HIRANUMA APPLICATION DATA	Karl Fischer Titrator	Data No.	KF14	Nov.29. 2018
Water contents	**Ketones and Aldehydes – KF Volumetry, Direct-Injection Methyl ethyl ketone, Acetone, and Cyclohexanone**			

1.Abstract

Water contents of ketones and aldehydes could be determined by Karl Fischer volumetric titrator. In volumetric titration, titrant have a factor which is the capacity to react with water per 1 mL of titrant. Factor is pre-determined before sample measurement and water content of sample is calculated from consumed titrant volume by sample measurement.

When the sample is liquid, generally sample is measured by direct injection into the titration cell. Dehydrated methanol is generally used for titration solvent. However, in the measurement of ketones and aldehydes, since these react with methanol to produce water, the measurement result tends to be higher than the true value (formula (1)).

$$R_2CO + 2CH_3OH \rightarrow R_2C(OCH_3)_2 + H_2O \quad \cdot\cdot\cdot (1)$$

For above reason, Karl Fischer reagent without methanol should be used for water determination of ketones and aldehydes. There are commercially available reagents with a special composition for ketones and aldehydes. This chapter introduces an example for the water determination in methyl ethyl ketone, acetone and cyclohexanone with water added to them to 1 %. These samples are often used as paint solvents, raw materials of adhesives, and synthetic resins.

2. Apparatus and Reagents

(1) Apparatus

Titrator	:	HIRANUMA Karl Fischer Volumetric titrator AQV-series or MOIVO-A19
Titration cell	:	Standard Cell

(2)Reagents

Titrant	:	HYDRANAL Composite 5K
Titration solvent	:	HYDRANAL Working Medium K

3. Procedure

(1) Fill 50 mL of titration solvent into the titration 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 titration 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.

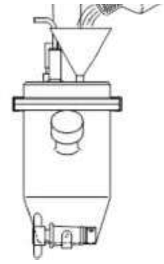

Fig.3.1 Preparation of the reagents

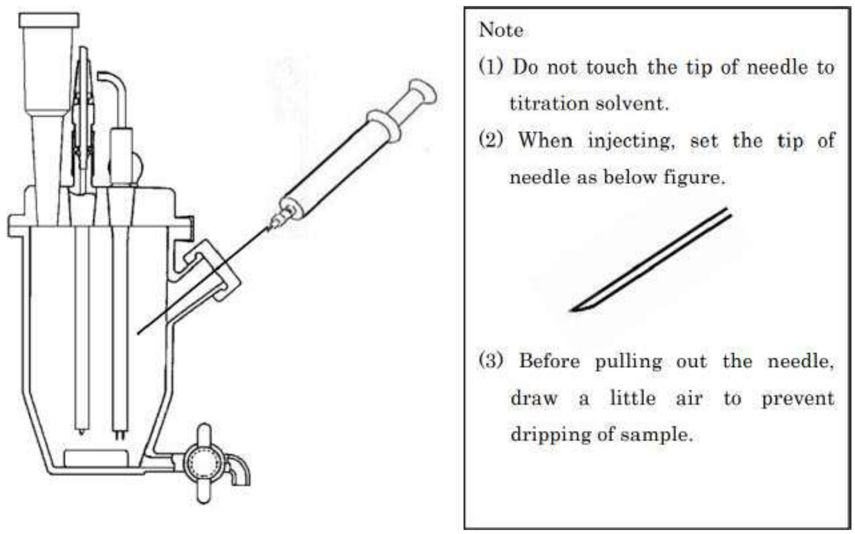

Fig.3.2 Injection of sample

4. Parameters and results

Table 4.1. Parameter.

Condition File		
Cal Mode	0:Sample weight(net)	
	X=(H-b)xFx1000/SIZE	
Interval Time	30	sec
Max Volume	20	mL
Min Feed Vol.	0.01	mL
S.Timer	0	min
KF Factor	5.4839	mg/mL
KF Buret No.	1	
KF Speed(OUT)	12	mL/min
KF Speed(IN)	24	mL/min
Back Ground	OFF	
Sample Size Input	Every Time	
Blank Value	0	mL

C.P Level	150 μA
E.P Level	200 μA
Auto Interval	0 g

Table 4.2 Results of water content measurement in ketones

Sample	Sample size (g)	Titer (mL)	Water (mg)	Water content (%)	Statistics result	
Methyl ethyl ketone	0.5410	1.04	5.759	1.0645	Avg.	1.062 %
	0.4792	0.92	5.095	1.0632	SD	0.003 %
	0.5652	1.08	5.981	1.0582	RSD	0.31 %
Acetone	0.4925	0.91	5.039	1.0231	Avg.	1.025 %
	0.5003	0.93	5.150	1.0294	SD	0.004 %
	0.4171	0.77	4.264	1.0223	RSD	0.38 %
Cyclohexanone	0.4023	0.74	4.098	1.0186	Avg.	1.021 %
	0.3848	0.71	3.932	1.0218	SD	0.003 %
	0.3841	0.71	3.932	1.0237	RSD	0.25 %

5. Note

(1) When the side reaction cannot be suppressed even though you use reagent for ketones, phenomena such as unstable blanking or undetectable endpoint can be obtained. In that case, it may be improved by reducing the amount of sample or replacing titration solvent with new one.

(2) Sampling tools should be dried up well before use.

(3) Purge and fill the titrant to fill it homogeneously into the buret.

Address:1739 Motoyoshida,Mito,Ibaraki,310-0836,JAPAN