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
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.

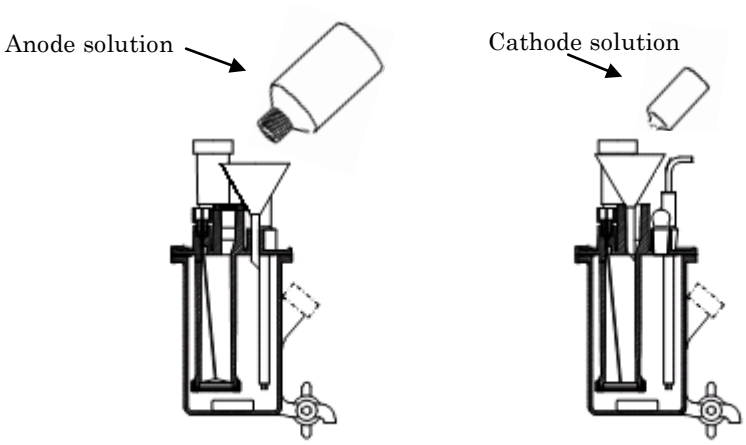
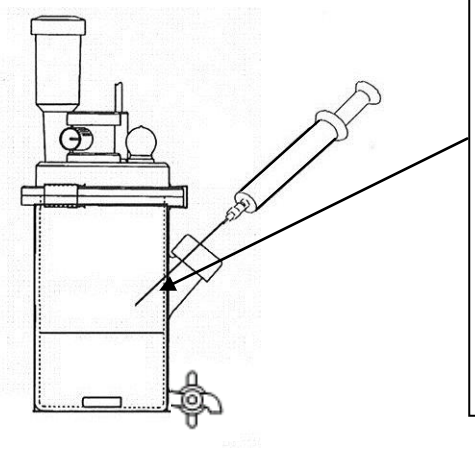


Fig.3.1. Preparation of the reagents.



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.

Sample	Apparatus	Cell	Reagent	Sample Size (g)	Water (µg)	Water Content (ppm)	Statistics Results		
Heptane	AQ	Standard	AG	1.0418	35.6	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.

(3) Select anode solution according to the solubility of the sample. For example, Hydranal Coulomat AG-H and Oil are suitable for long chain hydrocarbons and oils.

(4) The optimized electrolysis control for fritless cell of AQ series released after 2009 improves 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