AOAC Official Method 965.33
Peroxide Value of Oils and Fats

Titration Method
First Action 1965
Final Action 1969

AOCS-AOAC Method

(*Note*: Conduct analysis in diffuse daylight or in artificial light shielded from direct light source.)

A. Reagents

(**a**) *Acetic acid-chloroform solution.*—Mix 3 volumes CH_3COOH with 2 volumes $CHCl_3$, USP.

(**b**) *Potassium iodide solution, saturated.*—Dissolve excess KI in freshly boiled H_2O. Excess solid must remain. Store in dark. Test daily by adding 0.5 mL to 30 mL CH_3COOH-$CHCl_3$, (**a**); then add 2 drops 1% starch solution, (mix ca 1 g soluble starch with enough cold H_2O to make thin paste, add 100 mL boiling H_2O, and boil ca 1 min while stirring). If solution turns blue, requiring >1 drop 0.1M $Na_2S_2O_3$ to discharge color, prepare fresh solution.

(**c**) *Sodium thiosulfate standard solutions.*—0.1 and 0.01M. Prepare and standardize as in **942.27** (*see* A.1.13). For 0.01M, dilute 0.1M with freshly boiled and cooled H_2O.

B. Determination

(**a**) *Fats and oils.*—Weigh 5.00 ± 0.05 g test portion into 250 mL glass-stoppered Erlenmeyer. Add 30 mL CH_3COOH-$CHCl_3$, (**a**), and swirl to dissolve. Add 0.5 mL saturated KI solution, (**b**), from Mohr pipet, let stand with occasional shaking 1 min, and add 30 mL H_2O. Slowly titrate with 0.1M $Na_2S_2O_3$ with vigorous shaking until yellow is almost gone. Add ca 0.5 mL 1% starch solution, and continue titration,

shaking vigorously to release all I_2 from $CHCl_3$ layer, until blue just disappears. If <0.5 mL 0.1M $Na_2S_2O_3$ is used, repeat determination with 0.01M $Na_2S_2O_3$.

Conduct blank determination daily (must be 0.1 mL 0.1M $Na_2S_2O_3$). Subtract from test portion titration.

Peroxide value (milliequivalent peroxide/kg oil or fat) = S M 1000/g sample, where S = mL $Na_2S_2O_3$ (blank corrected) and M = molarity $Na_2S_2O_3$ solution.

(**b**) *Margarine.*—Melt fat by heating with constant stirring on hot plate at low heat, or heat in air oven at 60-70°C. (Avoid excessive heat and long exposure >40°C.) When completely melted, hold in warm place until aqueous portion and most of solids have settled. Decant oil into clean beaker and filter through Whatman No. 4, or equivalent paper. Do not reheat unless necessary to obtain clear filtrate. Proceed as in (**a**).

References:

J. Am. Oil Chem. Soc. **26,** 345(1949).
AOCS Method Cd 8-53.
JAOAC **48,** 175(1965).