

Seet No.

GT200-PE026ER Oil

# Determination of base number in diesel engine oil ( Perchloric acid titration ) \_\_\_\_\_ 1/4

\*This application sheet is provided as reference, and does not assure the measurement results. Please consider analysis environment, external factors and sample nature for optimal conditions before the measurement.

## Outline

Base number in diesel engine oil by perchloric acid titration is determined with titration by perchloric acid in acetic acid titrant after dissolving sample into acetic acid and chlorobenzene. The titration result is used as reference of efficiency of basic component such as antioxidant, cleaning agent etc. in the oils.

Titration Type : Non-aqueous Neutralization, Titration mode: INF, Detection: mV  
◆Reference : **ASTM D2896 (Method B)** Standard Test Method for Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration

## Apparatus

Automatic titrator : GT-200  
Electrodes : Reference Electrode sleeve type, Glass electrode  
Reference electrode solution : Saturated sodium perchlorate in acetic acid solution  
(Fill the supernatant liquid into the electrode)

## Reagents

[Titrant]

■0.1mol/L . perchloric acid in acetic acid for non-aqueous titration

[Prepared reagents]

■Saturated sodium perchlorate in acetic acid: Saturate sodium perchlorate mono-hydrate, special grade reagent, in acetic acid. Crystal of excessed sodium perchlorate has to be appeared in the liquid.

[Commercial reagents]

■Chlorobenzene (special grade reagent)

■Acetic acid (special grade reagent)

## Analytical Procedure

[Blank measurement]

- (1) Add 40ml of chlorobenzene and 20ml of acetic acid into a 100ml beaker.
- (2) Titrate with 0.1mol/L .perchloric acid in acetic acid titrant

[Sample measurement]

- (1) Add proper size of sample depends on the estimated base number, 1g in this sample, into a 200ml beaker.
- (2) Add 40ml of chlorobenzene and 20ml of acetic acid into the above mentioned beaker.
- (3) Titrate with 0.1mol/L . perchloric acid in acetic acid titrant

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[ Calculation ]

$$\text{Base number ( mg KOH/g )} = ( A1 - BL ) \times M \times E \times f \times FW / S \times R$$

(Used prefixed formula on GT-200)

A1 : Titration volume of 0.1mol/L- perchloric acid in acetic acid titrant for sample measurement (ml)

BL : Titration volume of 0.1mol/L- perchloric acid in acetic acid titrant for Blank measurement (ml)

M : Molarity of 0.1mol/L- perchloric acid in acetic acid titrant (0.1)

E : Equivalent number of 0.1mol/L- perchloric acid in acetic acid titrant (1)

f : Factor of 0.1mol/L- perchloric acid in acetic acid titrant

FW : Formula weight of potassium hydroxide (56.1)

S : Sample size (g)

R : Dilution rate (1)

**Other Requirements**

■Before measurement, check the electrodes with the following procedure.

(1)Dissolve 0.1g of potassium hydrogen phthalate, standard material for volumetric analysis, into 60ml of acetic acid. And, record potential with immersing the electrode into the solution.

(2)After washing the electrodes by chlorobenzene, record potential with immersing them into mixed solution, 50ml of acetic acid and 0.75ml of 0.1mol/L-perchloric acid in acetic acid solution.

Check the difference in between the above two potentials. Tolerance: above 0.3V (300mV)

■After a measurement, wash the electrodes by chlorobenzene or the titration solvent and remove contaminations completely with wiping by soft paper, cloth etc. if necessary. Then, immerse the electrodes in pure water for 5min. as conditioning. It causes unstable potential and less repeatable results that omission of cleaning and conditioning.

■Confirm reagent labels and safety data sheets for safety

■Wear protective equipment (eye protector, gloves and others.) when handling reagents.

**Measurement Results**

	Sample size(g)	Titrant (ml)	Results(mg KOH/g)
1	1.0046	0.7839	4.31
2	1.0051	0.7795	4.28
3	1.0091	0.7822	4.28

N 3

Average 4.29

SD 0.016

RSD(%) 0.37

Base number in diesel engine oil (10W-30) is measured by GT-200. Average of three measurements on the perchloric acid titration is around 4.29mgKOH / g. The result is repeatable on RSD(%)=0.37%.

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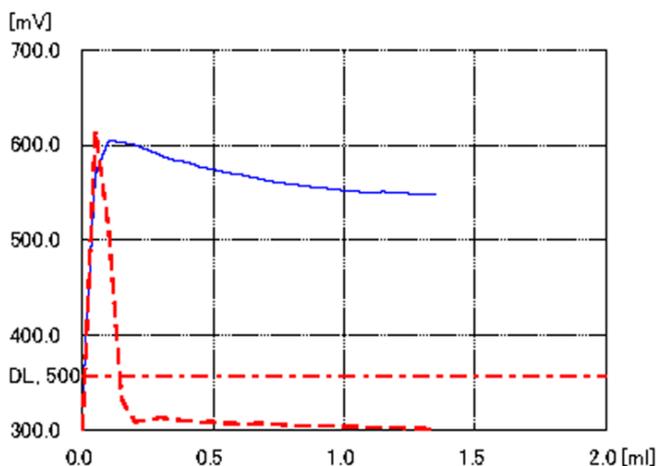
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ID No. : 1R GT No.1

User : GT-200

Measurement : 2014/09/12 10:32  
Sample Name : BLANK

Type : Sample Titr  
Sample Size : 60 [ml]



P-initial : 330 [mV]  
Start) : 0 [ml] 330 [mV]  
End) : 1.35 [ml] 549 [mV] Measuring Time : 2' 0"

File No. : 15 OIL / Base Number  
Titr File No. : 43 Base Number B/Blank  
Mode : INF End1, End1 Width : 500 [mV] ± 1000 [mV]  
Detect : mV1  
BRT No. : 1  
Reagent : 9  
WTint : 10 [sec]  
Vup : 50 [μl]  
Vlow : 50 [μl]  
dE : 3 [mV]  
dT : 3 [sec]  
DL : 500 [mV/ml]  
DetCnt : 1  
Vmax : 5 [ml] C1 : A1  
Vover : 0.05 [ml] [ml]

Reag : 0.1M HClO4/AcOH E : 1 M : 0.1 [Mol/l]  
F : 0.9845

Buret Injection Speed) : 250 [ul/sec]

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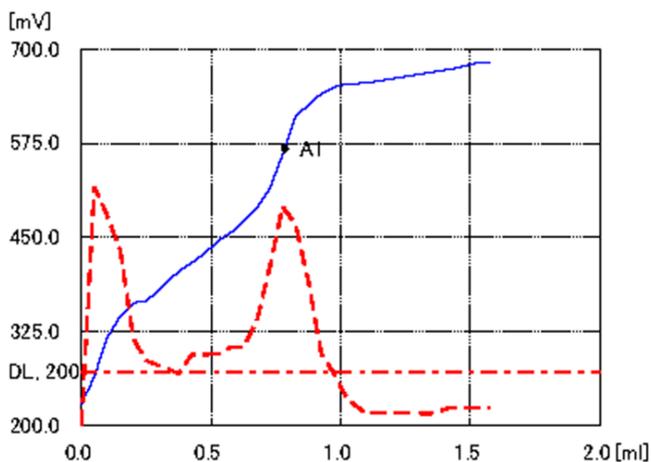
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ID No. : 7 GT No.1

User : GT-200

Measurement : 2014/09/12 15:15  
Sample Name : Diesel oil

Type : Sample Titr  
Sample Size : 1.0046 [g]



C1 : 4.31 [mg KOH/g]

A1 : 0.7839 [ml] 568 [mV]

P-initial : 228 [mV]  
Start : 0 [ml] 228 [mV]  
End : 1.575 [ml] 683 [mV] Measuring Time : 3'21"

File No. : 15 OIL/Base Number  
Titr File No. : 46 Base Number B  
Mode : INF End1, End1 Width : 600 [mV] ± 100 [mV]  
Detect : mV1  
BRT No. : 1  
Reagent : 9  
WTint : 30 [sec]  
Vup : 250 [μl]  
Vlow : 50 [μl]  
dE : 3 [mV]  
dT : 3 [sec]  
DL : 200 [mV/ml]  
DetCnt : 6  
Vmax : 20 [ml] C1 : (A1-BL)\*M\*E\*f\*FW/S\*R  
Vover : 0.5 [ml] [mg KOH/g]

Reag : 0.1M HClO4/AcOH E : 1 M : 0.1 [Mol/l]  
F : 0.9845 BL : 0 [ml]  
FW : 56.1 R : 1

Buret Injection Speed : 250 [ul/sec]