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HIRANUMA APPLICATION DATA		Karl Fischer Titrator	Data No.	KF17	Apr. 19, 2018
Water contents	Sugars – KF Volumetry, Powder Sample Glucose, fructose, and sucrose				

1. Abstract

Water contents of sugars could be determined by Karl Fischer volumetric titrator. In volumetric titration, titrant have a factor which means 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.

This application introduces an example for the water determination in glucose, fructose, and sucrose. Sugars have tendency to dissolve in formamide. Therefore a mixed solvent of formamide and methanol was chosen as the titration solvent.

2. Apparatus and Reagents

(1) Apparatus

Titrator	: HIRANUMA Karl Fischer Volumetric titrator AQV-series or MOIVO-A19
Titration cell	: Standard Cell, without drain valve P/N D327511-1
Powder funnel	: less than 13 mm outer diameter of leg

(2) Reagents

Titrant	: HYDRANAL Composite 1 (Honeywell)
Methanol	: HYDRANAL Methanol (Honeywell)
Formamide	: HYDRANAL Formamide dry (Honeywell)
Titration solvent	: Mixed solvent of methanol and formamide at a volume ratio of 1: 2

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) Put a sample container, powder funnel and spoon on the balance. Record its read (S_1 [g]).
- (4) Open the glass stopper of titration cell lid to introduce the sample with powder funnel as shown in Fig.3.2.
- (5) Start titration. Measurement parameter is shown in Table 4.1.
- (6) Weigh the sample container, powder funnel and spoon again and record its read (S_2 [g]). The difference of ($S_1 - S_2$ [g]) is set as sample size.

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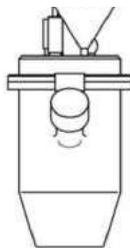


Fig.3.1 Preparation of the reagents

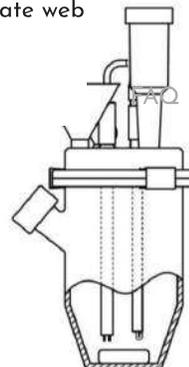


Fig.3.2 Introduction of sample with powder funnel

4. Parameters and results

Table 4.1 Parameters

Condition File	
Cal Mode	o:Sample weight(net) $X=(H-b) \times F \times 1000 / \text{SIZE}$
Interval Time	30 sec
Max Volume	20 mL
Min Feed Vol.	0.01 mL
S.Timer	0 min
KF Factor	1.0868 mg/mL
KF Buret No.	1
KF Speed(OUT)	12 mL/min
KF Speed(IN)	12 mL/min
Back Ground	OFF
Sample Size Input	Every Time
Blank Value	0 mL
Unit Mode	AUTO
E.P Detection	uA
Solvent	FM
C.P Level	150 μ A
E.P Level	200 μ A
Auto Interval	0 g

Table 4.2 Results of water content measurement in sugars

Sample	Sample size (g)	Titer volume(mL)	Water (mg)	Water content (ppm)	Statistics result	
Glucose	0.7383	1.41	1.532	2075.0	Avg.	2076.3 ppm
	0.6805	1.27	1.380	2028.0	SD	49.0 ppm

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Fructose	0.6485	0.35	0.380	586.0	Avg.	585.1 ppm	
	0.7050	0.38	0.413	585.8	SD	1.4 ppm	
	0.5965	0.32	0.348	583.4	RSD	0.2 %	
Sucrose	0.4116	0.29	0.315	765.3	Avg.	762.5 ppm	
	0.6171	0.43	0.467	756.8	SD	4.9 ppm	
	0.7238	0.51	0.554	765.4	RSD	0.6 %	

5. Note

- (1) The blanking might become unstable when time has passed since the solvents of methanol and formamide were mixed. The cause could be due to the ammonia generated by mixing solvents. Adding 3 g of benzoic acid to 50 mL of the titration solvent improves the unstable state of blanking.
- (2) AQP series released after 2009 have the current detection method which is added as the endpoint detection method in addition to the conventional potential detection method. The current detection method doesn't need the adjustment of the end point parameter "EP level" for each titration solvent type. In this application, it is set to the current detection method. When performing with the potential detection method, set "E.P. Detection" to "mV", and set "E.P. level" to "3" for proper parameter of the titrant solvent (methanol/formamide).

Keywords: Karl Fischer, Volumetric titration, Sugar



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