

Petroleum

## Peroxide number of Kerosene

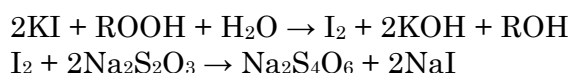
Redox titration by  
Automatic Potentiometric Titrator

Standard

JPI 5S-46-96

### 1. Abstract

The extent of oxidation of kerosene can be known by measuring peroxide number. A kerosene sample is tested as follows: the test sample dissolved in toluene added with potassium iodide is reduced, and then, free iodine is titrated with standard sodium thiosulfate where peroxide number is obtained in mg/kg (ppm).



### 2. Reference

- 1) Institutional standard: Test Method for Peroxide number of Kerosene JPI-5S-46-96

### 3. Cautions in measurement

- 1) Use toluene with minimum amount of sodium thiosulfate used in blank test.
- 2) Prepare potassium iodide each time after measurement since it is unstable.
- 3) When potassium iodide is prepared, use water without carbon dioxide contained.
- 4) Each time after measurement, clean the electrode with toluene either rinsing in wash bottle or using a dropper.

### 4. Post-measurement care

After a series of measurements is over for the day, clean the electrode with toluene, and then, ethanol. Finally rinse with water. Keep it in a beaker filled with water.

### 5. Test equipment

Main unit : Potentiometric titrator (standard preamplifier: STD- )  
Option : POV titration unit [№ 12-004-7700-48] optional  
Electrode : Trace type combination Pt. electrode C-778 [№ 98-100-C778] optional  
(junction liquid 3.3M-potassium chloride [№ 98-811-5001] )

## 6.Reagent

Solvent	:	Toluene Acetic acid (4mL hydrochloric acid + 996mL acetic acid)
Additive	:	Potassium iodide (60g potassium iodide dissolved in 50mL water)
Reagent	:	0.005mol/L sodium thiosulfate (f = 1.004 )
Inactive gas	:	Nitrogen gas

## 7.Measurement procedure

—Measurement—

- 1) Prepare a 200mL conical flask, and purge inside air with nitrogen for 3 minutes.
- 2) Pipette 50mL sample in the flask, and weigh it.
- 3) Add 25mL toluene, and blow N<sub>2</sub> gas into the liquid.
- 4) While N<sub>2</sub> gas is blowing, add 20mL acetic acid solution.
- 5) Slow down gas blow to one bubble per minute, add 2mL potassium iodide.
- 6) Shake up the conical flask exactly for 30 seconds.
- 7) Leave the flask for 5 minutes.
- 8) Add 100mL water into the flask.
- 9) Titrate with 0.005mol/L standard sodium thiosulfate to obtain peroxide number.

Note: Prior to sample measurement perform a blank test in advance in the same way as above to obtain blank level.

## 8.Formula

Peroxide number ( mg/kg ) = ( EP1 - BL1 ) \* TF \* K1 \* R / SIZE

EP1	:	Titration volume ( mL )
BL1	:	Blank level ( 3.8740mL )
TF	:	Factor of reagent ( 1.004 )
K1	:	Concentration of standard liquid ( 0.005 )
R	:	Constant ( 1000 * 8 )
SIZE	:	Sample size ( g )

## 9. Example of measurement

— Ambient condition —

Room temperature: 25.4°C	Humidity : 45%	Weather : Fair
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The below printout data were obtained from titration by AT-510:

— Titration parameter —

Model : AT-510 [Titration Parameter] Data : 2006/07/14 11:00 Method No. : 12 <Auto Intermit> Method Type : Titration	[Result Parameter] Data : 2006/07/14 11:02 Method No. : 12 <Auto Intermit> Method Name : POV-Sample
<Titration> Form : EP Stop APB No. : 1 Unit No. : 2 Detector No. : 1 Unit : mV Max. Volume : 20.00mL Wait Time : 0s Direction : Auto	<Calculation> Calc.Type : Sample Conc.1 : Set CO1=(EP1-BL1)*TF*K1*R/S IZE Unit : mg/kg Ep No. : 1 Temp.Comp. : Off
[Control Parameter] Data : 2006/07/14 11:01 Method No. : 12 <Auto Intermit> Method Name : POV-Sample Titr. Foem : EP Stop <Control> End Point No. : 1 End Sense : Auto End Point Area : Off Separation : Off Over Titr.Vol. : 0.0mL Gain : 10 Data Samp.Pot. : 4.0mV Data Samp.Vol. : 0.1mL Stability : 10mV/s Delay Time : 0s Limit Time : 0s	<Constant> K1 : 0.005 R : 8000  <Titr. Constant> Factor : 1.004 Conc. : 0.005  <Blank> Blank 1 : 3.8740

— Titration curve —

*** Result *** Sample No. : 18-01 Data : 2006/07/14 11:41 Sample ID : Method No. : 12 <Auto Intermit> Method Name : POV-Sample Titr.Reagent Name: <u>Na2S2O3</u>
Titr.Time : 00:06:04  Size : <u>39.302g</u>  Conc-1 : <u>0.1238mg/kg</u>
End Point-1 Volume : <u>3.9952mL</u> Potential : 232.7mV

Meaning of data on printout:

« Titration parameter »

Form: of titration / APB No. the number of power burette used/ Unit No.: **APB Unit File number**

Detector No. the number of detector used/ Unit: detection unit / Max Volume: of titration / Wait Time: before titration starts/Direction: of titration

« Control parameter »

End Point No.: number of endpoints / End sense: EP detection method/ End Point Area: of detection

Separation: of potential / Over Titr.Vol.: over titration volume / Gain: sensitivity of detection signal

Data samp.Pot. potential change of sampling signal / Data samp.Vol. titration volume for signal sampling

Stability: level / Delay Time: before stability check / Limit Time: for stability sensing

« Result parameter »

Calc.Type: of formula / Conc.1 formula 1 / EP1: **first endpoint**/ BL1: blank level/TF: factor of reagent

K1: unit conversion coeff. / R : constant/ **SIZE: of sample**/ Unit of results/EP No. the endpoint for calculation

Temp.Comp.: temperature compensation coeff. / Blank 1: **blank level 1**

TF: factor of reagent/ EP1: **titration volume for the first endpoint**/ BL1: blank level/ **SIZE: sample size**

Blank1: blank level 1

—Measurement results—

	Sample ( g )	Titrated ( mL )	Peroxide number ( mg/kg )
1	<u>39.302</u>	<u>3.9952</u>	<u>0.1238</u>
2	39.313	4.6474	0.7901
3	39.288	4.2972	0.4326

Statistics	
Mean	0.4488 mg/kg
SD	0.3334 mg/kg
RSD	74.292 %
Difference	0.6663 mg/kg

□ Precision specified by Japan Petroleum Institute □

Tolerance at room condition ( mg/kg ) =  $0.9 + 0.07 * A$   
 = 0.9314 ( mg/kg )

A : test average ( mg/kg )

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

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

## 10. Summary

The key in this measurement is how to minimize oxidation of air during reagent preparation and titration, and nitrogen ventilation is important each time.

Thus, the sample measurement succeeded within the JPI tolerance at room condition. (difference between max and min: 0.6663 mg/kg )

Such measurement is possible by the automated potentiometric titration, and the peroxide number of kerosene can be measured precisely by any of the following titration systems made by Kyoto Electronics (KEM).

### 【AT-610】



#### Awarded Product of Supreme Technology from Kyoto City

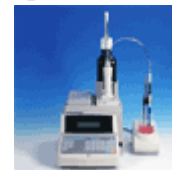
- Easy key entry by touch panel of large color LCD (8-inch wide)
- Simultaneous titration in parallel
- Both potentiometric and Karl Fischer moisture titration (coulometric • volumetric) can be performed at a time.

### 【AT-510】



- Compact and cost performance model
- PC card expands data memory for convenience and versatility.

### 【AT-500N-1】



- Low cost and high performance
- Easy view with back light LCD
- GLP/GMP conformed model

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