HIRANUMA APPLICATION DATA	Karl Fischer Titrator	Data No.	KF3	Jun.6. 2017
Water contents	**Aromatic Hydrocarbon**			

1. Abstract

Water content of Aromatic hydrocarbons 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.

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

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

Aromatic hydrocarbons 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. When fritless cell is used, cathode solution is not necessary.

2. Apparatus and Reagents

(1) Apparatus

Titrator	:	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.

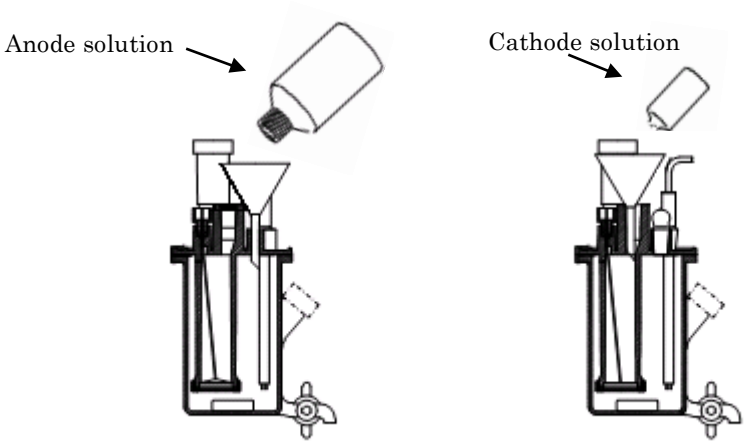
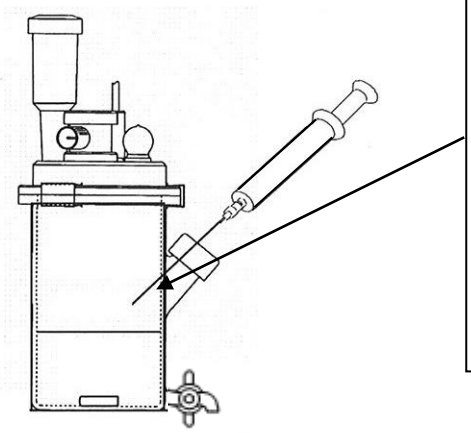


Fig.3.1. Preparation of the reagents.



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) $X=(H_2O\text{-}BLANK)/SIZE$	
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 aromatic hydrocarbons

Sample	Apparatus	Cell	Reagent	Sample Size (g)	water (μg)	Water content
Toluene	AQ	Standard	AG	1.7520	94.3	53.8 ppm
			CG	1.7483	93.3	53.4
				1.7348	92.7	53.4
		Fritless	AG	1.7549	91.4	52.1 ppm
				1.7398	92.1	52.9
				1.7356	91.9	53.0
Xylene	AQ	Standard	AG	1.77164	110.5	62.4 ppm
			CG	1.62378	94.3	58.1
				1.65876	99.9	60.2
		Fritless	AG	1.62232	93.2	57.4 ppm
				1.51125	88.0	58.2
				1.77412	104.5	58.9

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.

Note : these reagents does not correspond to Fritless cell.

Fig.5.1. Draw the sample from syringe vial.