Application Note

№KVR-07031

Organic Chemical

Moisture of Soluble Cutting Oil

Volumetric Titration (Direct Method) by
Karl Fischer Moisture Titrator

Standard	JIS K 0113 JIS K 0068 ISO 760	ASTM E 203	

1.Abstract

Moisture titration using Karl Fischer reagent is popularly practiced water determination worldwide as the most reliable method. The procedure is adopted in many official standards as test method specified in ISO, ASTM, DIN, BS and JIS.

Here in this application we show an example of measurement of water content in cutting oil by volumetric moisture titration.

2.Reference

1) JIS K 0113-2005 : Standard Test Method by Potentiometric, Amperometrtic, Coulometric and Karl Fischer Titration
2) JIS K 0068-2001 : Test Method for Water Content in Chemical Products
3) ISO 760:1978 Determination of Water-Karl Fischer method (General method)
4) ASTM E 203-16 Standard Test Method for Water Using Volumetric Karl Fischer Titration
5) KEMAQUA technical information on water determination from Kyoto Electronics Manufacturing Co., Ltd.

3.Cautions in measurement

1) In order to refrain from the effect of ambient humidity, the test must be conducted in a well air-conditioned room.
2) The resolution of mass balance is desirable to the nearest 0.01mg.
3) Obtain the factor of Karl Fischer reagent using the solvent in advance.

4. Post-measurement care

After the reagent in flask is drained out and the electrode is cleaned, keep the titration flask filled with extracting medium.

5.Test equipment

Main unit : Karl Fischer moisture titration volumetric system
Electrode : Twin platinum electrode for KF titration

6.Reagent

Titrant : KEMAQUA TR-5
Solvent : KEMAQUA MET (for general use)
Additive : Salicylic acid

7.Test procedure

—Preparation—
1) Prepare approximately 30mL solvent in titration cell.
2) Add 5g salicylic acid to dissolve in solvent.
3) Dehydrate the titration cell by performing pre-titration.

—Measurement—
1) Prepare test sample in a syringe.
2) Weigh the syringe on balance.
3) Discharge the sample into titration flask.
4) Press Start key of titrator.
5) Weigh the mass of item 3) empty syringe.
6) Input Wt1 with weight of item 2) and Wt2 with 5).
7) Obtain water content from titration volume at the endpoint automatically detected.

8.Formula

Water content (%) = ((Data × TF – Drift × t – Blank) / (Wt1 – Wt2)) × k

Data	:	Titration volume (mL)
TF	:	Factor of titrant (mg H_2O /mL)
Drift	:	Drift level (mg/s)
t	:	Measuring time (s)
Blank	:	Blank level (0.00 mg)
Wt1	:	Sample + Syringe (g)
Wt2	:	Empty syringe (g)
k	:	Unit conversion coefficient (0.1)

9.Example of measurement

－Ambient condition－

Room temperature : 25 ℃	Humidity : 65 %	Weather : Fair

-Titration parameters-

Model : MKA-610
Method : 1

[Titration]

Titr.mode	:	Normal
t(stir)	:	2 s
t(wait)	:	0 s
t(max)	:	0 s
t(interval)	:	0 s
Max.Volume	:	20 mL
Titr.bur.No.	:	1
Dose mode	:	Off

[Control]

End time	:	30 s
Final Vol.	:	0.01 mL
Titr.speed	:	3
Detect.mode	:	1
Drift titr.	:	On
Start mode	:	Manual
End level	:	75 mV
Samp.time	:	5 s
Stir.speed	:	4

[Calculation]

Calc.type	:	Sample
Blank No.	:	1
Calc. No.	:	2
Unit	:	%
Decimal	:	2
Fraction	:	Half adjust
Drift comp.	:	Off
Evalution	:	Off

-Titration curve-

*** R e s u l t ***

Sample No. : 01-01
Date : 2007/08/09 15:05

Moisture	:	72.2318 mg
Result	:	92.13 %
Burette 1	:	15.395 mL
Titr.time	:	00:04:16
Wt1	:	5.2772 g
Wt2	:	5.1988 g
Net	:	0.0784 g

(Printout data from titration by MKA-610)

≪Titration parameter≫
Titr.mode: titration mode / t(stir): stir time before titration / t(wait): wait time before EP
t(max): max titration time allowed / t(interval) cutoff time / Max volume: maximum titration volume
Titr.bur.No.: burette number / Dose mode: fixed dose mode
≪Control parameter≫
End time: EP sense time / Final vol.: final dose volume / Titr.speed: of titration / Detect.mode: detection mode
Drift titr.: drift titration / End level: EP potential / Samp.time: sampling time / Stir.speed: of stirrer
≪Calculation parameter≫
Calc.type.: of titration / Blank No.: blank number / Calc.No. formula number / Unit: of results
Decimal: number of digits after decimal point / Fraction: rounding / Drift comp.: compensation / Evaluation: of results

－Measurement results－

N	Sample (g)	Titration (mL)	Water (%)
1	0.0784	15.395	92.13
2	0.0677	13.355	92.56
3	0.0595	11.645	91.83

Statistics	
Mean	92.17 %
SD	0.37 %
RSD	0.40 %

＊ The data were obtained by 3 tests of the same sample.

＊ Red underline shows the data from page 3/4.

10.Summary

Cutting oil has dual purpose, that is, to reduce friction of cutting in mechanical work and to cool the heat caused by the friction. Water soluble cutting oil is used mainly for cooling. With its lower environmental load, it is now the mainstream of cutting oil.

Sample measurement in the above example shows fair repeatability of 0.4% relative standard deviation. Stable measurement of moisture content is assured by Karl Fischer moisture titration.

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