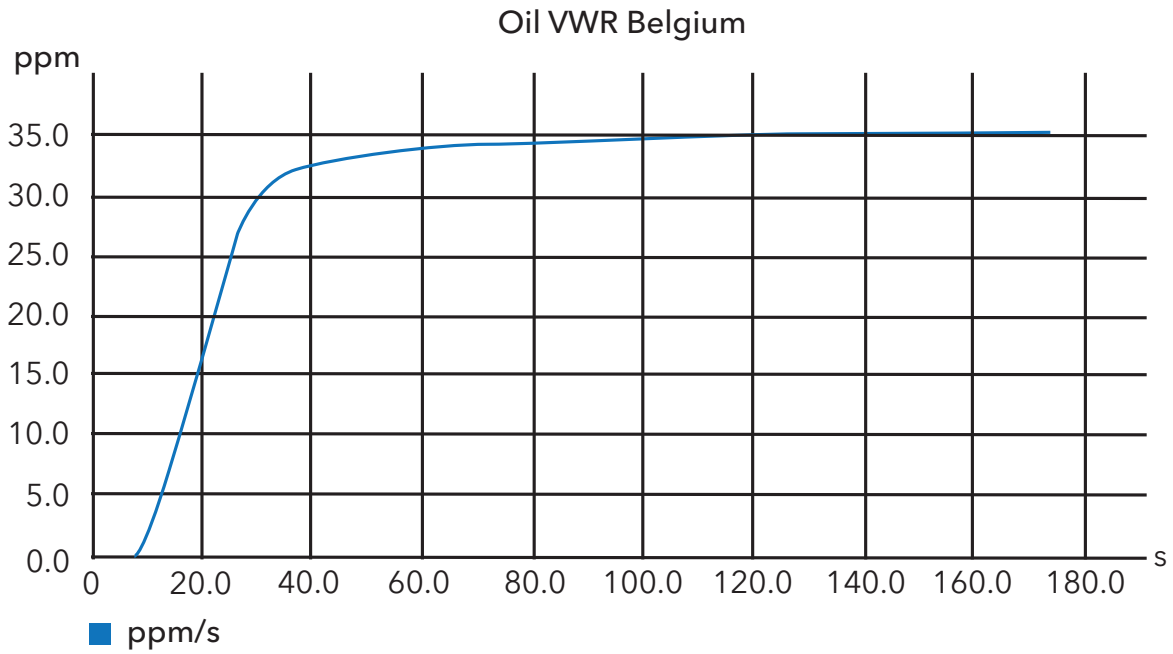
Note:

Sample must be a homogenous representation of the product being tested. Homogenize prior to titrating if necessary.

Sample Result (Page 1)

GLP documentation

Titration graph



Method data

Method name:	Oil (ppm)	Titration duration	2 m 54 s
End date:	16.05.13	End time:	11:17:42

Titration data

Sample ID:	Oil VWR Belgium	Weight:	4.78470 g
Start drift:	4.3 µg/min	End drift:	6.1 µg/min
Water:	169.173 µg		
Water:	35.4 ppm		

Calculation formula

Water:	µg*M*F1/(F2*W)	Mol (M):	1.00000
Factor 1 (F1):	1.0000	Factor 2 (F2):	1.0000
Weight (W)	4.78470 g (m)	Statistics:	Off

Method data

Method data overall view

Method name:	Oil (ppm)	Created at:	05/16/13 11:09:09
Method type:	Automatic titration	Last modification:	05/16/13 11:09:09
		Documentation:	GLP

Start drift:	10.0 µg/min
Stop drift (delta)	2.0 µg/min
Stop drift tolerance	0.02 µg/min^2
Stop delay time:	5 s
Min. titration time	60 s
Max. titration time:	600 s
Working point:	300 mV
Control factor:	4

Contact Information

Please contact our titration experts if you have any application or product questions. Thanks!

Xylem Inc.
1725 Brannum Lane
Yellow Springs, Ohio 45387

Application/Technical Support:
+ 1-845-258-1200
titration.ysi@xyleminc.com

Ordering:
+1-937-767-7241
orders@ysi.com

Publication 44810418