HIRANUMA APPLICATION DATA	Automatic Titrator	Data No.	A10	Nov 27, 2023
FOOD	**Peroxide value measurement for cooking oil**			

1. Abstract

Degraded fats and oils include peroxide chemical species. Therefore, determination of peroxide value is an effective analytical method to know the index of fat and oil degradation. This method is described in a variety of official standards in fats, oils, health science and pharmaceutical area.

Definition of peroxide value depends on respective standard method. For example, “Method of Analysis in Health Science” in Japan defines peroxide value as “milliequivalents of iodine generated from potassium iodide in 1 kg of sample [meq/kg]” (Formula (1)). The generated iodine is finally titrated with sodium thiosulfate (Formula (2)).

Example of titration for peroxide value in cooking oil is introduced here. The experimental procedure conforms to the “Japanese Agricultural Standards”.

$$R-OOH + 2I^- + 2H^+ \rightarrow R-OH + I_2 + H_2O \quad \cdots (1)$$

$$I_2 + 2Na_2S_2O_3 \rightarrow 2NaI + Na_2S_4O_6 \quad \cdots (2)$$

2. Configuration of instruments and Reagents

(1) Instruments

Main unit	:	Automatic Titrator COM series
Electrode	:	Platinum-Reference combination electrode(long type) PR-733BZ
Options	:	Shaft modifications to the stirrer supplied with the unit, buret tip modifications and modifications to the tube holder and other ancillary components are required. (Refer to “5. Note (4)”)

(2) Regents

Titrant	:	0.01 mol/L Sodium thiosulfate standard solution
Additive	:	Saturated potassium iodide solution Dissolve 80 g of potassium iodide in 50 mL of DI water.
Solvent	:	Mixed solvent of 2,2,4-trimethylpentane and acetic acid with 2 : 3 ratio [v/v]
Purge gas	:	Nitrogen gas

3. Measurement procedure

(1) Take about 10 g of the sample into 200 mL Erlenmeyer flask with stopper and weigh it accurately.
(2) Add 60 mL of mixed solvent to dissolve sample.
(3) The air in flask is purged with nitrogen gas for 1 minutes. The flow rate of nitrogen gas is 300 mL/min.
(4) Add 1 mL of saturated potassium iodide solution with pipette. Close the flask with stopper and swirl gently for 1 minute.
(5) Leave the flask to stand for 5 minutes in dark place.
(6) Add 60 mL of DI water to the flask.
(7) Immerse the electrode and titrate with 0.01 mol/L sodium thiosulfate standard solution.
(8) Measure the blank value by testing of (2) ~ (7) without sample.
(9) After the measurement finished, the electrode should be immersed in mixed solvent, and additionally washed with DI water. Besides immerse it in DI water between each measurement to activate electrode.

4. Measurement conditions and Results

Examples of titration conditions

Measurement of blank

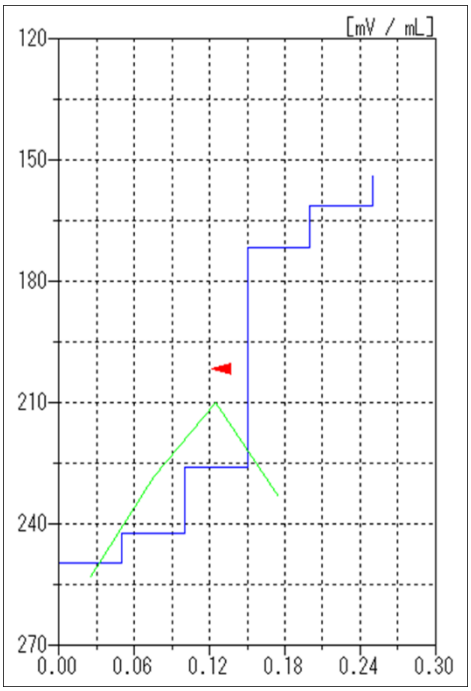
Condition No. 1								
Method	Auto		Constant No.	1		Mode No.	15	
Buret No.	1		Size	0.00000	g	Pre Int	1	sec
Amp No.	1		Blank	0.0000	mL	Del K	3	
D.Unit	mV		Molality	0.0100	mol/L	Del Sens	0	mV
S-Timer	30	sec	Factor	1.00400		Int time	5	sec
CP mL	0	mL	K	0.00000		Int Sens	3	mV
T-Timer	0	sec	L	0.00000		Buret Speed	2	
D.P. mL	0	mL	Unit	mL		Pulse	40	
End Sens	200		Formula	D			0.05	mL
Over mL	0	mL	Digits	3				
Max. Vol.	20	mL	Auto input Parameter		None			

Measurement of sample

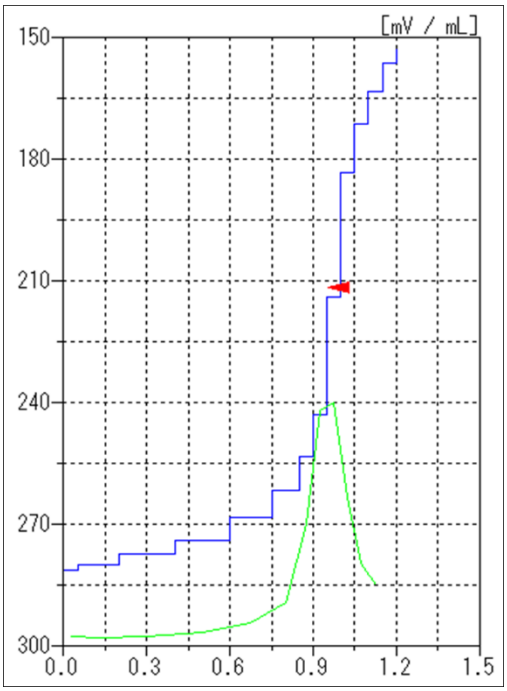
Condition No. 2								
Method	Auto		Constant No.	2		Mode No.	20	
Buret No.	1		Size	0.00000	g	Pre Int	1	sec
Amp No.	1		Blank	0.1270	mL	Del K	5	
D.Unit	mV		Molality	0.0100	mol/L	Del Sens	0	mV
S-Timer	30	sec	Factor	1.00400		Int time	5	sec
CP mL	0	mL	K	10.00000		Int Sens	3	mV
T-Timer	0	sec	L	0.00000		Buret Speed	2	
D.P. mL	0	mL	Unit	meq/kg		Pulse	40	
End Sens	200		Formula	(D-B)*K*F/S			0.050	mL
Over mL	0.2	mL	Digits	4				
Max. Vol.	20	mL	Auto input Parameter		None			

Measurement results

Sample	Measurement No.	Size (g)	Titrant volume (mL)	Peroxide value (meq/kg)	Statistical result		
BLANK	1	-	0.122	-	Avg.	0.127	mL
	2	-	0.131	-			
SAMPLE	1	10.0262	0.954	0.8281	Avg.	0.82	meq/kg
	2	10.0213	0.929	0.8035	SD	0.01	meq/kg
	3	10.0051	0.949	0.8219	RSD	1.56	%



Measurement of blank



Measurement of sample

Examples of measurement curves

5. Note

(1) Please use Erlenmeyer flask with stopper to avoid sublimation of iodine and entraining of oxygen from air.

(2) Timely measure the blank because saturated potassium iodide solution is easy to degenerate. Additionally, please periodically prepare fresh solution.

(3) The type of solvent used in this measurement, the amount of solvent added, and the time left in the dark vary with each standard test methods. Please make the appropriate modifications according to the standard test methods used.

(4) In this report, since a 200 mL Erlenmeyer flask is used as a titration vessel, platinum-reference combination electrode PR-733BZ (Long type) was used. The stirrer is modified to accommodate Erlenmeyer flasks as shown in Fig. 1 to attach the long type electrode.

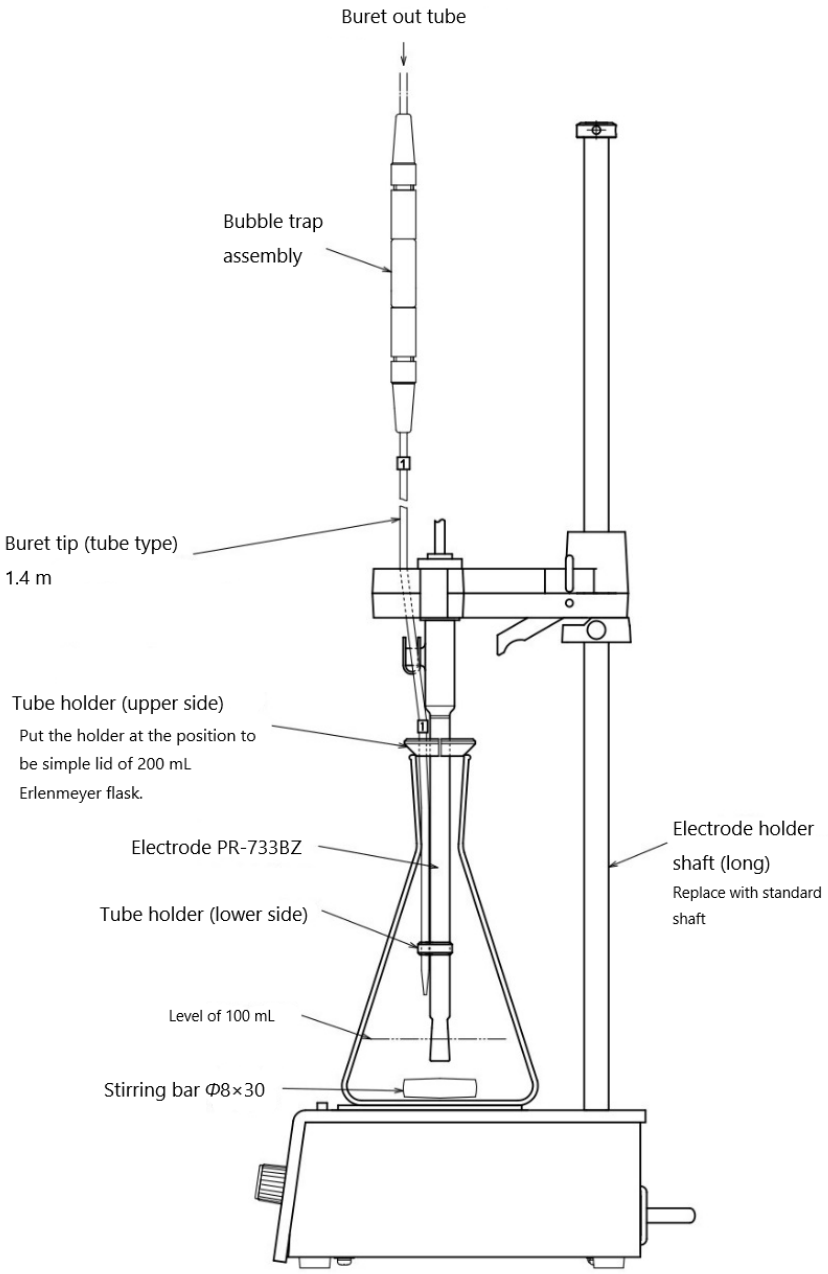


Figure 1　A set of electrode and stirrer parts for Erlenmeyer flasks

Keywords：Cooking oil, Peroxide value, Redox titration

※Some measurement would not be possible depending on the optional configuration of system.