

# Karl Fischer Reagents and Standards



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## KARL FISCHER REAGENTS

#### **VOLUMETRIC** (Item - Description)

#### One Component Reagents

1600 - 5 mg/ml PYRIDINE-FREE SINGLE SOLUTION 1601 - 2 mg/ml PYRIDINE-FREE SINGLE SOLUTION 1884 - 1 mg/ml PYRIDINE-FREE SINGLE SOLUTION 1893 - 5 mg/ml SINGLE SOLUTION, NON-HAZ 1894 - 2 mg/ml SINGLE SOLUTION, NON-HAZ

#### One Component Solvents

1610 - SOLVENT, GENERAL PURPOSE

3569 - METHANOL, ANHYDROUS

1885 - METHANOL PRIME

1609 - SOLVENT, METHANOL-FREE, FOR ALDEHYDES & KETONES

2978 - SOLVENT FOR OILS, ONE-COMPONENT SYSTEM

#### Two Component Reagents

1604 - 5.0 mg/ml TITRANT, NON-HYGROSCOPIC

1603 - 2.0 mg/ml TITRANT, NON-HYGROSCOPIC

1602 - 1.0 mg/ml TITRANT, NON-HYGROSCOPIC

1970 - 0.5 mg/ml TITRANT, NON-HYGROSCOPIC

1616 - 5.0 mg/ml TITRANT IN METHANOL

#### Two Component Solvents

5322 - SOLVENT, for ALDEHYDES AND KETONES, WITH CHLOROFORM

2991 - SOLVENT FOR OILS, TWO-COMPONENT SYSTEM

1609 - SOLVENT, FOR ALDEHYDES & KETONES

1610 - SOLVENT, GENERAL PURPOSE

### COULOMETRIC

#### Vessel (Anolyte) Reagents

1607 - VESSEL SOLUTION, CFC FREE

1612 - VESSEL SOLUTION, PYRIDINE-FREE

1619 - VESSEL SOLUTION, FOR ALDEHYDES & KETONES

5202 - VESSEL SOLUTION, for OILS

1671 - VESSEL SOLUTION, DIAPHRAGMLESS, CHLOROFORM-FREE

1889 - VESSEL SOLUTION, OVEN

#### Generator Solutions

2321 - GENERATOR SOLUTION, UNIVERSAL

### **STANDARDS**

2303 - WATER STANDARD, 10.0 mg/g (10,000 ppm)

2304 - WATER STANDARD, 5.0 mg/g (5,000 ppm)

2302 - WATER STANDARD, 1.0 mg/g (1,000 ppm)

2301 - WATER STANDARD, 0.10 mg/g (100 ppm)

3493 - WATER STANDARD, 0.50 mg/g (500 ppm)

2311 - WATER STANDARD, 0.05 mg/g (50 ppm)

2385 - WATER STANDARD, POTASSIUM CITRATE

805 - SODIUM TARTRATE, DIHYDRATE REAGENT (ACS)

## **BUFFERING AND ADDITIONAL PRODUCTS**

1615 - BUFFER ACID

1973 - IMIDAZOLE, ACS

672 - BENZOIC ACID, ACS

963 - MOLECULAR SIEVE, 3A, 8 - 12 Mesh





## Watermark® One-Component

#### Volumetric Reagents

In one-component volumetric Karl Fischer Titration, a known concentration of iodine rich titer (Reagent) is added to the vessel solution (Solvent) to consume the water in the sample. By measuring the volume of the titer added, we can calculate the amount of water in the sample. In this method, the titrant contains all the ingredients required by the KF reaction: iodine, base, sulfur dioxide, and a solvent. The Watermark range includes titrants with titer values of 2 and 5 mg/mL of water in order to cover an array of applications.

#### Watermark® One-Component Titrants

The Watermark brand offers users a wide range of options for selecting the correct titrant. Thanks to the methanol-free formulation, all GFS KF volumetric titrants can be used for the determination of water in aldehydes and ketones.

## Advantages of Watermark® One-Component Titration:

- No changing titrants for specialty compounds...just the solvent!
- Fast and stable endpoints
- · Long shelf life
- Convenient and simple to use





## **VOLUMETRIC**

### **One Component Volumetric Reagents**

Product Number	Product Name
1600	5 mg/ml PYRIDINE-FREE SINGLE SOLUTION
1601	2 mg/ml PYRIDINE-FREE SINGLE SOLUTION
1884	1 mg/ml PYRIDINE-FREE SINGLE SOLUTION
1893	5 mg/ml SINGLE SOLUTION, NON-HAZ
1894	2 mg/ml SINGLE SOLUTION, NON-HAZ

DID You Know! Due to interfering side reactions caused by ketones and aldehydes, solvents with methanol cannot always be used in KF titrations. Watermark 1600 and 1601 (one component single solutions) are formulated to work with 1609 (a specially designed methanol-free solvent) in lieu of standard methanol, to quickly find moisture in ketones. Methanol-free solvents allow for faster titration times as well as being less hazardous than conventional methanol based solvents.

### **One Component Solvents**

Product Number	Product Name					
1610	SOLVENT, GENERAL PURPOSE					
3569	METHANOL, ANHYDROUS					
1885	METHANOL PRIME					
1609	SOLVENT, METHANOL-FREE, FOR ALDEHYDES & KETONES					
2978	SOLVENT FOR OILS, ONE-COMPONENT SYSTEM					





## Watermark® Two-Component Volumetric Reagents

For users who do frequent KF analysis and need a higher degree of accuracy than one-component volumetric titration can provide, two-component volumetric titration is the solution. The major differentiating factor between the two methods is that the components of the KF reaction are separated in two-component titration as opposed to being in the same bottle for one-component volumetric titration.

#### Advantages of Watermark® Two-Component Titration:

- Eliminates frequent standardizations
- Exact and stable titer strength
- Faster titrations
- Longer shelf life
- Greater accuracy for low water content

## Watermark® Non-Hygroscopic Titrants

Watermark non-hygroscopic titrants are designed to be exceptionally stable with a guaranteed minimum shelf life of 2 years. The Watermark line also offers the titrant with the lowest titer strength on the KF market: 0.5 mg/mL for samples of very low water content - perfect for labs that don't have a coulometric titrator. Our products maintain a titer strength of +/- 0.05 mg/mL, making them the most advanced and stable KF reagents available.



## **VOLUMETRIC**

### **Two Component Volumetric Reagents**

Product Number	Product Name						
1604	5.0 mg/ml TITRANT, NON-HYGROSCOPIC						
1603	2.0 mg/ml TITRANT, NON-HYGROSCOPIC						
1602	1.0 mg/ml TITRANT, NON-HYGROSCOPIC						
1970	0.5 mg/ml TITRANT, NON-HYGROSCOPIC						
1616	5.0 mg/ml TITRANT IN METHANOL						

**DID YOU KNOW!** Watermark non-hygroscopic titrants can be used with both methanol-based and methanol-free solvents (1609/1610). This allows for faster titrations as there is no need to change the titrant and clean the burette when switching to aldehyde and ketone analysis. Another key advantage of Watermark non-hygroscopic reagents is less frequent standardizations, providing users with increased accuracy.

## **Two Component Solvents**

	Product Number	Product Name				
5322 SOLVENT, for ALDEHYDES AND KETONES, WITH CHLOROFORM						
2	SOLVENT FOR OILS, TWO-COMPONENT SYSTEM					
-	1610	SOLVENT, GENERAL PURPOSE				
	1609	SOLVENT FOR ALDEHYDES & KETONES				



## COULOMETRIC

Known for its highly effective micro-determinations and automation, it's obvious why coulometric titration has found popular application in the KF determination of water. Watermark Karl Fischer Reagents are designed for use with most coulometric systems on the market. They are specially formulated to provide the ultimate in analytical performance.

#### Advantages of Watermark Water Standards:



- Lowest increment of detection (50x lower than volumetric)
- Ideal for small samples (<10 mg water)
- Auto-adjusts as the material reacts with water vapor in the air
- High accuracy

### Watermark® Coulometric Range: Performance

Watermark Coulometric reagents offer the best value and performance for Karl Fischer titration on the market today. With extended shelf life, rapid moisture determinations, and a stable end point, it's easy to see why we outperform the competition. Our custom analyte and catholyte solutions allow for faster and more accurate titrations than ever before, even when determining moisture content at microgram levels.





## COULOMETRIC

### Coulometric Vessel (Anolyte) Reagents

Product Number	Product Name						
1607	VESSEL SOLUTION, CFC FREE						
1612	VESSEL SOLUTION, PYRIDINE-FREE						
1619	VESSEL SOLUTION, FOR ALDEHYDES & KETONES						
5202	VESSEL SOLUTION, for OILS						
1671	VESSEL SOLUTION, DIAPHRAGMLESS, CHLOROFORM-FREE						
1889	VESSEL SOLUTION, OVEN						

**DID YOU KNOW!** The Watermark 1671 Fritless Reagent is both versatile and easy to use. Designed for use with fritless cells, Watermark 1671 can also be used with titrators that contain diaphragms or ceramic frits providing:

- Convenience of a single solution
- Accurate and reproducible results
- Fast pre-titration times
- Free of chlorinated hydrocarbons
- Suitable for all titrators and titration cell types

### **Coulometric Generator Solutions**

Product Number	Product Name
2321	GENERATOR SOLUTION, UNIVERSAL





#### Watermark® Reference Materials

Standardization of a KF reagent is necessary in order to determine its water equivalency. GFS is the primary manufacturer of a broad range of reference materials, all made in-house at our Columbus, Ohio facilities. The (liquid) water standards are delivered in boxes of 10 single-use ampoules with a shelf life of over 5 years. Watermark water standards are methanol-free and can be used for all applications. All standards come with a certificate of analysis.

### Advantages of Watermark Reference Materials:

- Manufactured according to ISO 9001:2008 & 17025
- NIST Traceable
- Single-use ampoules
- C of A included

Custom reference materials can also be made for applications requiring other levels of water content.





## STANDARDS

#### **Karl Fischer Standards**

Product Number	Product Name					
2303	WATER STANDARD, 10.0 mg/g (10,000 ppm)					
2304	WATER STANDARD, 5.0 mg/g (5,000 ppm)					
2302	WATER STANDARD, 1.0 mg/g (1,000 ppm)					
2301	WATER STANDARD, 0.10 mg/g (100 ppm)					
3493	WATER STANDARD, 0.50 mg/g (500 ppm)					
2311	WATER STANDARD, 0.05 mg/g (50 ppm)					
2385	WATER STANDARD, POTASSIUM CITRATE					
805	SODIUM TARTRATE, DIHYDRATE REAGENT (ACS)					

**DID YOU KNOW!** Watermark water standards are perfect tool to assist you in validation a new process as well as your titrator to meet GMP, ISO or other guidelines within your organization. Compare them to pure water and see the increased reproducibility they can provide.

## BUFFERING

### **Buffering & Additional Products**

Product Number	Product Name			
1615	BUFFER ACID			
1973	IMIDAZOLE, ACS			
672	BENZOIC ACID, ACS			
963	MOLECULAR SIEVE, 3A, 8 – 12 Mesh			



## WHITE PAPER

#### Water Determination in an Insecticide

#### Introduction

Water determination in powders can be performed through coulometric or volumetric Karl Fischer titration. Some of these materials do not dissolve in any of the common solvents used for Karl Fischer (KF) titration (i.e. methanol, 2-methoxyethanol, ethanol, etc.). Because powder materials can clog the frit of a coulometric KF unit, or because the user does not have access to this equipment, alternate methods must be found.

Anon-hygroscopic volumetric titrant of low titer strength (0.5 mg/mL (+/- 0.05 mg/mL) is specially designed for volumetric titrations of samples with low moisture content. This application note demonstrates use of the reagent and its efficacy for the determination of water.

The following equipment and reagents were used. Instrumentation: model DL38 volumetric titrator (Mettler-Toledo, Greifensee, Switzerland). Reagents: item #1970, non-hygroscopic titrant, 0.5 mg/mL; item #1610, general purpose solvent, methanol-based; item #2302, water standard, 1.00 mg/g; and item #1604, non-hygroscopic titrant, 5 mg/mL.

All reagents were from GFS Chemicals, Inc. (Powell, OH).

#### **Procedure**

The solvent was changed after each determination. Because the titrant is of very low strength (0.5 mg/mL), pre-titration was accelerated by adding item #1604 (5 mg/mL titrant) to the cell using a syringe. Addition of the titrant ceased when the drift of the titrator decreased to approximately 100 microgram per milliliter.

The following parameters were used in the DL38 titrator method – current (microampere): 20; endpoint (mV): 100; dV min(microliter): 3.0; dV max (microliter): 8.0; dV max factor (%): 40; drift stop: relative drift (microgram per minute): 50.

Titer determination was performed by using a 1.00 mg/g water standard. The standard was first checked by coulometric titration at 0.99 mg/g.

Results for the titer determination (three titrations) are as follows:

Concentration: 0.473 mg/mL; standard deviation: 0.005 mg/mL; relative standard deviation: 0.96%.



## WHITE PAPER

#### Determination of the water in the insecticide

The water content of this insecticide was known to be fairly low, and this powder is not soluble in methanol. To check method validity and to obtain better accuracy, water determination was performed by spiking samples with various known amounts of water (from item #2302). The same method as that used for the titer determination was used, but this time with a stir time of 60 sec. When spiking the sample, the known amount of water standard was added to the vessel during the stirring time, after addition of the sample. The results are shown in Table 1.

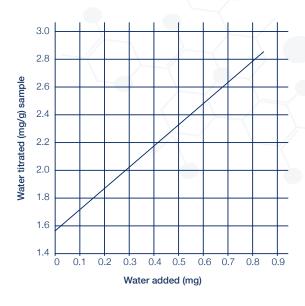
Table 1 **Titration results: Four samples spiked with various amounts of water** 

Experiment No.	Sample size (g)	Water Spike (mg)	Titration volume (mL)	Amt of water titrated per gram of sample (mg/g)
1	0.530	0.000	1.501	1.33
2	0.585	0.850	3.576	2.87
3	0.570	0.505	2.873	2.37
4	0.600	0.250	2.518	1.97

The water titrated in each sample, per gram of sample, is proportional to the known amount of water added. Therefore, the intercept of the graph in Figure 1 (water titrated per gram of sample vs.water added) is the amount of water initially present in the sample, per gram of sample.

The following parameters can be calculated. Equation: Y = 1.5704 X + 1.5549; regression parameter: R2=0.998. Y is the water titrated (in mg) per gram of sample, and R is the correlation coefficient. The R2 value (close to 1) indicates the efficiency and validity of the method. Water in the sample can be estimated to be 1.555 mg/g.

Originally published in American Laboratory News, March, 2004 and submitted by Jerome P. Bozon, GFSChemicals,Inc.





## SPECIALTIES

GFS Chemicals is a US based manufacturer of specialty and fine chemicals serving customers worldwide since 1928. As an ISO 9001:2015 and an ISO 17025:2017 manufacturer, GFS' manufactures and offers a vast variety of materials for use in the research, analytical and manufacturing laboratories. Besides Karl Fischer Reagents, some of our specialties include:

- ACS Reagents
- Veritas® HPLC Solvents
- HPLC/GC/UV-Vis Solvents
- Reagent Grade Solvents
- LC-MS Solvents
- LC-MS Eluents
- Spectroscopy Products
- Primary Standard Salts
- Normality Solutions
- Dyes and Indicators
- Reagents for Water & Wastewater Labs
- Organic Halogen ReagentOHR/Halogen Level Field Test Kits
- Conductivity Standards and pH Buffers
- AA and ICP Standard Solutions
- Standard Solutions for Trace Metal Analysis

For more information about GFS Chemicals, please visit: www.gfschemicals.com or call GFS at (800) 858-9682.



## **ABOUT GFS**

GFS Chemicals is a Columbus, OH, USA based ISO 9001:2015 certified manufacturer. GFS' capability to produce specialty alkynes and olefins, pharmaceutical building blocks, trace metal salts and solutions, low moisture/anhydrous salts, and specialty rare earth salts and solutions makes GFS a preferred partner for organizations across an array of industries. GFS has delivered results in demanding markets – including: pharmaceutical, electronics, energy, flavors & fragrances, agrichemicals, and specialty polymers. From research lab to pilot plant, from production to commercialization, GFS Chemicals is here to give you knowledgeable answers to any of your technical questions.

In addition to GFS' proprietary product portfolio of 8000 discrete high-quality products available from small bottles to drum quantities, GFS offers:

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