

Petroleum

Iodine value in Bio Diesel Fuel (BDF)

Redox titration by
Automatic Potentiometric Titrator

Standard	JIS	K 0070	K2390
	ASTM	D1959	
	ISO	3961	

1. Abstract

The iodine value is expressed in grams of iodine for the amount of halogens linked with 100g test sample, and is used as degree of unsaturated bond of fats and oils or Bio Diesel Fuel.

The test method to determine iodine value of Bio Diesel Fuel (BDF) is standardized as in ASTM D1959-97 Standard Test Method for Iodine Value of Drying Oils and Fatty Acids, ISO 3961:1996 Animal and vegetable fats and oils – Determination of iodine value, and JIS K 0070-1992 Test Method for Acid value, Saponification number, Ester number, Iodine number, Hydroxyl value of Chemical products and Unsaponifiable matter.

The test sample is made out of bio diesel fuel dissolved in cyclohexane, added in excess with Wijs reagent (0.1mol/L iodine monochloride acetic acid solution), left in a dark room for 30 minutes reaction, then added with potassium iodide, potentiometric titrated with 0.1mol/L sodium thiosulfate solution for the excessive iodine monochloride to the endpoint, which is the highest inflexion point.

The iodine value of BDF is calculated from titration volume of sodium thiosulfate solution.

2. Reference

- 1) JIS K 0070-1992 Test Method for Acid value, Saponification number, Ester number, Iodine number, Hydroxyl value of Chemical products and Unsaponifiable matter
- 2) ASTM D1959-97 Standard Test Method for Iodine Value of Drying Oils and Fatty Acids
- 3) ISO 3961:1996 Animal and vegetable fats and oils – Determination of iodine value
- 4) JIS K 2390 Automotive fuels-Fatty acid methyl ester (FAME) as blend stock

3. Cautions in measurement

- 1) Wijs reagent (0.1mol/L iodine monochloride acetic acid solution) is added to 50~60% in excess.
- 2) Wijs reagent is liable to change, and blank titration is required accordingly.
- 3) When Wijs reagent and sample react, iodine tends to evaporate. Therefore, use a 200mL conical flask with joint stopper.

4. Post-measurement care

No special note

5. Test equipment

Main unit : Automatic potentiometric titration system (preamplifier: STD)

Electrode : Combination platinum electrode C-778 (junction liquid: 3.33M-potassium chloride solution)

6. Reagent

Titrant : 0.1mol/L sodium thiosulfate (f = 1.003)

Additive : Cyclohexane

Wijs reagent (iodine trichloride 7.9g and iodine 8.9g each dissolved in acetic acid, then, mixed together to make it total 1L)

100g/L potassium iodide solution

7. Test procedure

—Measurement—

- 1) Take 0.3g sample in a 200mL conical flask with stopper.
 - 2) Add approximately 10mL cyclohexane, and dissolve.
 - 3) Add Wijs reagent, and stir.
 - 4) Plug the flask with stopper, and leave it for 30 minutes in a dark room at room temperature.
 - 5) Then, add 20mL of 100g/L potassium iodine solution and 100mL pure water.
 - 6) Titrate with 0.1mol/L sodium thiosulfate solution, and obtain iodine value.
- * Obtain blank level beforehand by performing blank test likewise.

8. Calculation formula

Iodine value (g/100g) = (BL1 – EP1) × TF × C1/s

BL1 : blank level (47.5850mL)

EP1 : Titration volume (mL)

TF : Factor of titrant (1.003)

C1 : Concentration conversion coefficient (1.269)

(iodine (mg) equivalent to 0.1mol/L Na₂S₂O₃·5H₂O 1mL)

S : Sample amount (g)

9. Example of measurement

-Ambient conditions-

Room temperature : 25°C	Humidity : 61%	Weather : Fair
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The below parameters and titration curve are printed out when titrated by AT-610 unit.

-Titration parameters-

Model : AT-610	
Serial No. : AT-00001	
Method No./Name : 01/Iodine value	[Calculation parameter]
Titr. mode : Auto Titr	Calc. type : Sample
Titr. form : EP Stop	CO1 : 0n
	Unit : g/100g
	Formula : (BL1-EP1)*TF*CI/S
[Titration parameter]	EP position : EP1
Burette No. : 1	Decimal : 2
Max volume : 60.00 mL	Fraction : Half adjust
Channel, Unit	Evaluation : Off
Ctrl. : Ch1, mV	
Ref. : Off	Active constant
pH polarity : Standard	C1 : 1.269
Direction : Auto	
Wait time : 0 s	
Dose mode : None	
	[Reagent parameter]
	Burette No./Reagent name
	01/0.1mol/L Na2S2O3
	[Reagent info]
	No. : 1
	Reagent name :
	01/0.1mol/L Na2S2O3
	TF : 1.003
	[Blank list]
	Blank1 : 47.58500
	[Control parameter]
Number of EP : 1	
End sense : Auto	
Gain : 1	
Data samplong : Std.	
Ctl. speed mode : Std.	
Other control : Std.	
Stir. speef : 4	

-Titration results-

Level(ctrl.)[mV]	145.0	386.3
0.0000 mL		
22.0000 mL		
*** Result ***		
Method No./Name :	01/Iodine value	
Sample No. :	01-01	
Date :	2007/07/30 11:50	
Titr. Reagent Name :	01/0.1mol/L-Na2S2O3	
Titr. time :	00:04:11	
Size :	<u>0.3317 g</u>	
End point-1		
Volume :	<u>18.2186mL</u>	
Potential :	246.5 mV	
Conc-1 :	<u>112.69g/100g</u>	

Meaning of parameters on printout:

«Titration parameter»

Burette No.: the burette used for titration / Max Volume: of titration
 Channel,Unit: Detector number and the unit of detected potential
 pH polarity: direction of **pH potential** / Direction: of titration / Wait Time: before titration starts
 Dose mode: the way of fixed dose

«Control parameter»

Number of EP: **total EPs** / End sense: direction / Gain: sensitivity of detection signal
 Data sampling: conditions / Ctl. speed mode: Control speed mode
 Other control: / Stir Speed: of stirrer

«Calculation parameter»

Calc.Type: calculation type / CO1: concentration formula 1 / Unit: of calculated results
 BL1: blank level / EP1: first endpoint / TF: factor / C1(mg/mL): concentration conversion coefficient
 / K1: unit conversion coefficient S: sample size / EP position: for calculation /
 Decimal number of digits after decimal point
 Fraction: rounding fraction / Evaluation of calculated results

-Measurement data-

n	Sample (g)	Titrated (mL)	Iodine value (g/100g)	Statistics	
				Mean	SD
1	<u>0.3317</u>	<u>18.2186</u>	<u>112.69</u>	112.44 g/100g	0.5722 g/100g
2	0.3117	20.0750	112.34		
3	0.3160	19.5926	112.75		
4	0.3239	19.2113	111.50		
5	0.2902	21.8341	112.95		

				RSD	0.5089 %
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* The data were obtained from 5 tests of the same sample.

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

10. Summary

The iodine value is expressed in grams of iodine for the amount of halogens added with 100g test sample, and is indicator of number of unsaturated bond in the molecule of fats , oils or Bio Diesel Fuel.

This measurement used the automatic potentiometric titrator presented a result with good repeatability :0.5% relative standard deviation.