IODINE NUMBER (Wijs Method)

PRINCIPLE

The vegetable oil sample is reacted with an excess of iodine monochloride solution (Wijs reagent) under controlled conditions. Halogens add quantitatively to the double bonds in the unsaturated fatty acids, principally oleic and linoleic acids in the case of corn oil. Unreacted halogens are determined by titrating with thiosulfate. The iodine number is defined as the grams of halogen, expressed as iodine, reacting with 100 g of oil.

SCOPE

The procedure is applicable to fats and oils from corn, grain sorghum and other vegetable sources, which contain no significant amount of conjugated unsaturation (Note 1).

SPECIAL APPARATUS

Reaction Flasks: Standard 250 mL iodine flasks are recommended. Erlenmeyer flasks, 250 mL capacity, with standard-taper covers may be used as a substitute.

REAGENTS

1. Carbon Tetrachloride: Reagent grade

2. Iodine Monochloride, Wijs Reagent, 0.22 *N*: Fisher Scientific Co., Cat. No. SI106-1, or equivalent reagent from other suppliers. For laboratory preparation of Wijs reagent, see Appendix I. If determination of halogen ratio is desirable, see Appendix II.

3. Starch Indicator Solution, 1%

4. Sodium Thiosulfate Solution, 0.1 *N*: Standard

5. Potassium Iodide Solution, 30%: Dissolve 30 g of KI in purified water and dilute to 100 mL.

IODINE NUMBER (Wijs Method) — continued

SPECIAL SAFETY NOTE

Carbon tetrachloride is a carcinogen in laboratory animals. Avoid breathing vapors or skin contact. Work in a well ventilated hood but avoid dispersing to atmosphere.

PROCEDURE

Weigh accurately 0.22-0.25 g of dry corn oil (Notes 2 and 3) into a 250 mL Erlenmeyer flask, and add 20 mL of carbon tetrachloride. Add 20 mL of carbon tetrachloride to each of two additional flasks to serve as blanks. Pipet 25.0 mL of Wijs reagent into each flask. Stopper flasks, mix contents by swirling and store in a dark place at 25 ± 5 °C.

At the end of 30 mins., add 10 mL of 30% potassium iodide solution and 100 mL of purified water to the sample solution. Titrate immediately with standard 0.1 *N* thiosulfate solution until the yellow color almost disappears. Add 1 mL of starch indicator solution and titrate dropwise with vigorous swirling to disappearance of the blue starch-iodine color (Note 4).

Titrate the blanks in the same manner. If the sample titer is not between 50 and 60% of the average blank titer, adjust sample size accordingly and repeat the determination.

CALCULATION

$$\text{Iodine Number} = \frac{(\text{Blank Titer - Sample Titer, mL})(0.01269)(100)}{\text{Sample Wt., g}}$$

NOTES AND PRECAUTIONS

1. Electron structure of the halogen and the presence or absence of double conjugation determine completeness of the addition reaction and the extent of possible substitution reactions. Wijs reagent is chosen to give complete addition to isolated double bonds with almost no interference from substitution reactions. Addition to conjugated double bonds is not complete under the test conditions. It is necessary to maintain close control of reaction conditions to assure reproducible results.

IODINE NUMBER (Wijs Method) — continued

2. If the iodine number is to be characteristic of the oil, the sample should be substantially free of moisture and foreign materials.

3. The iodine number of refined corn oil is usually about 125, and this value is assumed for the recommended sample size. For samples with higher or lower iodine numbers, use proportionately smaller or larger samples, respectively.

4. Agitation must be vigorous to assure removal and titration of iodine dissolved in the carbon tetrachloride.

APPENDIX I

Iodine Monochloride Solution, Wijs Reagent: Dissolve 26.0 g of reagent grade iodine (I_2) in 2 L of reagent grade glacial acetic acid, heating gently if necessary to hasten solution. Cool to room temperature. Pipet 25 mL into a 250 mL Erlenmeyer flask. Add 100 mL purified water and titrate immediately with standard 0.1 *N* thiosulfate solution until the yellow iodine color almost disappears. Add 1 mL of starch indicator solution and complete the titration dropwise with vigorous swirling to the disappearance of the blue starch iodine complex. This is the original titer.

Remove 100 mL of the iodine solution (solution A), and store in a glass-stoppered bottle, for adjustment of the halogen ratio if necessary.

Dry chlorine gas from a cylinder by passing through a gas-washing bottle charged with concentrated sulfuric acid, and bubble the dried gas through a gas dispersion tube into the bulk of the iodine solution. Pass chlorine into the solution until the original titer is not quite doubled.

Check titer of reagent after chlorine addition by pipetting a 25.0 mL aliquot into 250 mL Erlenmeyer flask. Add 100 mL of distilled water, 10 mL of 30% potassium iodide solution, and titrate with standard 0.1 *N* thiosulfate solution as outlined in preceding paragraph. If original titer is doubled or greater, add a portion of iodine solution A and recheck.

IODINE NUMBER (Wijs Method) — continued

Store the Wijs reagent in a glass-stoppered amber bottle. Prepare fresh reagent every 30 days.

APPENDIX II

Determination of Halogen Ratio in Wijs Reagent: The halogen ratio (I_2:Cl_2) of the Wijs reagent shall be 1.10 ± 0.10. If the ratio is not in the prescribed range, add iodine or chlorine as required. The procedure for determining the ratio is:

Iodine Content: Pour 150 mL of saturated chlorine water into a 500 mL Erlenmeyer flask and add some glass beads. Add 5.0 mL of Wijs reagent by pipet, shake and heat to boiling. Boil briskly for 10 mins., cool and add 30 mL of 2% sulfuric acid solution and 10 mL of 30% potassium iodide solution.

Mix well, add 1 mL of starch indicator solution and titrate immediately with standard 0.1 *N* thiosulfate solution to disappearance of the blue starch-iodine color.

Total Halogen Content: Pour 150 mL of recently boiled purified water into a clean 500 mL Erlenmeyer flask. Add 10 mL of 30% potassium iodide solution. Pipet 20.0 mL of Wijs reagent into the flask and mix well. Add 1 mL of starch indicator solution, and titrate immediately with standard 0.1 *N* thiosulfate solution to disappearance of the blue starch-iodine color.

Calculate Halogen Ratio:

$$I_2 : Cl_2 \text{ Ratio} = \frac{2A}{3B - 2A}$$

where: A = Iodine content titer
B = Total halogen content titer

In the method, it is important that all volumetric measurements be made at the same temperature because acetic acid solutions have a large coefficient of expansion.