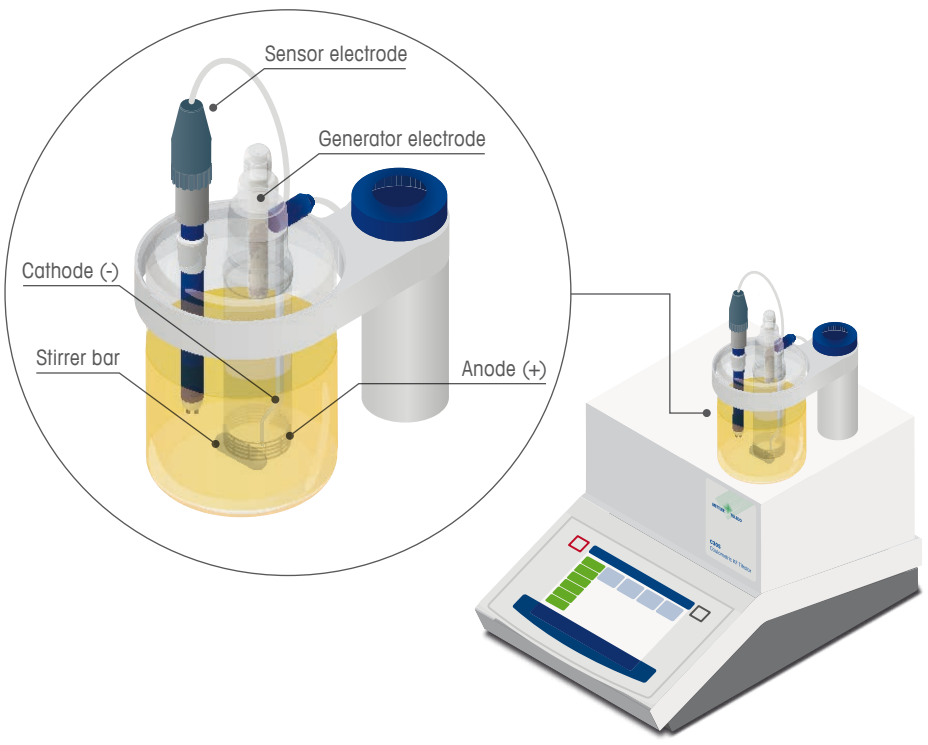
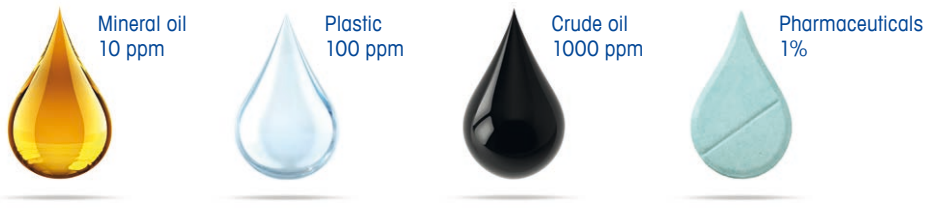
Karl Fischer Titration Guide

1 How much water to measure?

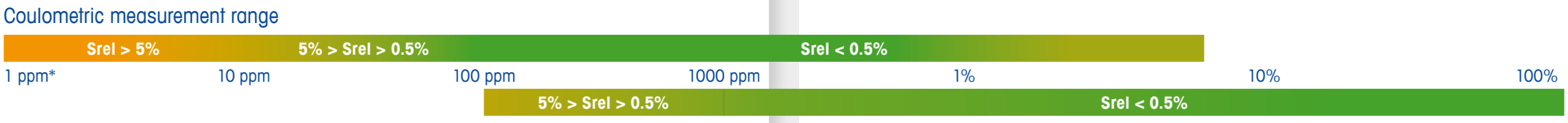
Coulometric

For low water contents from 1 ppm to 5%, iodine is generated electrochemically in the titration cell.





Coulometric measurement range

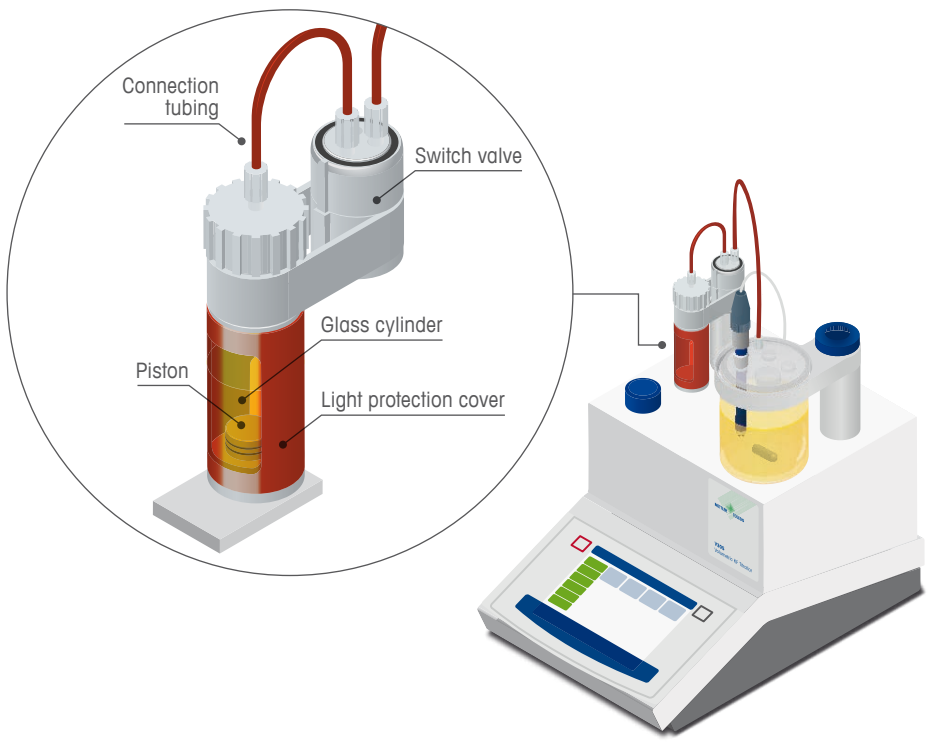


Srel = relative standard deviation between measurements

* For 1 to 10 ppm, a coulometric cell with diaphragm should be used.

Volumetric

For higher water contents from 100 ppm to 100%, a known amount of iodine contained in the titrant is metered via the burette.



Volumetric measurement range

2 How much sample to use?

Coulometric

The ideal coulometric titration sample contains 1 mg of water. Recommended practical working range is 0.1 to 5 mg.

Water content of the sample	Sample size (g)	Absolute water quantity (mg)
5%	0.1	5.0
1%	0.2	2.0
0.1% (1000 ppm)	1.0	1.0
0.01% (100 ppm)	5.0	0.5
0.001% (10 ppm)	10.0	0.1
0.0001% (1 ppm)	10.0	0.01

Recommended conditions
Borderline conditions for accuracy or sample size
— = not recommended

Volumetric

The ideal volumetric titration uses ½ of the burette volume. Recommendations below use a standard burette size of 5 mL.

Titrant concentration	5 mg/mL	2 mg/mL	1 mg/mL
For burette volume: 5 mL	Sample size (g)		
100%	0.015	—	—
60%	0.020	0.008	—
40%	0.030	0.013	—
30%	0.040	0.017	—
10%	0.125	0.05	0.025
1%	1.25	0.5	0.25
0.1% (1000 ppm)	12.5	5	2.5
0.01% (100 ppm)	25	25	25

3 Which reagents and titrants to use?

Coulometric

Most coulometric titrations require only one reagent in the titration cell – anolyte. Some require an additional reagent – catholyte.*

HYDRANAL™ anolytes:
- Coulomat AG, AG-Oven, AD, E (universal, based on alcohols)
- Sample-specific Coulomat types*

HYDRANAL catholytes:
- Coulomat CG (universal)
- Coulomat CG-K (methanol-free for ketones)

HYDRANAL-Water Standards
Calibration and validation of instruments and reagents per ISO, GMP, GLP and FDA guidelines should be performed with exactly confirmed water standards, e.g. 1.0%, 0.1% or 0.01% content.

* For samples requiring additional solvents or a methanol-free system, also for highest accuracy at low ppm water contents, special coulometric cells with a ceramic diaphragm to contain catholyte should be used.

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Volumetric

Volumetric titrations may use 1- or 2-component reagent systems with different advantages in flexibility, speed and accuracy.

HYDRANAL 1-component reagents:
- Composite 1
- Composite 2
- Composite 5 or 5K

1-component systems provide the best flexibility in solvent selection, convenience and ease of use.

HYDRANAL 2-component reagents:
- Titrant 2 or 2 E
- Titrant 5 or 5 E
- Solvent or Solvent E

2-component systems provide the highest titration speeds, best accuracy at low water amounts and highest titer stability.

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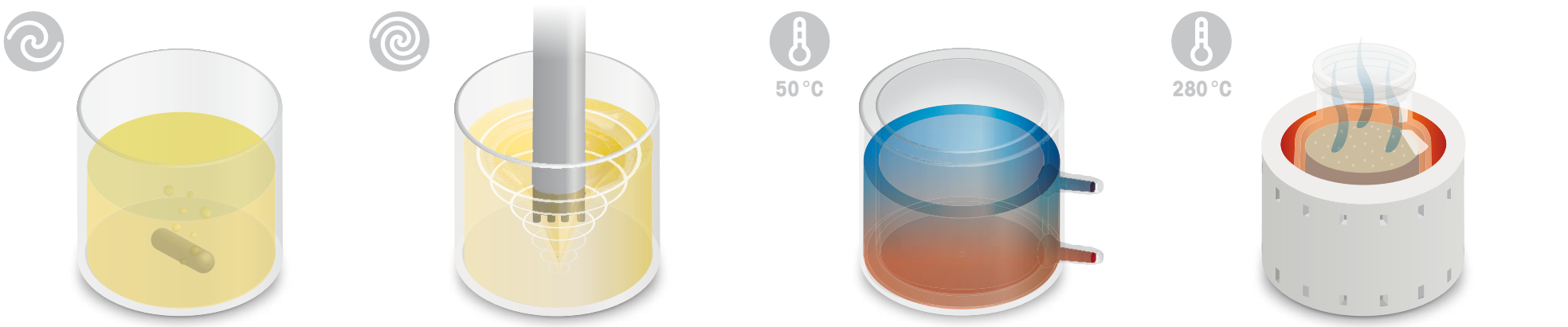
4 How to extract water from the sample?

Obtaining accurate Karl Fischer results requires extracting all water from samples. Many liquid sample types dissolve easily in the typical KF solvent methanol, while viscous oils, pastes or solid samples require other solvents, increased dispersion or higher temperatures to fully extract water.

Extraction solvents for sample types:
- Methanol for liquid foods, cosmetics, organic solids
- Chloroform for petroleum products, fats, organic solids
- Long-chain alcohols (e.g., octanol) for edible oils, fuels, creams
- Toluene or xylene for tars, waxes, crude oil
- Formamide for sugars, starches, solid foods, pharmaceuticals



A. Mixing
Many samples dissolve in methanol or other solvent using a normal magnetic stirring bar.

B. Dispersion
Thicker liquids, pastes or powders may require more intense dispersion with a high speed homogenizer to dissolve them fully.

C. Warming
A thermostatable titration vessel allows warming up to 50 °C with an external oil or water bath to improve dissolution and extraction.

D. Oven
For solids or liquids causing side reactions or releasing water very slowly, a drying oven heats samples up to 280 °C to drive water out and into the titration beaker.

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