SHORT COMMUNICATION

Chloramine T with Iodine: A New Reagent to Determine the Iodine Value of Edible Oils

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ABSTRACT: Chloramine T (*N*-chloro-*p*-toluenesulfonamide sodium salt) and iodine (2:1, w/w) in carbon tetrachloride and acetic acid (1:1, vol/vol), referred to as reagent (I) was found to be effective for the determination of Iodine value of edible oils. Reagent (I) reacted quantitatively with the double bonds of oils of known weight. The reagent left unreacted after 20–25 min was titrated against standard sodium thiosulfate solution (0.04 M) in presence of potassium iodide (10%, 5 mL). The difference in volume of sodium thiosulfate solution consumed by reagent (I) without and with oil was a basis to calculate the iodine value of oils used. The iodine values of different oils were also determined separately following the standard procedure of Wijs, and calculated iodine value was obtained from the gas chromatographic profile of fatty acids. The iodine value obtained by the new method was in agreement with the results of the standard methods. The results obtained indicate that the method could be a complementary or an alternative to the Wijs method.
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KEY WORDS: Chloramine T, edible oils, iodine, iodine number, Wijs method.

Iodine value (grams of iodine that react with the double bonds in 100 g of oil) is considered to be one of the important parameters to ascertain the identity and quality of edible oils (1). Different methods have been reported in the literature, but it is the Wijs method (2–4) that is commonly adopted for determining the iodine value of edible oils. Recently, chloramine T (*N*-chloro-*p*-toluenesulfonamide sodium salt) in acetic acid has been used for the quantitative determination of degree of unsaturation of oils (5). Earlier researchers showed that chloramine T adds to double bonds, and the products produced were characterized (6). Use of chloramine T has also been reported (7,8) to oxidize iodide. These observations have inspired the thought that chloramine T in the presence of iodine probably would be a better reagent than chloramine T in acetic acid (5). Once reagent is obtained, the rest of the procedure to be followed will in principle be similar to the Wijs method (2–4).

EXPERIMENTAL PROCEDURES

Materials. Chloramine T (Rolex Bombay, India, Rhodia, France); potassium iodide (IDPL, Hyderabad, India); carbon tetrachloride (Fisher Madras, Quligen Bombay, India); acetic acid (NICE Cochin, SD Fine Chemicals, Boisar, India); iodine, sodium thiosulfate, and potassium dichromate (SD Fine Chemicals, Boisar, India); and iodine monochloride (BDH-E Merck, Bombay, India) were used without further purification. Water used was distilled water.

The balance used was a Mettler H51AR (Neo Pharma Instruments Corporation, Calcutta, India; and Keroy Electrical, Calcutta, India). The gas chromatograph was Shimadzu GC–15A model with microprocessor attached (Shimadzu, Kyoto, Japan).

Preparation of reagent (I). Chloramine T (2.5 g) was dissolved in 75 mL of acetic acid taken in a dry beaker, and 1.25 g of iodine was dissolved separately in 75 mL of carbon tetrachloride. Both solutions were transferred to a 250-mL volumetric flask. The solution was diluted to the mark with carbon tetrachloride and acetic acid (1:1, vol/vol). The flask was stoppered and shaken well. The resulting nearly homogeneous brown solution was kept at room temparature for about 1 h. During that period there was a small amount of white precipitate formed, most of which settled at the bottom of the flask. The solution was filtered through a quantitative or an ordinary filter paper. The clear brown filtrate, reagent (I), was found to be stable for a week and satisfactory to determine the iodine value of the oils.

Determination of iodine value by the proposed method. A known weight of the oil (30–140 mg) was transferred into each of five clean and dry iodine flasks. A sixth flask, containing no oil, served as a control. Twenty-five mL of reagent (I) was pipetted into each of the flasks. The flasks were stoppered and mixed well by shaking and then kept at room temperature under diffused light. After 20–25 min, 5 mL of 10% potassium iodide solution was added to each flask *via* a measuring cylinder (10 mL capacity). The flasks were stoppered and mixed well by hand shaking, and each one was titrated separately against 0.04 M sodium thiosulfate solution, which had previously been standardized with 0.05 N potassium dichromate solution. The titration was continued with frequent shaking until the pale yellow color produced toward the end point just disappeared.

The iodine value of an oil was calculated based on the following relations: One mole of chloramine T + ½ mole I_2 produces 1 mole ICl, which adds to 1 mole of double bonds. One mole of double bonds consumes 1 mole of I_2, which would

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TABLE 1
Iodine Values of Edible Oils

Oil	Experimental Values[a]					Average value	Standard deviation	Literature value	Wijs method	Calculated value[a]
Palm	57.52	58.64	56.29	57.32	58.08	57.57	0.88	54–62	58.0	58.7
	(35.2)	(76.7)	(80.5)	(85.9)	(120.4)					
Ground nut	96.12	94.60	93.52	93.60	94.20	94.40	1.05	84–100	94.0	95.5
	(32.3)	(48.4)	(67.5)	(70.5)	(100.2)					
Sunflower	136.99	135.05	131.24	131.65	132.84	133.55	1.94	100–145	136.0	135
	(35.4)	(33.5)	(66.7)	(103.3)	(142.8)					
Mustard	102.02	106.20	105.32	102.96	103.21	103.9	1.74	96–112	105.0	104.3
	(60.2)	(37.4)	(68.6)	(113.4)	(120.2)					
Coconut	8.02	8.19	10.01	8.9	8.5	8.72	0.79	7.5–10.5	8.50	8.8
	(1059.2)	(1673.9)	(1348.7)	(1563.5)	(1402.3)					
Ginjelly	114.15	112.90	110.90	109.80	110.82	111.71	1.76	103–120	110.0	109.8
	(69.7)	(96.7)	(103.4)	(112.3)	(127.3)					
Safflower	142.30	140.28	139.68	141.90	139.70	140.77	1.24	135–148	140.0	141.9
	(46.7)	(52.3)	(80.1)	(97.7)	(125.4)					
Soybean	136.21	135.82	135.26	134.91	134.10	135.26	0.81	120–141	134.5	135.66
	(36.2)	(40.8)	(69.4)	(95.6)	(130.1)					

[a]Figures in parentheses represent weight (mg) of the oil taken for estimation.
[b]From gas chromatographic analysis.

react with 2 moles of sodium thiosulfate. Therefore, 1 mL of 0.1 M sodium thisulfate solution represents 0.05 mole, or 12.69 mg, of, I_2, and iodine value of an oil = $[(V_1 - V_2) \times 12.69 \times 1000 \times M]/W$, where V_1 and V_2 are volumes in mL of sodium thiosulfate solution of molarity M consumed by a known volume of reagent (I) (25 mL = 1 mmole) without and with W mg of the oil.

Iodine value by Wijs method and calculated iodine values based on fatty acid composition. Iodine values of the oils were determined separately following the standard procedures of the Wijs method (2–4), and iodine values were also calculated using approved factors (1,9,10) from the fatty acid composition determined by gas chromatographic (GC) technique as follows. Sodium methoxide-catalyzed transesterification was used to prepare fatty acid methyl esters, which were separated on the Shimadzu GC-15A model gas chromatograph equipped with a hydrogen flame-ionization detector, under the following conditions: stainless steel column (5′ × 1/8″) packed with 15% diethylene glycol succinate on Chromosorb (80–100 mesh); column temperature, 185°C, isothermal; injector and detector ports adjusted to 240°C; carrier gas, nitrogen (15 mL/min), fuel gas, hydrogen (20 mL/min). Identification of fatty acids was made by comparison with standards (Sigma, St. Louis, MO).

RESULTS AND DISCUSSION

The iodine values of the edible oils obtained by the proposed method and the Wijs method (2–4) and calculated iodine values (1,9,10), as well as the literature values (2,3,5), are tabulated in Table 1. The fatty acid compositions of the oils determined by GC, along with calculated iodine values, are shown in Table 2. The iodine values of each oil obtained in quintet tri-

TABLE 2
Fatty Acid Compositions of Vegetable Oils Determined by Gas Chromatography

Fatty acid	Palm olein	Ground nut	Sunflower	Mustard	Coconut	Ginjelly	Safflower	Soybean
4:0	—	—	—	—	—	—	—	—
6:0	—	—	—	—	trace[a]	—	—	—
8:0	—	—	—	—	7.7	—	—	—
10:0	—	—	—	—	8.0	—	—	—
12:0	—	—	—	—	52.6	—	—	—
14:0	1.5	—	—	—	16.0	—	—	—
16:0	40.0	8.4	6.0	2.4	6.0	10.5	5.5	10.3
18:0	4.5	3.5	3.0	0.7	2.0	4.5	2.5	3.9
18:1	40.0	46.5	26.0	7.8	5.2	43.0	20.0	23.4
18:2	14.0	32.0	65.0	15.6	2.5	42.0	72.0	54.1
18:3	trace	trace	—	10.2	—	—	trace	8.3
20:0	—	1.5	—	—	—	—	—	—
20:1	—	1.5	—	4.9	—	—	—	—
22:0	—	4.5	—	—	—	—	—	—
22:1	—	—	—	58.4	—	—	—	—
24:0	—	2.1	—	—	—	—	—	—
Iodine values calculated	58.7	95.5	135	104.3	8.8	109.8	141.9	135.66

als indicate that they were reproducible, with a maximal standard deviation *ca.* 2 units. The results of the proposed method are in agreement with the results obtained by conventional methods (1–4,10) and also fall well within the range of literature values (2,3,5), indicating that the proposed method is suitable for determining the iodine values of edible oils.

The method reported in this paper has the following advantages over existing standard methods: (i) The preparation of the reagent, chloramine T with iodine, takes only about 30 min in comparison with the much longer duration needed to prepare the Wijs reagent. (ii) The method proposed here is cost effective. (iii) The proposed reagent is found to be stable for more than a week and yields satisfactory results when used in place of the existing Wijs reagent to determine the iodine values of the edible oils. (iv) The use of the chloramine T/iodine reagent reduces the reaction time to 20–25 min, compared to 1–2 h for existing methods, and reduces the amount of solvent.

However, it should be noted that the method does require the use of carbon tetrachloride, a known carcinogen, and that appropriate precautions must be taken when handling this chemical. Work is in progress that replaces carbon tetrachloride by acetic acid, cyclohexane, or a mixed solvent of both. The results will be communicated shortly.

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