

Combining speed and accuracy



## Aquamax KF *Reagents*

*The Truly high performance brand of  
Coulometric Karl Fischer Reagents*

## Principle of Measurement

Karl Fischer titration is simply a method for quantifying water content of samples. The fundamental principle behind this reaction is based on the Bunsen reaction between compounds such as iodine, sulphur dioxide to form sulphuric acid and hydrogen iodide. Karl Fischer initiated a new method based on Bunsen's reaction. He discovered that the reaction could be modified for determining water in a non-aqueous system containing an excess of sulphur dioxide in the presence of a suitable base. He used a primary alcohol (methanol) as the solvent and a base (pyridine) as the buffering agent.

### What is the Karl Fischer Reaction?



Reaction (1) is where the alcohol reacts with sulphur dioxide (SO<sub>2</sub>) and base to form an intermediate alkylsulfite salt



Reaction (2) is the oxidation step where the intermediate is then oxidised to an alkylsulphate salt. This reaction consumes water.

Water and iodine are consumed in a 1:1 ratio. Once all of the water present is consumed, the presence of excess iodine is detected by the indicator electrode. That signals the end-point of the titration. The amount of water present in the sample is calculated based on the concentration of iodine in the Karl Fischer titrating reagent and the amount of Karl Fischer Reagent consumed in the titration.

Depending on the type of base used the rate of reaction will vary. Classic Karl Fischer reagents contained pyridine, a noxious carcinogen, as the base. Due to its weak basicity the reaction is slow and the end point is not stable. The most common base used today is imidazole where greatest accuracy and repeatability can be achieved.

Coulometry is an absolute technique so standardisation of Karl Fischer reagents is not necessary.

### Coulometric Titration Method

In coulometric Karl Fischer, iodine is generated electrochemically *in situ* during the titration.

The anolyte contains iodide, sulphur dioxide, bases and solvents such as methanol. Electrolytic oxidation occurs when a sample is added to this anolyte.

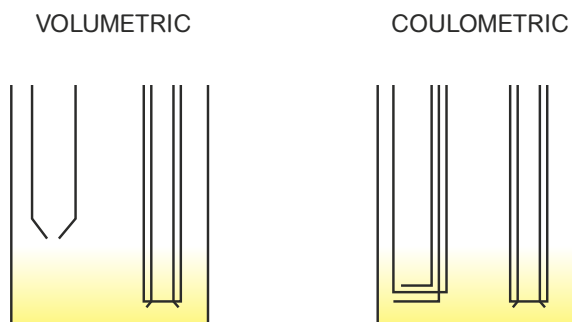
Iodine is generated at the anode of the titration cell as shown in formula (3)



Stoichiometrically, 1 mole of water will react with 1 mole of iodine, so that 1 milligram of water is equivalent to 10.71 coulombs of electricity

## Comparison of volumetric and coulometric titration cells.

### K.F. Titration Cell Differences



In the volumetric technique, which is used to determine higher water contents generally at levels above 5%, the iodine is introduced via a burette or similar dosing system. For lower water content detection, down to ppm levels, the coulometric technique produces the iodine in-situ by electrolysis.

Coulometry improves the Karl Fischer technique by removing the need for reagent calibration and provides an absolute result in micrograms of water. Low reagent usage: multiple samples can be analysed with a single charge of reagent which means less waste and less handling of chemicals, good news for Health and Safety.

## Aquamax KF Reagent

Aquamax KF Reagent A is a general coulometric KF analyte for use with generator electrodes which incorporate a frit or diaphragm to separate the anode and cathode chambers. Used in conjunction with Reagent C this formulation is supplied in a pack of 8 x 100ml bottles, 8 x 5ml cathode vials, all conveniently located in a single carton.

To conform to ASTM, API, EI, ISO (plus others) methodology for water content determination of oil and petroleum products, the anode reagent must be modified with xylene to improve sample solubility and miscibility. Aquamax KF Reagent is pre-mixed with xylene, and other solubilizers to eliminate side reactions, so that the operator does not have to store or mix chemicals.

We conducted extensive comparison trials of Aquamax KF Reagents against other leading brands with very positive results. Using water standards ranging from 10 µg water up to 10,000 µg water under controlled conditions we found Aquamax KF A and KF C to be equivalent in performance accuracy to the other brands but much faster in titration speed and with lower background drift values. Aquamax KF Reagents, performed much faster than other special oil reagents and at the 10,000 µg count level (as required by ASTM methods), gave a result of 10,004 µg which is within 0.04%.

## Aquamax KF Reagents combine speed and accuracy

- Single shot bottles
- Safer to use
- Safer to store
- Low cost
- Faster precondition
- Reduced downtime
- Improved sample miscibility / solubility
- For use with all coulometers

## Ordering Information

Part No.	Product description
620000	Aquamax KF Reagent Kit (8 x 100ml anode plus 8 x 5ml cathode)

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