

API Corporation

A subsidiary of  MITSUBISHI CHEMICAL CORPORATION

AQUAMICRON

KARL FISCHER REAGENTS



What is the Karl Fischer Method?

As shown in Formula (1) below, the Karl Fischer method uses Karl Fischer reagent, which reacts quantitatively and selectively with water, to measure moisture content. Karl Fischer reagent consists of iodine, sulfur dioxide, a base and a solvent, such as alcohols.



As described below, this method can be used in both volumetric and coulometric titration systems.

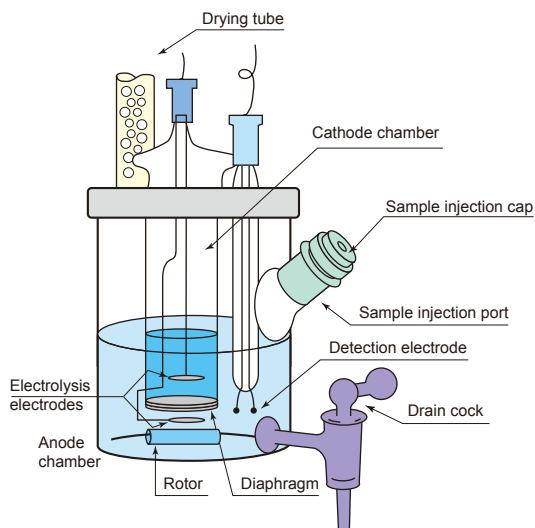
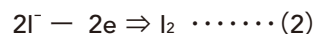


Figure1: Electrolysis cell of Coulometric Moisture Meter

Coulometric Titration

With coulometric titration, the sample is added to an electrolytic solution, the main constituents of which are iodide ions, sulfur dioxide, a base, and a solvent (such as alcohol). Electrolytic oxidation causes the production of iodine, as shown in Formula (2), resulting in an immediate Karl Fischer reaction.



According to Faraday's laws, the iodine is produced in proportion to the quantity of electricity. This means that the water content can be determined immediately from the coulombs required for electrolytic oxidation.

1mg of water = 10.71 Coulombs

As shown in Figure 1, two types of coulometric reagents are required: an anolyte, which is placed in the anode chamber of the electrolysis cell, and a catholyte, which is placed in the cathode chamber. Coulometric reagents do not need assessment. Another advantage is that they can be used repeatedly.

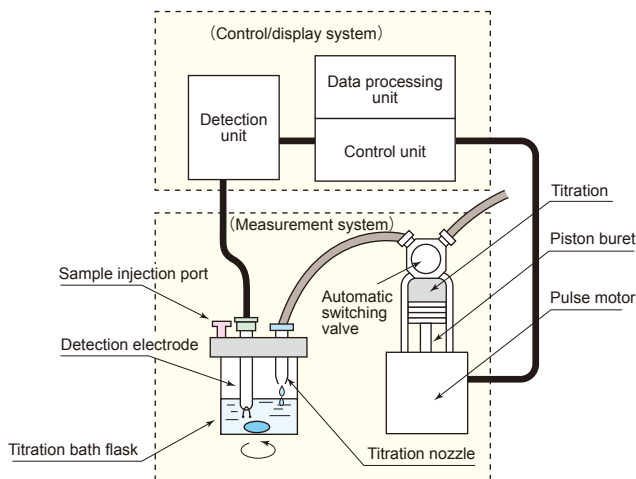


Figure2: Volumetric Moisture Meter

Volumetric Titration

A dehydrating solvent suitable for the sample is placed in a flask. Titrant is used to remove all moisture from the solvent. The sample is then added. Titration is carried out using a titrant, the titer (mgH₂O/mL) of which has previously been determined. The moisture content of the sample is determined from the titration volume (mL). The end point is detected using the constant-current polarization voltage method. Figure 2 shows the components of typical commercially available automatic volumetric titration system.

Mitsubishi Chemical Group API Corporation started in October, 2002, and succeeded to the KF reagents business. We provide excellent reagents, services and technical support for our customer satisfaction.

Our KF reagents, AQUAMICRON® is manufactured at Mitsubishi Chemical Corporation Kashima Plant which have quality systems that adhere to the requirements of ISO 9001 and have received third party accreditation.

AQUAMICRON series



AQUAMICRON® is a registered trademark of Mitsubishi Chemical Corporation.



Scope of Applications for Karl Fischer Method

The Karl Fischer method can be used with a wide range of substances. However, the method depends on a kind of iodometry based on the reaction between the Karl Fischer reagent and water. The results will therefore show a positive error if the sample includes substances that react with iodine, and a negative error if the sample includes substances that produce iodine by oxidizing iodide compounds. The following tables list substances that can be titrated directly with Karl Fischer reagents, substances that cannot be titrated directly but can be titrated by means of adjustments based on appropriate chemical reactions or processes, and substances that react with Karl Fischer reagents and are therefore unsuitable for direct titration. Even if a substance cannot be titrated directly, its moisture content can be measured by indirect means, such as the water vaporization method. Please contact mckkf.com for further information.

Compounds that can be titrated directly

Organic compounds Hydrocarbons(saturated, unsaturated compounds) Alcohols, Polyhydric alcohols, Phenols, Ethers Inert ketones(diisopropyl ketone, et al) Inert aldehydes(formaldehyde, chloral, et al) Organic acids, Hydroxylic acids, amino acids Acid anhydrides Esters, Lactones, Inorganic acid esters Amines(<pKa9), Amino alcohols Proteins, Amides, Anilides Nitriles, Cyanhydrins, Cyanic acid derivatives	Nitro compounds, Oximes, Hydroxamic acids Thiocyanate, Thioethers, Thioesters Halogenated hydrocarbons, Halogenated acyls Sugars, Organic salts and their hydrates Inorganic compounds Inorganic salts and their hydrates Inorganic acids Chelate compounds Fertilizers Calcium carbonate Polytungsten salts
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Compounds that react with Karl Fischer reagents and cannot be titrated directly

Organic compounds Ascorbic acid, Diacyl peroxides Peracids, Quinone Inorganic compounds Sodium sulfide, Sodium peroxide Chromates, Dichromates	Ferric oxide, Nickel oxide, Arsenic trioxide Arsenates, Arsenites, Borates, Boron oxide Hydrogencarbonates, Carbonates Metal hydroxides, Metal oxides Sulfites, Pyrosulfites Sodium nitrite, Thiosulfates Cupric salts, Stannous salts
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Compounds that can be titrated directly after special processing or under certain conditions

The examination will be necessary for the following compounds though it was said that these could be titrated directly by the following treatments in former references.

Compounds	Treatment
Ammonia	Add acetic acid.
Ferric salts	Add 8-hydroxyquinoline.
Hydrazine derivatives	Add acetic acid.
Hydroxylamine salts	Add sulfur dioxide: pyridine solution (1:1).
Thiol(mercaptan)	Prevent interference by adding olefins(octene, etc.).
Sulfuric acid	If the sulfuric acid is 92%pure or higher, add a large surplus of pyridine and titrate it as a salt.
Thioacid	Prevent interference by adding olefins(octene, etc.).
Thiourea	Prevent interference by adding olefins(octene, etc.).

Examples of how AQUAMICRON® can be used with compounds that cause interference

Compound	Interference reaction	Treatment	
		Volumetric titration	Coulometric titration
Ketones	React with methanol to produce ketal and water	Titrant SS-Z + Solvent KTX ----- Titrant SS + Solvent CP	AKX + CXU
Aldehydes	React with methanol to produce acetal and water React with sulfur dioxide and water	Titrant SS-Z + Solvent KTX ----- Titrant SS + Solvent CP	AKX + CXU (suitable only for a certain aromatic aldehydes)
Lower carboxylic acids	React with methanol to produce ester and water	Titrant SS-Z + Solvent KTX ----- Titrant SS + Solvent CP	AKX + CXU
Amines above pKa 9	Consume iodine gradually and the end-point becomes unstable	Titrant SS-Z + Solvent GEX salicylic acid 10g ----- Titrant SS + Solvent MS salicylic acid 10g	AXI or AX + CXU salicylic acid 10g

Using a Water Vaporization System

A Karl Fischer moisture measurement system can be used in conjunction with a water vaporization system to measure the moisture content of substances and industrial products that are not suitable for direct titration. These include polymers such as plastics and rubber, new materials such as carbon fiber, electronic materials such as printed circuit boards and wafers, and printing materials such as toners. Coulometric reagents are especially suitable for the measure the moisture content of samples that contain interference substances, such as petroleum products containing additives (e.g. lubricating oils).



Coulometric Reagents

There are two types of coulometric reagents: the anolyte (generating solution), which is placed in the anode chamber of the electrolysis cell, and the catholyte (counter electrolyte), which is placed in the cathode chamber. There are also special anolyte for use with ketones, lower carboxylic acids and silicone oils. AQUAMICRON® can be used in coulometric moisture measurement systems sold by various companies, and it has a worldwide reputation for excellent performance.

AQUAMICRON® Series

Product name	Code No.	Specification	Package	Main solvent	Use
AQUAMICRON®	AXI	XAMI	500mL glass bottle	Methanol, Propylenecarbonate	*AXI [For frit or fritless type cell, equivalent to FLS] *AXI, AX & AX01 [For General-use] Organic solvents, Inorganic chemicals, Oiles, Petroleum products, Virious kind of gases, etc.
	AX	XAMA			
	AX01	XAMA01			
	AS	SAMA	500mL glass bottle	Methanol, Chloroform	[For Oils] Naphtha, Gasolone, Diesel oil, Electrical insulation oil, etc.
	AKX	AKX	500mL glass bottle	Propylenecarbonate, Diethyleneglycole monoethylether	[For Ketones] Ketones. Silicone oils. Low carboxylic acids, etc.
	CXU	CXU	5mL ampule x 10/case	Methanol	[Catholyte] Both AX and AS and AKX available to combine with
	FLS	FLS	500mL glass bottle	Methanol, Propylenecarbonate	[For fritless type cell] Organic solvents, Inorganic gases, etc.

AQUAMICRON® AXI/CXU or AX/CXU

Use: General samples

Characteristics:

Minimal environmental contamination
Wide range of applications

High quality, high performance

Easy to use

- Does not include carbon tetrachloride or chloroform
- Suitable for petroleum products
- Can be used with moisture vaporization method (When using AQUAMICRON® AXI or AX in a moisture vaporization method, replenish the vaporized portion with methanol.)
- Excellent reputation for accurate moisture measurement and end-point stability Approximately 800mg of moisture can be measured per 100mL of AQUAMICRON® AXI or AX, and approximately 150mg per 5mL of AQUAMICRON® CXU
- These products can be used in the existing coulometric moisture measurement system.

AQUAMICRON® AS/CXU

Use: General samples

Characteristics:

Wide range of applications
High quality, high performance

Easy to use

- Especially suitable for oils, petroleum products
- Excellent reputation for accurate moisture measurement and end-point stability.
- Approximately 800mg of moisture can be measured per 100mL of AQUAMICRON® AS, and approximately 150mg per 5mL of AQUAMICRON® CXU.
- These products can be used in the existing coulometric moisture measurement system.

AQUAMICRON® AKX/CXU

Use: Ketones, lower carboxylic acids, some aldehydes (aromatics)

Characteristics:

Minimal environmental contamination
Wide range of applications
High quality, high performance

Easy to use

- Does not include chloroform or methyl cellosolve
- Suitable for ketones, lower carboxylic acids and some aldehydes
- Accurate moisture measurement, good end-point stability
- Approximately 300mg of moisture can be measured per 100mL of AQUAMICRON® AKX, and approximately 100mg per 5mL of AQUAMICRON® CXU (when used with AQUAMICRON® AKX).
- These products can be used in the existing coulometric moisture measurement system.

AQUAMICRON® AXI or FLS

Use: Suitable for alcohols, esters, benzene, toluene, inorganic gases, etc.

Characteristics:

Easy to use

Easy maintenance

- AQUAMICRON® AXI or FLS is a single-solution type reagent. It should be used in single-solution electrolytic cells without diaphragm, fritless type cell.
- Can be used with moisture vaporization method (When using AQUAMICRON® AXI or FLS in a moisture vaporization method, replenish the vaporized portion with methanol.)
- Ceramic diaphragm is not used, therefore it is easy to maintain electrolytic cell in good condition.



Examples of sample measurement using AQUAMICRON® AXI/CXU or AX/CXU.

Sample Name	AQUAMICRON® AXI/CXU		AQUAMICRON® AX/CXU	
	Measured Moisture	RSD, %	Measured Moisture	RSD, %
Toluene	82.4 (1min)	0.27	84.0 (1.7min)	1.2
Hexane	33.8 (0.7min)	0.5	33.9 (1.4min)	1.7
Ethyl acetate	227 (1.3min)	0.52	227 (2.1min)	0.19
1-Propanol	834 (1.5min)	1.2	831 (2.5min)	0.6
Ethylene glycol	227 (1.3min)	0.68	222 (1.9min)	1.9
N-Methyl pyrrolidone	670 (1.6min)	0.3	671 (2.6min)	0.26
Form amide	536 (1.5min)	0.61	533 (2.3min)	0.42
Solid water standard (KF-oven method)	3.76% (14-17min)	0.66	3.76% (14-15min)	1.5

Instrument: CA-200 Moisture Meter (Mitsubishi Chemical Analytech) Sample size: 0.7-1.7g, 0.08g at KF-oven method
 Reagent: AQUAMICRON® AXI or AX 100mL, AQUAMICRON® CXU 5mL
 Average value for n=3 Unit: ppm unless noted as %

Examples of sample measurement using AQUAMICRON® AKX/CXU.

Sample Name	AQUAMICRON® AKX/CXU
	Measured Moisture
Formic acid	unable to measure
Acetic acid	153
Propionic acid	659
Acetone	0.166%
Acetyl acetone	270
Methyl ethyl ketone	771
Methyl isobutyl ketone	626
Cyclohexanone	330
3-Phenyl propion aldehyde	0.226%
Chloral	249
Benzaldehyde	483
Salicylaldehyde	0.116%

Instrument: CA-200 Moisture Meter (Mitsubishi Chemical Analytech) Sample size: 0.5-1.0g
 Reagent: AQUAMICRON® AKX 100mL, AQUAMICRON® CXU 5mL
 Average value for n=3 Unit: ppm unless noted as %



Examples of sample measurement using AQUAMICRON® AXI or FLS

Sample Name	AQUAMICRON® AXI		AQUAMICRON® FLS	
	Measured Moisture	RSD, %	Measured Moisture	RSD, %
Methanol	91.8	1.27	93.4	0.95
Ethanol	299	0.79	310	0.26
Toluene	46.7	4.55	49.7	1.55
Hexane	29	4.03	30.7	2.68
Ethyl acetate	14.2	1.5	145	0.46
Methyl cellosolve	168	1.13	172	1.49
Ethylene glycol	97.8	7.47	96.3	3.16
Acetonitrile	94	1.85	95.1	3.59
N,N-Dimethyl formamide	837	0.05	851	0.32

Instrument: CA-200 Moisture Meter with single solution type electrode (Mitsubishi Chemical Analytech) Sample size: 0.5-2.0g
 Reagent: AQUAMICRON® AXI or FLS 100mL Average value for n=3 Unit: ppm unless noted as %

Sample Name (KF-oven method)	AQUAMICRON® AXI		AQUAMICRON® FLS	
	Measured Moisture	RSD, %	Measured Moisture	RSD, %
Polyethylene terephthalate	0.46%	0.22	0.46%	2.6
Polystyrene	245	1.18	238	2.5
Nylon6,6	2.35%	0.27	2.42%	1.01

Instrument: CA-200 with single solution type electrode + VA-200 (Mitsubishi Chemical Analytech)
 Sample size: 0.05-0.2g at KF-oven method
 Reagent: AQUAMICRON AXI or FLS 150mL Average value for n=3 Unit: ppm unless noted as %

Volumetric Reagents

The items required for volumetric titration are a Karl Fischer titrant and methanol or a dehydrated solvent (used to dissolve or disperse the sample).

AQUAMICRON® offers a wide range of products suitable for measuring the moisture content of various samples at every levels. Users can select a solvent that is suitable for the samples based on the following tables.

SS-Z Series (pyridine-free and chloroform-free type)

Product name	Code No.	Specification	Package	Main solvent	Use
AQUAMICRON®	Titant SS-Z	SSZ10M Titer 0.7-1.2mgH ₂ O/mL	500mL glass bottle	Diethyleneglycole monoethylether	[For General-use] Low moisture content Samples
		SSZ30M Titer 2.5-3.5mgH ₂ O/mL	500mL glass bottle	Diethyleneglycole monoethylether	[For General-use]
		SSZ50M *SSZ50L Titer 4.5-5.5mgH ₂ O/mL	500mL, *1L glass bottle	Diethyleneglycole monoethylether	[For General-use] High moisture content Samples
	Solvent GEX	GEX Moisture maximum 0.2mgH ₂ O/mL	500mL glass bottle	Methanol	[For General-use] Organic solvents, Inorganic chemicals, Agricultural chemicals, Pharmaceuticals, Fertilizers, Detergents, Foodstuffs, etc.
	Solvent OLX	OLX Moisture maximum 0.5mgH ₂ O/mL	500mL glass bottle	Hexanol, Ethanol	[For Oils] Naphtha, Gasolone, Diesel oil, Electrical insulation oil, etc.
	Solvent OLII	OL2 Moisture maximum 0.3mgH ₂ O/mL	500mL glass bottle	Chloroform, Methanol	[For Oils and Fats] Naphtha, Gasolone, Diesel oil, Heavy oil, Electrical insulation oil, Oils and Fats(Hardened oil, Margarine, etc.),etc.
	Solvent KTX	KTX Moisture maximum 0.5mgH ₂ O/mL	500mL glass bottle	Propylenecarbonate, Diethyleneglycole monoethylether	[For Ketones] Ketones, Silicone oils, Acetic and other lower carboxylic acids, aldehydes(except acetaldehydes),etc.
	Solvent SU	SSU Moisture maximum 0.2mgH ₂ O/mL	500mL glass bottle	Formamide, Methanol	[For Sugars] Sugars, Proteins, Gelatine, Additives, Animal feeds, etc.

When using a moisture vaporization method, mix Solvent GEX with propylene glycol(PG) in a 3:1 ratio (e.g. 90mL of GEX + 30mL of PG).

When using Solvent KTX, it will be needed to make proper adjustments to the parameters. Please contact a manufacturer of the instruments.

An addition of 3g of salicylic acid enables the time for dehydration to get shorter at the first measurment in combined use of 50mL of Solvent SU.

SS Series (pyridine type)

Product name	Code No.	Specification	Package	Main solvent	Use
AQUAMICRON®	Titant SS	GKS10M Titer 0.7-1.2mgH ₂ O/mL	500mL glass bottle	Chloroform, Pyridine	[For General-use] Low moisture content Samples
		GKS30M Titer 2.5-3.5mgH ₂ O/mL	500mL glass bottle	Chloroform, Pyridine	[For General-use]
		GKS100M Titer 8-12mgH ₂ O/mL	500mL glass bottle	Chloroform, Pyridine	[For General-use] High moisture content Samples
	Solvent ML	GML Moisture maximum 0.2mgH ₂ O/mL	500mL glass bottle	Methanol	[For General-use] Organic solvents, Inorganic chemicals, Agricultural chemicals, Pharmaceuticals, Fertilizers, Detergents, Foodstuffs, etc.
	Solvent MS	GMS Moisture maximum 0.2mgH ₂ O/mL	500mL glass bottle	Methanol, Pyridine	[For General-use] Organic solvents, Inorganic chemicals, Agricultural chemicals, Pharmaceuticals, Fertilizers, Detergents, Foodstuffs, etc.
	Solvent CM	GCM Moisture maximum 0.3mgH ₂ O/mL	500mL glass bottle	Chloroform, Methanol	[For Oils] Naphtha, Gasolone, Diesel oil, Heavy oil, Electrical insulation oil, Oils and Fats(Hardened oil, Margarine, etc.),etc.
	Solvent CP	GCP Moisture maximum 0.5mgH ₂ O/mL	500mL glass bottle	Chloroform, Propylenecarbonate	[For Ketones] Ketones, Silicone oils, Acetic and other lower carboxylic acids, aldehydes(except acetaldehydes),Anilines, etc.
	Solvent PP	GPP Moisture maximum 0.2mgH ₂ O/mL	500mL glass bottle	Pyridine, Propylene glycole	[For Aldehydes] Acetaldehydes, propionaldehydes, Butyraldehydes, etc.
	Solvent PE	GPE Moisture maximum 0.2mgH ₂ O/mL	500mL glass bottle	Pyridine, Ethylene glycole	[For Ketones] Ketones, Silicone oils, Acetic and other lower carboxylic acids, aldehydes(except acetaldehydes),Anilines, etc.
	Solvent FM	GFM Moisture maximum 0.2mgH ₂ O/mL	500mL glass bottle	Formamide, Methanol	[For Sugars] Sugars, Proteins, Gelatine, Additives, Animal feeds, etc.
	Solvent ME	GME Moisture maximum 0.2mgH ₂ O/mL	500mL glass bottle	Methanol, Ethylene glycole	[For Vaporizer] Gaseous samples, Nitrogen, etc.

When using a moisture vaporization method, mix Solvent MS with propylene glycol(PG) in a 3:1 ratio (e.g. 90mL of MS + 30mL of PG).

An addition of 3g of salicylic acid enables the time for dehydration to get shorter at the first measurment in combined use of 50mL of Solvent FM.

Examples of sample measurement.

Apparatus: Volumetric Moisture Meter KF-100 (Mitsubishi Chemical Corporation)

General samples

Sample Name	Sample Amount(g)	SS-Z 5mg	SS 3mg	Composite 5mg
		Solvent GEX	Solvent MS	Solvent GEX
Methanol	4.0	627ppm	622ppm	630ppm
Ethanol	3.8	524ppm	521ppm	530ppm
Isopropanol	4.0	227ppm	228ppm	226ppm
Ethylene glycol	5.5	237ppm	236ppm	242ppm
Propylene glycol	5.0	167ppm	164ppm	164ppm

Amines

Sample Name	Sample Amount(g)	SS-Z 5mg	SS 3mg	Composite 5mg
		(GEX+Salicylic acid)	(MS+Salicylic acid)	(GEX+Salicylic acid)
Diethanolamine	3.0	0.126%	0.128%	0.128%
Monoethanolamine	1.8	0.291%	0.301%	0.302%
Di-n-butylamine	0.8	0.221%	0.218%	0.224%

Oils

Sample Name	Sample Amount(g)	SS-Z 5mg	SS 3mg	Composite 5mg
		Solvent OLX	Solvent CM	Solvent for Oil
Salad oil	9.5	362ppm	359ppm	356ppm
Olive oil	10	409ppm	396ppm	401ppm

Sugars

Sample Name	Sample Amount(g)	SS-Z 5mg	SS 3mg	Composite 5mg
		Solvent SU	Solvent FM	Solvent SU
Instant coffee	0.1	2.23%	2.12%	2.23%
Honey	0.02	17.3%	17.2%	17.7%
Cream powder	0.1	2.94%	2.94%	2.94%

Ketones

Sample Name	Sample Amount(g)	SS-Z 5mg	SS 3mg	Composite 5mg
		Solvent KTX	Solvent CP	Solvent for Ketone
Acetone	3.9	0.162%	0.167%	0.173%
Methyl ethyl ketone	2.2	610ppm	619ppm	678ppm
Acetylacetone	4.7	461ppm	465ppm	525ppm
Cyclohexanone	0.8	755ppm	730ppm	890ppm

General samples

Sample Name	Sample Amount(g)	SS-Z 1mg	Composite 1mg
		Solvent GEX	Solvent GEX
Toluene	4.6	139ppm	139ppm
Xylene	4.1	168ppm	170ppm
Chloroform	15	44ppm	44ppm

Oils

Sample Name	Sample Amount(g)	SS-Z 1mg	Composite 1mg
		Solvent OLX	Solvent for Oil
Turbine oil	8.6	41ppm	38ppm
Insulating electric oil	8.9	39ppm	38ppm



SS-Z series



SS series

Water Standards

Reliable reference materials are necessary to standardize reagents, and to check the titrator according to the requirements of ISO, ASTM, JIS guidelines.

AQUAMICRON® offers a series of excellent standards that have advantages: low toxicity, easy to use and to manage. To ensure the high quality, all of our Water Standards are manufactured under strict control and accurately measured using validated procedures traceable to the international prototype at the BIPM in France.

Also AQUAMICRON® Liquid Water Standard (AWS01,02,10,100) is directly traceable to NIST SRM 2890.

Liquid Water Standard (with Test Certification)

Product name	Code No.	Specification	Package	Main solvent	Use
AQUAMICRON® Water Standard 0.1	AWS01	Titer $0.1 \pm 0.01 \text{ mgH}_2\text{O/g}$	5mL ampule ×10/case	Propylenecarbonate	For checking the accuracy of the coulometric moisture meter
AQUAMICRON® Water Standard 0.2	AWS02	Titer $0.2 \pm 0.01 \text{ mgH}_2\text{O/g}$	5mL ampule ×10/case	Propylenecarbonate	For checking the accuracy of the coulometric moisture meter
AQUAMICRON® Water Standard 1	AWS10	Titer $1 \pm 0.05 \text{ mgH}_2\text{O/g}$	5mL ampule ×10/case	Propylenecarbonate	For checking the accuracy of the coulometric moisture meter
AQUAMICRON® Water Standard 10	AWS100	Titer $10 \pm 0.5 \text{ mgH}_2\text{O/g}$	8mL ampule ×10/case	Propylenecarbonate	For determination of Karl Fischer reagent titer

Solid Water Standard (with Test Certification)

Product name	Code No.	Specification	Package	Main solvent	Use
AQUAMICRON® Solid Water Standard	SWS	Moisture $3.83 \pm 0.1\%$	10g glass bottle	Potassium tetrataretrate hemihydrate	For evaluation a moisture meter with a water vaporizer

Check Solution

Product name	Code No.	Specification	Package	Main solvent	Use
AQUAMICRON® Check Solution P	GCHP	Moisture $4.0 \pm 0.2 \text{ mgH}_2\text{O/mL}$	100mL glass bottle with septum cap	Propylenecarbonate	The end-point adjustment solution can be used with both AX and AXK. It can also be used for day-to-day management of coulometric moisture measurement systems.

Liquid Standard for assess titer

Product name	Code No.	Specification	Package	Main solvent	Use
AQUAMICRON® Standard Water/Methanol	GMW20	$2.0 \pm 0.04 \text{ mgH}_2\text{O/mL}$	250mL glass bottle	Methanol	Used to assess titer of Karl Fischer reagents ($3\text{--}10 \text{ mgH}_2\text{O/mL}$). Also can be used to reverse titration.



API Corporation

Tokyo Head Office
2-3-4, Nihonbashi, Chuo-ku, Tokyo 103-0027, Japan
Telephone: +81-3-5205-0632 Facsimile: +81-3-5205-0641
Email: mcckf@cc.m-kagaku.co.jp

AQUAMICRON® Information
● Home Page <http://www.mcckf.com/english>

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