

Determination of Water Content in Benzoic Acid

This application method is applicable for the determination of water in Benzoic acid. However, this method can be used for many kinds of organic compound if they are sufficiently soluble in the reagent.

Titration	AQV-2000/AQV-200 (Volumetric titrator) AQ-2000/AQ-200 (Coulometric titrator)	
KF Oven	N/A	
Reagents for Volumetric Titrator	Titration medium	HYDRANAL [®] Composite 5 or equivalent
	Titration medium	Dehydrated methanol
Reagents for Coulometric Titrator	Anode solution	HYDRANAL [®] Coulomat AG or equivalent
	Cathode solution	HYDRANAL [®] Coulomat CG or equivalent

PARAMETER SETTINGS for the Titrator

Volumetric Titrator		Coulometric Titrator	
End Mode	4	CAL Mode	0
CAL Mode	0	Interval	20 sec
Interval	20 sec	Current	Fast
MIN Feed	0.01 ml	S-timer	0 min
S-timer	0 min	T-timer	0 min
T-timer	0 min		

PARAMETER SETTINGS for Oven

Temperature	-
Carrier flow rate	-

PROCEDURE:

1. Collect the sample in a tightly closed container.
2. Titrant the titration medium to zero. (Blanking)
3. Press SAMPLE key.
4. Using a spoon, put approx. 2 g of the sample into titration cell by opening the glass stopper.
5. Press TITRATION key.
6. Press S.SIZE key and enter the sample size.

OPERATING NOTES:

1. The sample should be put in as quickly as possible to prevent the atmospheric moisture to enter into the titration cell.
2. Injected sample size is calculated by subtracting AFTER injection container weight from BEFORE injection container weight.
3. To get the most accurate result, the titrant should be consumed around 5 ml. To make titrant consumption into this range, calculate the optimum sample size by this equation ;

$$\text{SIZE (g)} = 5 \times F / (C \times 10)$$

Where ;

F : Factor of titrant. ex. Factor of Composite 5 is 5 (mgH₂O/ml).

C : Sample moisture in %

For example, sample moisture is 1%, and Factor is 5, then optimum sample size is,

$$5 \times 5 / (1 \times 10) = 2.5\text{g}$$

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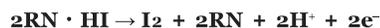
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HIRANUMA APPLICATION DATA		Karl Fischer Titrator	Data No.	KF3	Jun.6. 2017
Water contents	Aromatic Hydrocarbon				

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.



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

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Fig.3.1. Preparation of the reagents.

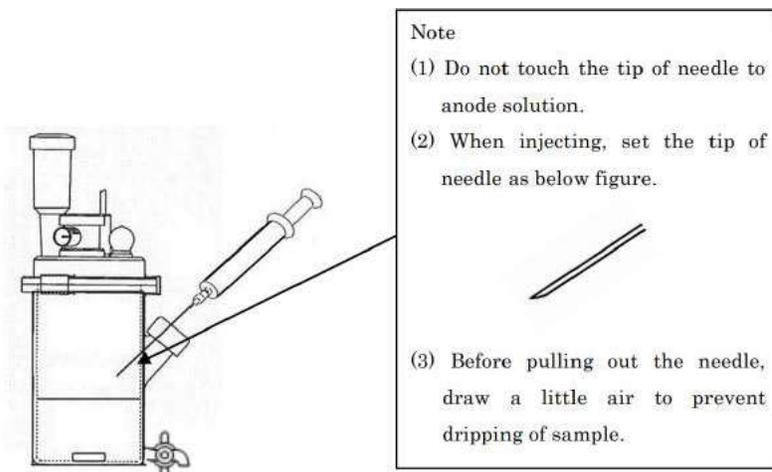


Fig.3.2. Injection of sample.

4. Parameters and results

Table 4.1. Parameters

Condition File	
Cal Mode	o:Sample weight(net) X-(H ₂ O-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

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		pages.			Water (µg)	Water Content
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		CG	1.7483	94.3	53.8 ppm	
			1.7348	93.3	53.4	
				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.



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