Determination of Water Content in Alcohols

This application method is applicable for the determination of water in alcohols.
However, this method can be used for many kinds of the organic solvents.

Titrator	AQV-2000/AQV-200 (Volumetric titrator) AQ-2000/AQ-200 (Coulometric titrator)	
KF Oven	N/A	
Reagents for Volumetric Titrator	Titrant	HYDRANAL^{R} Composite 5 or equivalent
	Titration medium	Dehydrated methanol
Reagents for Coulometric Titrator	Anode solution	HYDRANAL^{R} Coulomat AG or equivalent
	Cathode solution	HYDRANAL^{R} Coulomat CG or equivalent

PARAMETER SETTINGS for the Titrator

Volumetric Titrator		**Coulometric Titrator**	
End Mode	4	CAL Mode	0
CAL Mode	0	Interval	20 sec
Interval	20 sec	Current	Fast
MIN Feed	0.01 ml	S-timer	0 min
S-timer	0 min	T-timer	0 min
T-timer	0 min		

PARAMETER SETTINGS for Oven

Temperature	-
Carrier flow rate	-

PROCEDURE:

1. Fill a 5 ml glass syringe with the sample. Before filling, rinse the syringe with the sample 2 – 3 times.
2. Tiitrate the titration medium to zero. (Blanking)
3. Press SAMPLE key.
4. Inject approx. 1 ml of the sample into titration cell by piercing the rubber septum.
5. Press TITRATION key.
6. Press S.SIZE key and enter the sample size.

OPERATING NOTES:

1. The measurement of water in alcohols is easy. Pay attention not to absorb the atmospheric moisture.

2. Stop the end of the syringe needle with a small silicon block. This prevents the loss of the sample caused by evaporation.

3. After injection of the sample, pull slightly the syringe plunger to suck the sample drops sticking on the end of syringe needle.

4. Injected sample size is calculated by subtracting AFTER injection syringe weight from BEFORE injection syringe weight.

5. Pierce different part of the rubber septum for each injection. If the same part is pierced for many times, atmospheric moisture will get into the titration cell, which will result in a high background or taking a long time for blanking.

6. To get the most accurate result, the titrant should be consumed around 5 ml. To make titrant consumption into this range, calculate the optimum sample size by this equation ;

   SIZE (g) = 5 x F / (C x 10)

   Where ;

   F : Factor of titrant. ex. Factor of Composite 5 is 5 (mgH_2O/ml).
   C : Sample moisture in %

   For example, sample moisture is 1%, and Factor is 5, then optimum sample size is,

   5 x 5 / (1 x 10) = 2.5g

HIRANUMA APPLICATION DATA	Karl Fischer Titrator	Data No.	KF1	Mar. 8 2018

Water contents

Hydrocarbon, Halogenated hydrocarbon

1.Abstract

Water content of hydrocarbon and halogenated hydrocarbon could be determined by Karl Fischer coulometric titrator. In coulometric titration, iodine of Karl Fischer reagent is generated by electrolysis and generated iodine quantitatively reacts with water. Reaction formula is described below.

$$H_2O + I_2 + SO_2 + 3RN + CH_3OH \rightarrow 2RN \cdot HI + RN \cdot HSO_4CH_3$$

$$2RN \cdot HI \rightarrow I_2 + 2RN + 2H^+ + 2e^-$$

Hydrocarbon and halogenated hydrocarbon do not interfere the Karl Fischer reaction and direct injection method could apply. Anode solution is selected in accordance with sample solubility. General use of anode solution contain methanol as solvent. When the sample like a long chain hydrocarbon have poor solubility in methanol, anode solution containing chloroform or hexanol or toluene is used. When fritless cell is used, cathode solution is not necessary.

2. Apparatus and Reagents

(1) Apparatus

Titrator	:	HIRANUMA Karl Fischer Coulometric titrator AQ-series or MOICO-A19
Electrolytic cell	:	Standard Cell
		Fritless Cell

(2) Reagents

Anode solution	:	Hydranal coulomat AG (Honeywell)
Cathode solution	:	Hydranal coulomat CG (Honeywell)

3. Procedure

(1) Fill 100 mL of anode solution and one ampoule of cathode solution into the electrolytic cell as shown in Fig.3.1.
(2) Start blanking to attain stable background.
(3) Wash the syringe with sample.
(4) Draw the sample into syringe and then weigh the syringe.
(5) Inject sample from rubber septum of electrolytic cell as shown in Fig.3.2.
(6) Start titration. Measurement parameter is shown in Table 4.1.
(7) Weigh the syringe again and then set the difference of weight to sample size.

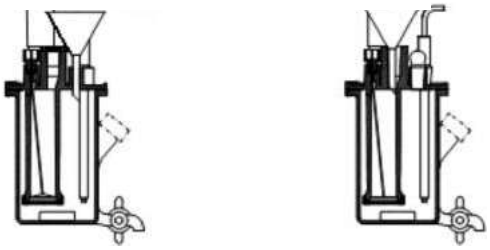
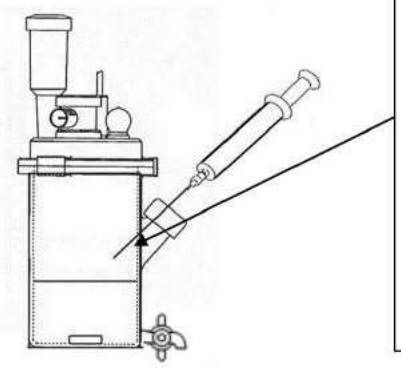


Fig.3.1. Preparation of the reagents.



Note

(1) Do not touch the tip of needle to anode solution.

(2) When injecting, set the tip of needle as below figure.

(3) Before pulling out the needle, draw a little air to prevent dripping of sample.

Fig.3.2. Injection of sample.

4. Parameters and results

Table 4.1. Parameter.

Condition File		
Cal Mode	0:Sample weight(net)	
	X=(H_2O-BLANK)/SIZE	
Interval Time	20	sec
Current	SLOW	
S-Timer	0	min
Blank Value	0	ug
Unit Mode	AUTO	
Auto Interval	0	g
Minimum Count	5	ug
Back Ground	ON	
Sample Size Input	Every Time	
Cell Type	Standard/Fritless	

Table 4.2. Results of water content measurement in hydrocarbons.

						ter Content (ppm)	Statistics Results	
						34.2	Avg.	32.8 ppm
			CG	1.0617	34.4	32.4	SD	1.2 ppm
				1.0936	34.8	31.8	RSD	3.8 %
		Fritless	AG	1.1425	36.8	32.2	Avg.	31.5 ppm
				1.0688	33.1	31.0	SD	0.6 ppm
				1.0431	32.6	31.3	RSD	2.0 %
Chloroform	AQ	Standard	AG	2.9461	88.1	29.9	Avg.	30.7 ppm
			CG	2.9798	93.0	31.2	SD	0.7 ppm
				2.9071	89.9	30.9	RSD	2.2 %
		Fritless	AG	2.7729	84.6	30.5	Avg.	30.2 ppm
				2.9253	85.8	29.3	SD	0.8 ppm
				2.9486	90.7	30.8	RSD	2.6 %
Cyclohexane	AQ	Standard	AG	1.5958	61.9	38.8	Avg.	38.0 ppm
			CG	1.5791	59.0	37.4	SD	0.7 ppm
				1.5795	59.9	37.9	RSD	1.9 %
		Fritless	AG	1.6039	60.2	37.5	Avg.	37.4 ppm
				1.5384	57.7	37.5	SD	0.2 ppm
				1.6157	60.1	37.2	RSD	0.5 %
Isooctane	AQ	Standard	AG	1.32027	63.7	48.2	Avg.	48.6 ppm
			CG	1.47262	71.8	48.8	SD	0.3 ppm
				1.43564	69.9	48.7	RSD	0.7 %
		Fritless	AG	1.43916	68.0	47.2	Avg.	47.3 ppm
				1.42384	66.2	46.5	SD	0.9 ppm
				1.44880	69.9	48.2	RSD	1.8 %

5. Note

(1) Use dried syringe and syringe vial at sampling for prevention of contamination by atmospheric water.

(2) To measure 100 µg or less water detection, make sure stability of blanking. Low and stable background value is important factor for trace level of water measurement.

omat AG-H and Oil are suitable for long the measurement accuracy of fritless cell. It can be used with the evaporator as well. Suitable reagent for fritless cell is required. For example, Hydranal coulomat AG and AG-Oven are compatible with fritless cell.

Keywords: Karl Fischer, Coulometric titration, Direct injection, Hydrocarbon, Fritless cell

Address:1739 Motoyoshida,Mito,Ibaraki,310-0836,JAPAN