Determination of Water Content in Aldehydes and Ketons

This application method is applicable for the determination of water in Aldehydes and Ketons. Aldehydes and Ketons reacts with the Karl Fischer reagent and subsequently generates water. To avoid this reaction, the reagent of special formulation is available.

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 5K or equivalent
	Titration medium	HYDRANAL^{R} KetoSolver or equivalent
Reagents for Coulometric Titrator	Anode solution	HYDRANAL^{R} Coulomat AK or equivalent
	Cathode solution	HYDRANAL^{R} Coulomat CG-K 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 background level may increase after several injection of the sample. In this case, exchange the anode solution with the fresh one.

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.	KF2	Jun.6. 2017

Water contents

Alcohols

1.Abstract

Water content of Alcohols is 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.

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

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

Alcohols do not interfere the Karl Fischer reaction and direct injection method could apply. Anode solution is selected in accordance with sample solubility. General-use anode solution contains methanol as solvent. Alcohols with long carbon chain have low solubility in methanol. In that case, use of anode solution for oil is appropriate.
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 (for general use, nonhalogenated)
Cathode solution	:	Hydranal coulomat CG

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.

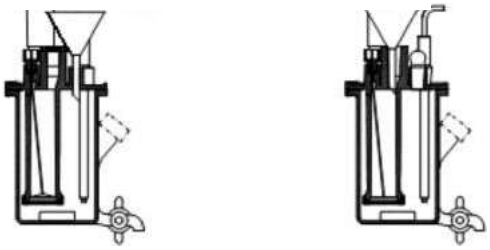
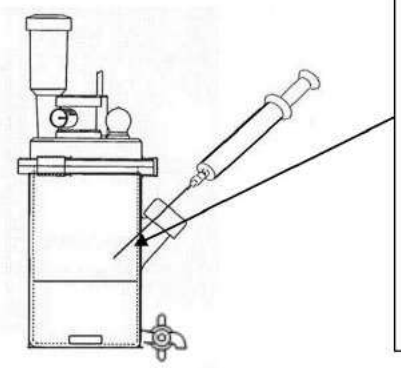


Fig.3.1. Preparation of the reagents.



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. Parameters

Condition File		
Cal Mode	0:Sample weight(net) X-(H2O-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 alcohols

				ple (g)	Water (µg)	Water Content
				88	672.9	1124 ppm
			CG	0.8890	991.6	1115
				0.8710	973.6	1118
		Fritless	AG	0.9415	1047.4	1112 ppm
				0.8964	996.7	1112
				0.8739	972.3	1113
2-propanol	AQ	Standard	AG	1.5078	138.9	92.1 ppm
			CG	1.6015	145.7	91.0
				1.6574	152.6	92.1
		Fritless	AG	1.5623	142.7	91.3 ppm
				1.5496	139.4	90.0
				1.5832	142.9	90.3
2-methoxyethanol	AQ	Standard	AG	0.86583	156.4	180.6 ppm
			CG	1.36865	243.5	177.9
				0.83448	150.1	179.9
		Fritless	AG	1.0574	188.2	178.0 ppm
				0.95761	172.1	179.7
				1.02781	185.3	180.3
n-hexylalcohol	AQ	Standard	AG	0.87698	132.1	150.6 ppm
			CG	0.92031	138.0	149.9
				0.81105	124.0	152.9
		Fritless	AG	0.80510	122.9	152.7 ppm
				0.85949	131.1	152.5
				0.97448	148.7	152.6

5. Note

(1) Use dried syringe and syringe vial in Fig.5.1, for preventive of contamination by atmospheric water.

(2) Put appropriate anode solution in use according to the solubility of the sample. For example, Hydranal Coulomat AG-H and Oil are suitable for oils.

Fig.5.1. Draw the sample from syringe vial.

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