The JOCS Standard Methods for the Analysis of Fats,Oils and Related Materials

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Contents

2.3.4 Iodine Value

2.3.4.1-1996 Iodine Value (Wijs-Cycohexane Method)

1. Definition

The iodine value is expressed in terms of the number of grams of iodine absorbed per 100 g of sample, by converting the absorbed amount of halogen to the equivalent amount of iodine.

2. Scope

Applicable to fats and oils[①].

3. Apparatus

Erlenmeyer flasks with glass-stoppers—300-500 mL, preferably with a slightly longer neck.

4. Reagents

4.1. Cyclohexane[②] (JIS K 8464).

4.2 Wijs solution[③].

4.3 10 g/100 mL Potassium iodide solution.

4.4 0.1 mol/L Sodium thiosulfate standard solution.

4.5 Starch solution.

5. Procedure

5.1 Weigh accurately the specified amount of sample into an Erlenmeyer flask according to the sample size corresponding to the estimated iodine value in Table 1.

5.2 Add 10 mL of cyclohexane into the flask and dissolve the sample completely[②,④].

5.3 Add accurately 25 mL of Wijs solution into the flask then put the stopper on the flask and shake it gently. Immediately store the flask in a dark place at ambient temperature for the required reaction time in Table 1[①] and shake the flask occasionally during the reaction time.

5.4 Add 20 mL of 10 g/100 mL potassium iodide solution and 100 mL of distilled water, and shake the flask well.

5.5 Titrate with 0.1 mol/L sodium thiosufate standard solution with vigorous shaking. When the yellow color of the sample solution has faded, add several drops of starch solution, and continue titration[⑤].

Titration is completed when the faint purple color just disappears.

5.6 At the same time blank test should be conducted by the same procedure[⑥].

5.7 Results must be recorded with the employed method, i.e., Wijs-cyclohexane method.

5.8 Calculations

$$\text{Iodine value} = \frac{(A - B) \times F \times 1.269}{C}$$

Where—

A = Required volume of 0.1 mol/L sodium thiosulfate standard solution for blank (mL).

B = Required volume of of 0.1 mol/L sodium thiosulfate standard solution for samples (mL).

F = Factor of 0.1 mol/L sodium thiosulfate standard solution.

C = Weight of sample (g).

Table 1

Estimated Iodine value	<3	3-(10)	10-(30)	30-(50)	50-(100)	100-(150)	150-(200)	200≦
Size of sample (g)	5-3	3.0-2.0	2.5-0.6	0.60-0.40	0.30-0.20	0.20-0.12	0.15-0.10	0.12-0.10
Reaction time (min)	30	30	30	30	30	60	60	60

(Less than the value)

Notes

① When the iodine value is determined on samples having conjugated unsaturated fatty acids or deteriorated oils, the results show lower value than the actual unsaturation. The results of iodine value of the samples containing cyclopropenic acids and/or antioxidants appear to give even some fluctuation. For these samples the conditions of the measurement, i.e. excess rate of Wijs solution, reaction time and reaction temperature, should preferably be recorded. For the measurement of tung oil, oiticica oil and dehydrated castor oil, use 155±3% of Wijs solution and allow it to react 1 h at 20-25°C.

② Cyclohexane should be fresh.

When a sample is hardly soluble to cyclohexane, the amount of cyclohexane can be increased properly. However, the results of this measurement tend to give lower values[1]. Therefore, it is preferable to use as little amount of cyclohexane as possible. In case of measuring fats and oils with low solvency such as extremely hardened oil, about 50 mL of cyclohexane is required to dissolve completely. Also, in case of measuring the varied amount of solvent, the same amount of solvent should be used for the blank test.

③ Wijs solution containing a slightly excessive iodine is more stable. The results of measurement give even higher values in case of the excessive chlorine content. Wijs solution should be stored in a brown bottle in a dark place. Wijs solution which is frozen during winter should be used after thawing at around below 40°C.

④ Dissolved samples in the solvent are so sensitive to air and light that Wijs solution should be immediately added into the flasks[2]. Melted samples by heating should be cooled before addition of Wijs solution.

⑤ In titration, add starch indicator after the color of the solution turns to faint yellow. Otherwise, some errors occur because color change at the end point is hardly distinguished. Approaching to the end point of titration, a drop by drop titration with vigorous shaking is required to migrate iodine from cyclohexane to aqueous layer.

⑥ When halogen absorption is more than 50% for samples of unknown iodine value, the sample

size should be reduced.

References

1) *J. Jpn. Oil Chem. Soc.*, **40**, 592 (1991).
2) AOCS Official Method Cd 1-25.