AQUA COUNTER

Weekly Application Note

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Category

Coulometric Karl Fischer Titrator AQ series

Pharmaceuticals
Cosmetics

Water in freeze-dried serum

by Indirect method (Evaporator + Coulometric KF titrator)

Referenced methods

Key words; freeze dried serum, lyophilized products, Karl Fischer titration

Outline

In this application, the indirect Karl Fischer method is introduced for the water determination in lyophilized products by using AQUACOUNTER® AQ-2100 (or AQ-300) coulometric KF titrator interfaced with Solid Evaporator EV-2000.

Reagents

Anode solution : Hydranal® Coulomat AG 100mL

Cathode solution : Hydranal® Coulomat CG

Additional solvent : Dehydrated methanol 50mL

Recommended configurations

KF Titrator: AQ-2100S (High-end, LCD screen)

• Evaporator. EV-2000 Solid evaporator

*Optional Thermal Printer PR-2000T2 / Dot impact printer PR-302B



▲ AQ-2100S + Printer + EV-2000



Procedure

- 1. Place an aluminum foil cup into the evaporation chamber and set it on the evaporation base.
- 2. Press F1[EV Start] key on the AQ-2100 to start pre-heating.
- 3. Place a sample container, a funnel, and a sampling spoon on the balance, and tare it.
- 4. Press SAMPLE key once the instrument is ready for measurement.
- 5. Open a glass stopper and introduce about 0.05g of sample through the funnel once the background stabilized.
- 6. Close the stopper and press TITRATION key to start titration.
- 7. Weigh the total weight of the sample vial and other sampling tools again.

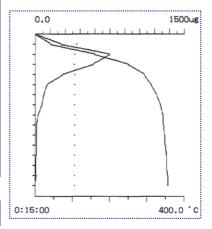
 The difference between the total weight before/after sampling shall be the sample size.
- 6. Press S.SIZE/No. key to input the amount of sample added.

Condition parameters and an example result

Condition file		
Cal Mode	0	
Interval Time	20 sec	
S-Timer	0 min	
T-Timer	0 min	
Current	FAST	
Blank Value	0.0 μg	
Unit Mode	AUTO	
Back Ground	ON	
Auto Interval	0.00000 g	
Minimum Count	5 μg	

EV file				
Step1 Temp.	105 °C			
Time	15 min			
Pre Heat End Time	10 min			
B.G.	0 µg			
Cooling Time	5 min			
B.G. Count	10			
Back Purge Time	5 sec			

N ₂ Carrier gas flow rate	50mL/min
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Sample No.	Sample size (g)	Measured H ₂ O (μg)	Water content (%)	
1	0.0988	1339.5	1.3558	Mean. 1.39 %
2	0.0508	708.3	1.3943	SD 0.04 %
3	0.0603	863.4	1.4318	CV 2.7 %

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