HIRANUMA APPLICATION DATA	Karl Fischer Titrator	Data No.	KF8	Jun.6. 2017

Water contents

Oil products – Fuel oil

1.Abstract

Water content of oil products 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.

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

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

Generally the fuel oil does not interfere the Karl Fischer reaction and direct injection method could apply. Suitable anode solution is selected for dissolving oil samples.

It is known that some of the oil additives interfere Karl Fischer reaction. In that case, azeotropic distillation method with Oil evaporator is appropriate. Water is separated from oil sample by distillation and introduced to electrolytic cell with carrier gas.

2. Apparatus and Reagents

(1)Apparatus

Titrator	:	HIRANUMA Karl Fischer Coulometric titrator AQ-series or MOICO-A19
Evaporator	:	Oil Evaporator EV-2000L
Electrolytic cell	:	Standard Cell

(2)Reagents for direct injection method

Anode solution	:	Hydranal coulomat AG-H / Oil
Cathode solution	:	Hydranal coulomat CG

(3)Reagents for azeotropic distillation method

Anode solution	:	Hydranal coulomat AG (for general use, nonhalogenated)
Cathode solution	:	Hydranal coulomat CG
Distillation solvent	:	Dehydrated toluene
Carrier gas	:	Nitrogen gas

3. Procedure

3.1. Direct injection method

(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.

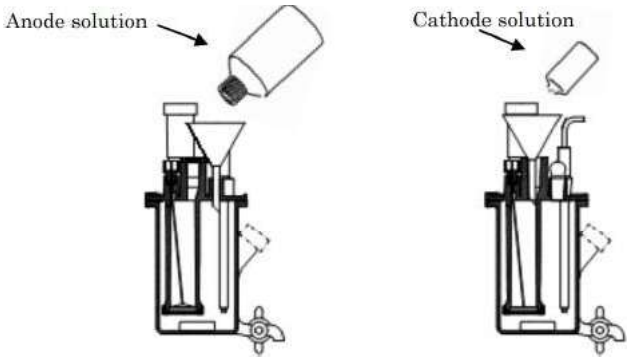


Fig.3.1. Preparation of the reagents.

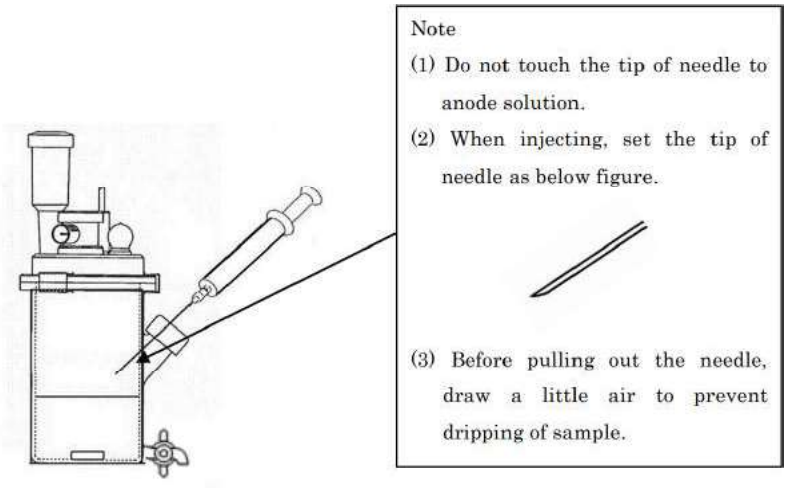


Fig.3.2. Injection of sample.

3.2. Azeotropic distillation method

(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) Connect electrolytic cell and evaporation chamber with tube. Flow carrier gas with 50 mL/min
(4) Fill 5 mL of distillation solvent in evaporation chamber and heat the chamber at 120 °C.
(5) Keep blanking to attain stable background with carrier gas flowing into electrolytic cell.
(6) Wash the syringe with sample.
(7) Draw the sample into syringe and then weigh the syringe.
(8) Inject sample from rubber septum of distillation chamber as shown in Fig.3.3 and Fig.3.4.
(9) Start titration. Measurement parameter is shown in Table 4.2.
(10) Weigh the syringe again and then set the difference of weight to sample size.

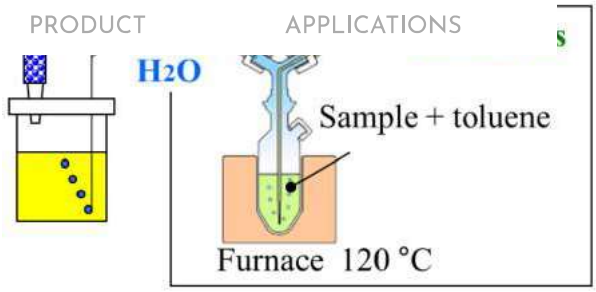
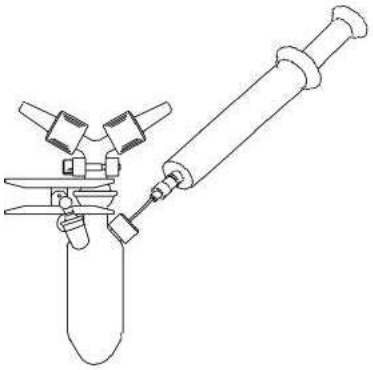


Fig.3.3. Schematic diagram of azeotropic distillation method.



Note

(1) Do not touch the tip of needle to distillation solvent level.

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

(3) If air is drawn at inside of evaporation chamber, distillation solvent would be involved. When drawing air, do it after pulling out the needle from evaporation chamber to prevent weighing error.

Fig.3.4. Sample injection into evaporation chamber.

4. Parameters and results

Table 4.1 Parameters for direct injection method

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

Table 4.2 Parameters for azeotropic distillation method

Interval Time	40	sec
Current	SLOW	
Min.Timer	5	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	

Table 4.3 Results of water content measurement in Oil products

Sample	Apparatus	Cell	Reagent	Sample Size (g)	Water (µg)	Water Content
Gasoline	AQ+EV-L	Standard	AG	2.1268	82.8	39.0 ppm
			CG	2.2044	83.0	37.7
				2.1970	77.6	35.3
Heavy oil A	AQ+EV-L	Standard	AG	2.0054	132.3	66.0 ppm
			CG	1.9829	122.3	61.7
				1.9999	130.4	65.2
Heavy oil C	AQ+EV-L	Standard	AG	0.9529	15482.0	1.6248 %
			CG	0.2509	3911.3	1.5589
				0.4938	7982.0	1.6164
Aviation fuel	AQ	Standard	AG-H	9.5000	398.8	42.0 ppm
			CG	9.5402	388.0	40.7
				9.6370	398.1	41.3

5. Note

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

background value is important factor for

, Hydranal Coulomat AG-H and Oil are

suitable for oils with direct injection method.

Note : these reagents does not correspond to Fritless cell.

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