

HIRANUMA APPLICATION DATA	Karl Fischer Titrator	Data No.	KF5	Jun.6. 2017
Water contents	Ethers and Esters			

1. Abstract

Water content of Ethers and Esters are 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.



Ethers and esters do not interfere the Karl Fischer reaction and direct injection method could apply. Anode solution is selected from General-use or Oil in accordance with sample solubility. General-use anode solution contains methanol as solvent. When the sample has low solubility in methanol, the use of anode solution for oil is appropriate. One exception is a sample containing vinyl group, which reacts with KF reagent and interferes the titration. When fritless cell is used, cathode solution is not necessary.

2. Apparatus and Reagents

(1) Apparatus

Titration	:	Karl Fischer Coulometric titrator	AQ-2200A
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.

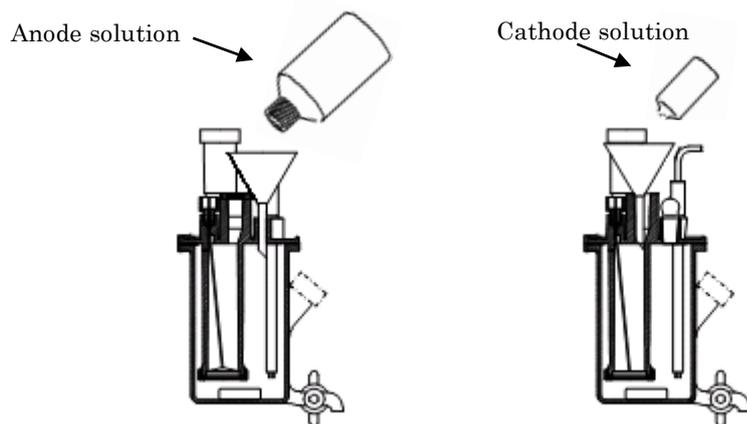


Fig.3.1. Preparation of the reagents.

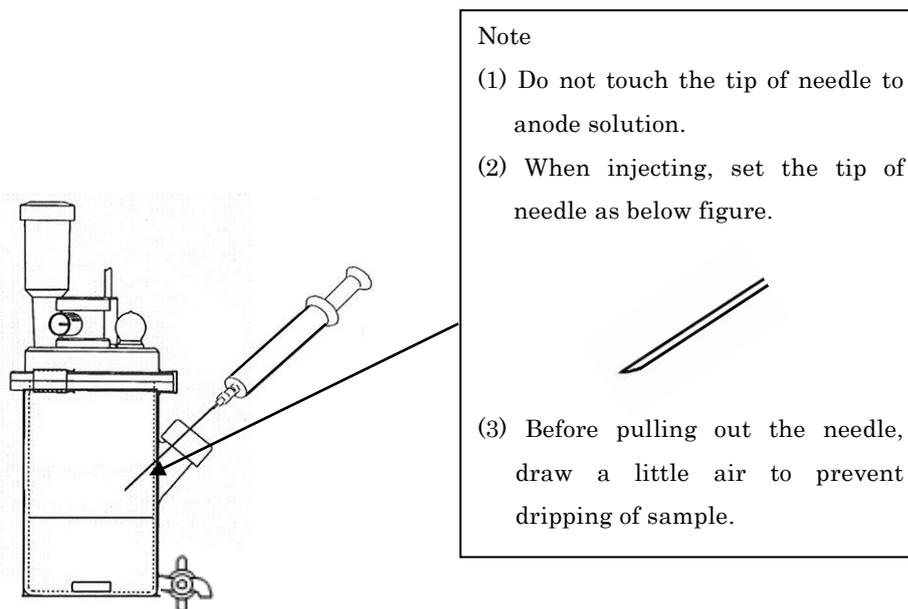


Fig.3.2. Injection of sample.

4. Parameters and results

Table 4.1. Parameters

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 ethers and esters

Sample	Apparatus	Cell	Reagent	Sample Size (g)	water (µg)	Water content	
Anisole	AQ	Standard	AG	2.0827	341.4	163.9 ppm	
			CG	1.9764	323.9	163.9	
				2.0089	329.1	163.8	
		Fritless	AG		2.0984	341.8	162.9 ppm
					1.9812	323.3	163.2
					1.9649	321.9	163.8
Ethyl acetate	AQ	Standard	AG	0.8797	395.0	449.0 ppm	
			CG	1.0007	449.8	449.5	
				1.0423	470.4	451.3	
		Fritless	AG		0.9985	451.7	452.4 ppm
					1.1310	512.8	453.4
					1.0082	456.6	452.9
Diethyl ether	AQ	Standard	AG	0.7746	567.1	732.1 ppm	
			CG	0.7844	573.4	731.0	
				0.8011	585.9	731.4	
		Fritless	AG		0.8379	608.9	726.7 ppm
					0.9185	672.9	732.6
					0.8504	626.0	736.1
1,4-dioxane	AQ	Standard	AG	0.3480	330.8	950.6 ppm	
			CG	0.5230	493.2	943.0	
				0.3995	381.5	954.9	
		Fritless	AG		0.4492	425.1	946.3 ppm
					0.5779	547.7	947.7
					0.5552	525.0	945.6
4-butyrolactone	AQ	Standard	AG	0.9274	61.2	66.0 ppm	
			CG	1.19304	70.8	59.3	
				1.22069	72.3	59.2	
		Fritless	AG		1.13511	79.2	69.8 ppm
					1.10311	70.2	63.6
					0.81464	51.3	63.0
Propylene carbonate	AQ	Standard	AG	1.27881	230.2	180.0 ppm	
			CG	0.90269	159.9	177.1	
				1.17183	204.3	174.3	
		Fritless	AG		1.29844	229.5	176.8 ppm
					1.27775	224.7	175.9
					1.18092	210.5	178.3

5. Note

- (1) Use dried syringe and syringe vial 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.

Note : these reagents does not correspond to Fritless cell.