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HIRANUMA APPLICATION DATA	Karl Fischer Titrator	Data No.	KF7	Nov.28. 2018
Water contents	**Nitriles**			

1.Abstract

Water content of Nitriles 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.

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

$$2RN \cdot HI \rightarrow I_2 + 2RN + 2H^+ + 2e^-$$
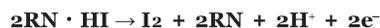
Nitriles do not interfere the Karl Fischer reaction and direct injection method could apply. Anode solution is selected in accordance with sample solubility. General use of anode solution contain methanol as solvent. When the sample like a long chain hydrocarbon has poor solubility in methanol, anode solution containing chloroform or hexanol or toluene is used.

2. Apparatus and Reagents

(1) Apparatus

    Titrator   : HIRANUMA Karl Fischer Coulometric titrator AQ-series or MOICO-A19

    Electrolytic cell   : Standard Cell

        Fritless Cell (No cathode solution required)

(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. When fritless cell is used, cathode solution is not necessary.

(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.

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Fig.3.1. Preparation of the reagents. (Standard Cell)
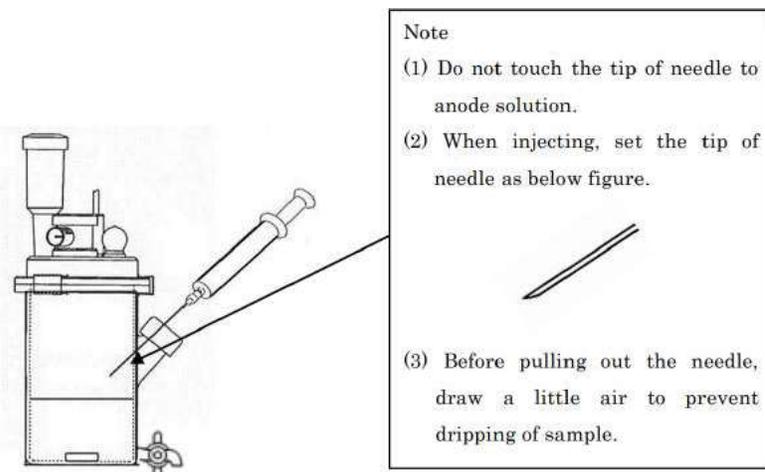
Fig.3.2. Injection of sample.

4. Parameters and results

Table 4.1. Parameter.

| 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 μg
Unit Mode	AUTO
Auto Interval	0 g
Minimum Count	5 μg
Back Ground	ON
Sample Size Input	Every Time
Cell Type	Standard/Fritless

Table 4.2. Results of water content measurement in nitriles.

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Compound	Product			(content, n)	Statistics Results	
				6	Avg.	99.3 ppm
		1.5675	158.5	101.1	SD	2.0 ppm
		1.5602	151.5	97.1	RSD	2.0 %
	Fritless	1.5305	153.7	100.4	Avg.	99.8 ppm
		1.4616	147.1	100.6	SD	1.2 ppm
		1.5896	156.4	98.4	RSD	1.2 %
3-Ethoxypropionitrile	Standard	0.78380	255.4	325.8	Avg.	325.9 ppm
		0.77352	253.0	327.1	SD	1.2 ppm
		0.97904	318.0	324.8	RSD	0.4 %
	Fritless	0.95859	312.6	326.1	Avg.	325.3 ppm
		0.87339	283.5	324.6	SD	0.8 ppm
		0.95782	311.6	325.3	RSD	0.2 %

5. Note

(1) Use dried syringe and syringe vial in Fig.5.1 for preventive 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 Hydranal coulomat Oil are suitable for long chain hydrocarbons and oil samples.

(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.
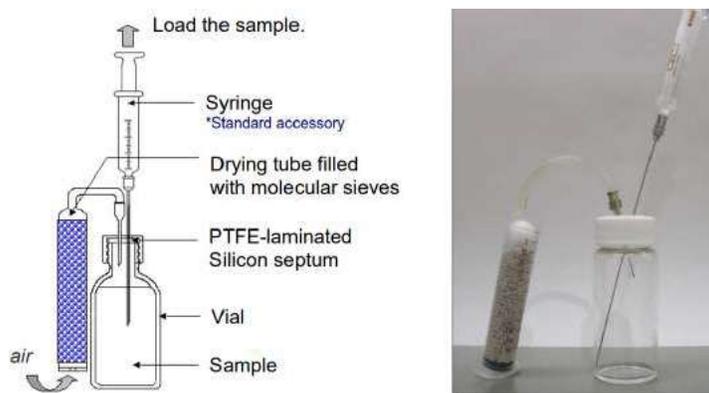
FFig.5.1. Draw the sample from syringe vial.

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pages.

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