

<b>KEM</b>		<b>KF Application Note</b>	<b>No. K VX-01531</b>
<b>By type of Industry</b>	<b>Water Determination in Plastics</b>		<b>Volumetric titration (Indirect method)</b>

## 1. Abstract

Many of plastics are hardly dissolved in KF solvents and they cannot be measured by the Karl Fischer direct method but by its indirect method using an evaporator. The evaporated water in the oven is transferred to the KF titration cell with the dehydrating solvent by a carrier gas, followed by being measured by KF volumetric titration in accordance with "JIS K 0113 ('92)—General rules for methods of potentiometric, amperometric, coulometric, and Karl-Fischer titrators."

The evaporated water in the oven is transferred to the KF titration cell with the dehydrating solvent by air or an inert carrier gas for the water determination in plastics by titration. You should use either the solvent of a 1:1 mixture of Methanol and Ethylene glycol or the Dehydrating solvent ME for the solvent.

And some of measured samples in this application note are as follows:

Nylon-6/Polycarbonate/Polyethylene/ABS resin etc.

## 2. References

- 1) JIS K 0113 ('92)—General rules for methods of potentiometric, amperometric, coulometric, and Karl-Fischer titrators
- 2) ISO 760: 1978, Determination of Water — Karl Fischer method (General method)
- 3) ASTM E203-96, Standard Test Method for Water Using Volumetric Karl Fischer Titration
- 4) "HYDRANAL®-Manual", published by RdH

## 3. Cautions on measurement

- 1) For evaporation of sample, use an evaporator exclusively designed for moisture titration.
- 2) Heating temperature for samples should be determined according to "10. Measurement examples" as the temperature differs from sample to sample.
- 3) Previously determine the factor of a KF reagent with the solvent used for the measurements.

## 4. Equipment

Volumetric Karl Fischer Moisture Titrator  
MKA-510N, MKS-510N, MKA-510, MKS-510, MKS-500  
Evaporator ADP-511S

## 5. Reagents

Titrant: Composite 2 (made by *RdH*)  
Solvent: Dehydrating solvent ME (made by *Hayashi*)

## 6. Analysis procedure

### Measurement:

- 1) Take 0.2 to 4g of the sample onto a sampler of the evaporator.
- 2) Weigh the above 1) by an electronic balance with the readability of 0.1mg, and attach the sampler at the designated position on heating tube of the evaporator.
- 3) Deliver 50mL of dehydrating solvent ME into the titration cell.
- 4) After checking the tube line between the titration cell and the evaporator as well as other gas-tight sections and air-vent opening, carry out the gas purge by a carrier gas cell throughout a heating tube, a sampler and a titration cell.
- 5) Carrier gas should keep flowing from the heating tube to the titration cell.
- 6) Raise oven temperature to a designated value, and maintain the evaporation temperature.
- 7) Carry out pre-titration for dehydrating the solvent in the cell.
- 8) Turn the sampler, and allow the sample to fall onto the sample boat in the heating tube.
- 9) Move the sample boat loaded with the sample to the hot zone in the heating tube in order to start the evaporation of water.
- 10) Press [Start] key of the KF titrator.
- 11) Weigh the sampler of 8).
- 12) Enter the weight obtained from the above 2) and 11) steps in 'Wt1' and 'Wt2' entries on the KF titrator, respectively.
- 13) Repeat the above steps, 1), 2) and 8) through 12), three to five times.
- 14) After completing a series of measurements, calculate the average of moisture content using the statistical feature of the titrator.

## 7. Titration parameter setting

MKA-510N MKS-510N	MKA-510 MKS-510	MKS-500
<b>Method</b> 1 <b>Titr Mode</b> Normal <b>Titr Buret No.</b> 1 <b>End Time</b> 0 s <b>Final Vol,</b> 0.01 mL <b>Titr Speed</b> : 3 <b>Detector Mode</b> 1 <b>t(stir)</b> 0 s <b>t(wait)</b> 10 s <b>t(max)</b> 1200 s <b>Drift Titr</b> : On <b>Start</b> : Manual <b>Max. Volume</b> : 10 mL <b>Dose mode</b> : Off <b>Oven</b> : ADP- <b>Oven Temp.</b> : See "10. Measurement examples" <b>Pre Treat</b> : 1 <b>Back Purge</b> : 180 s <b>Cell Purge</b> : 120 s <b>Sample Purge</b> : -	<b>Method</b> 1 <b>Titr Mode</b> 1 <b>Titr Buret No.</b> 1 <b>End Time</b> 0 s <b>Final Vol,</b> 0.01 mL <b>Titr Speed</b> : Slow <b>Detector Mode</b> 1 <b>t(stir)</b> 0 s <b>t(wait)</b> 10 s <b>t(max)</b> 1200 s <b>Drift Titr</b> : On <b>Start</b> : Manual <b>Max. Volume</b> : 10 mL <b>Dose mode</b> : Off <b>Oven</b> : ADP- <b>Oven Temp.</b> : See "10. Measurement examples" <b>Pre Treat</b> : 1 <b>Back Purge</b> : 180 s <b>Cell Purge</b> : 120 s <b>Sample Purge</b> : -	<b>Method</b> Direct <b>Titr Speed</b> : 3 <b>End Time</b> 0 s <b>Final Vol,</b> 0.01 mL <b>Detector Mode</b> Normal <b>t(stir)</b> 0 s <b>t(max)</b> 1200 s <b>Drift Titr</b> : On <b>Max. Volume</b> : 10 mL
<b>ADP-511S</b>	<b>ADP-511S</b>	<b>ADP-511S</b>
<b>Flow rate</b> : 200mL/min	<b>Flow rate</b> : 200mL/min	<b>Flow rate</b> : 200mL/min <b>Temp.</b> : See "10. Measurement examples"

## 8. Calculation parameter setting

MKA-510N/MKS-510N	MKA-510/MKS-510	MKS-500
<b>Calc.</b> 2	<b>Calc.</b> 2	<b>g→%</b>
<b>Unit</b> %	<b>Unit</b> %	
<b>Weight</b> Variable	<b>Weight</b> Variable	

## 9. Sample parameter setting

MKA-510N/MKS-510N	MKA-510/MKS-510	MKS-500
<b>ID</b> 0(arbitrary)	<b>Sample. No.</b> 01-01(arbitrary)	<b>Operator</b> (arbitrary)
<b>Operator</b> (arbitrary)	<b>Lot No.</b> (arbitrary)	<b>Sample. No.</b> 01-01(arbitrary)
<b>Sample. No.</b> 01-01(arbitrary)	<b>Blank</b> 0	<b>S.name</b> (arbitrary)
<b>Lot No.</b> (arbitrary)	<b>Wt1</b> Weight of meas. Item 2	<b>Sample. ID</b> (arbitrary)
<b>Blank</b> 0	<b>Wt2</b> Weight of meas. Item 11	<b>Blank</b> 0
<b>Wt1</b> Weight of meas. Item 2		<b>Weight1</b> Weight of meas. Item 2
<b>Wt2</b> Weight of meas. Item 11		<b>Weight2</b> Weight of meas. Item 11

## 10. Measurement examples

Sample	Sample size (g)	Evaporation temperature (°C)	Water content	
			mg	%
Nylon-6	0.214	150	4.47	2.09
Polycarbonate	2.498	150	3.23	0.13
Polyethylene	2.944	130	0.16	0.005
ABS resin	1.009	180	2.05	0.20

Commercial dehydrating solvent ME was used.

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