Thermo. Titr. Application Note No. H-076

Title:	**Determination of Iodine Value in Fats and Oils**

***Scope*:**	Determination of Iodine Value (IV) in fats and oils.

***Principle*:**	Iodine value (IV) is a measure of the total number of double bonds present in fats and oils. It is expressed as the *"number of grams of iodine that will react with the double bonds in 100 grams of fats or oils"*. The determination is conducted by dissolving a weighed sample in a non-polar solvent such as cyclohexane, then adding glacial acetic acid. The double bonds are reacted with an excess of a solution of iodine monochloride in glacial acetic acid ("Wijs' solution"). Mercuric ions are added to hasten the reaction. After completion of the reaction, the excess iodine monochloride is decomposed to iodine by the addition of aqueous potassium iodide solution, which is then titrated with standard sodium thiosulfate solution.

Reagents:	*Titrant:* 0.1 mol/L Na2S2O3 solution (may be standardized against KIO3) *Wijs' solution:* 0.1 mol/L ICl (iodine monochloride) in glacial acetic acid. Riedel-de Haën cat. no. 35071 *Potassium iodide:* 15% w/v KI solution in DI water *Mercuric acetate solution:* Dissolve 2.5g mercuric acetate in 100mL glacial acetic acid. Alternatively, dissolve 2.1g mercuric chloride in 100mL glacial acetic acid

Method:	Basic Experimental Parameters:	
	Titrant delivery rate (mL/min.)	5
	No. of exothermic endpoints	1
	Data smoothing factor	55
	Stirring speed (802 stirrer)	15
	Wijs' solution calibration procedure: Prepare a titration program to dispense Wijs' solution in amounts of 0.5, 1.0, 1.5, 2.0 and 2.5mL into 30mL DI water with the addition of 10mL KI solution. Titrate with 0.1 mol/L $Na_2S_2O_3$ solution Prepare three Dosinos with the reagents in section 1 above Plot mL Wijs' solution (y-axis) against mL 0.1 mol/L $Na_2S_2O_3$ (x-axis). Compute the	

gradient and y-intercept. This enables computation of the excess of Wijs' solution in terms of mL 0.1 mol/L $Na_2S_2O_3$ solution In the Titrotherm software, open Edit>Setup. Place the gradient factor in the "mol/L" box and the y-intercept value in the "Blank" box. This will permit automatic computation of the IV in the dedicated Excel spreadsheet.

Titration Procedure:

Weigh accurately approximately 0.2g of liquid fat or oil into a titration vessel. In the case of fats which are solid at room temperature, warm gently in a microwave oven beforehand. Add 10mL cyclohexane to dissolve, add 0.5mL mercuric acetate solution and 20mL glacial acetic acid. In the case of high melting point fats, it is useful to add the cyclohexane to the titration vessel before weighing. Dropping the fat directly into the cyclohexane aids dissolution. However, it is important to rapidly tare the balance and add the sample, just allowing the balance to stabilize before recording the mass.

The titration procedure is designed to eliminate operator involvement in the determination. The Wijs' solution is added, with a 300 seconds (5 minutes) wait programmed before 10mL of 15% KI solution is added. The titration commences automatically. The reaction with the Wijs' solution should be carried out in the dark, although low room lighting is probably satisfactory.

After titration, the titration assembly is rinsed first with DI water, then with alcohol (methylated spirits). It is then gently wiped dry prior to the next titration.

Results:

	Iodine value, g I_2/100g sample	
Sample No.	**Titrotherm method**	**Reported by customer (claimed to be approximate values)**
1	33.2±0.08 (n=5)	35
2	50.5±0.05 (n=5)	52
3	50.1±0.13 (n=5)	50
4	54.9±0.13 (n=5)	56
5	33.8±0.09 (n=5)	35

Calculations:

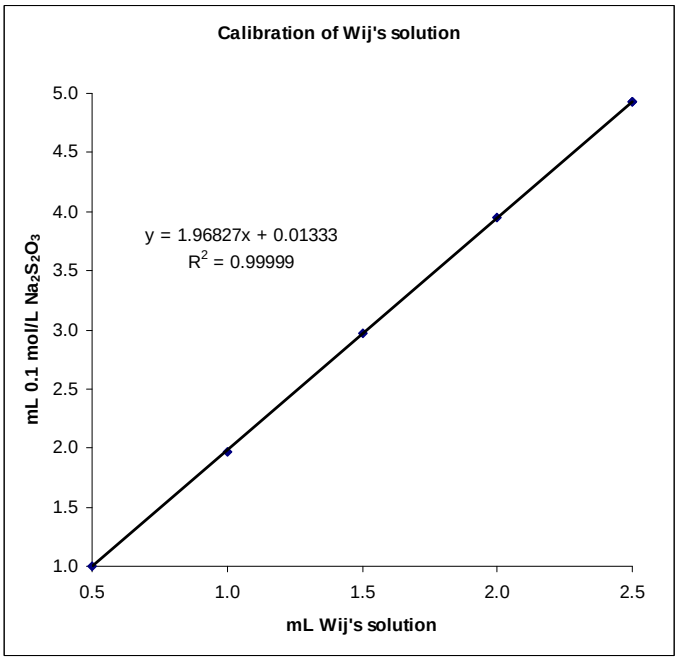
$$Iodine\ Factor(IF) = \frac{0.01269 \times M\ Na_2S_2O_3}{0.1}$$

$$Iodine\ Value\ (IV) = \frac{((blank - titration) \times IF \times 100)}{sample\ mass, g}$$

Calibration of Wijs' Solution:

The calibration factors are used to calculate the blank titration volume of 0.1 mol/L $Na_2S_2O_3$ for a given dose of Wijs' solution.

*Example: 6mL of Wijs' solution dosed. Equivalent blank of 0.1 mol/L $Na_2S_2O_3$ =6*1.98627+0.01333 = 14.775mL*



Thermometric Titration Plot:

Legend:

Red = solution temperature curve

Black =second derivative curve