LETTER TO THE EDITOR

Chloramine-T with Iodine: A New Reagent to Determine the Iodine Value of Edible Oils. Part II

Sir:

Recently we suggested a new method (1) employing chloramine-T (*N*-chloro-*p*-toluenesulfonamide sodium salt) with iodine to determine the iodine values of edible vegetable oils, since it is considered to be one of the most important parameters necessary to assess their quality and identity (2,3). The method (1) is cost-effective, and the reagent can be prepared in the laboratory in a short time, in contrast to traditional (3,4) or "official" (5–8) methods, which are dependent on Wijs reagent whose preparation is time-consuming and requires hazardous and toxic chemicals. Although chloramine-T is recorded as a topical veterinary antiseptic *(The Merck Index),* a recent study of its use as an aquaculture disinfectant reports that the gas exchange was not adversly affected in rainbow trout which were intermittently exposed to chloramine-T (9). Chloramine-T was also found to reduce cobra venom factor activity (10). However, since our method (1) involves the use of carbon tetrachloride, which is a known carcinogen, we suggest here a modification replacing carbon tetrachloride by any one among the relatively safe solvents, namely solvent I: cyclohexane + acetic acid (1:1, vol/vol) or solvent II: acetic acid, or solvent III: cyclohexane, which have been employed as alternative solvents to carbontetrachloride in the context of studies on fish oils (11–13). In the proposed modification, the reagent is once again cost-effective, can be prepared in the laboratory in a short time, is stable for more than a week, and the solvents employed are safe.

Apart from the materials already listed in Reference 1, cyclohexane (IDPL, Hyderabad, India) is used in the proposed method. Procedure: iodine, 6.5 g, was placed in a 100-mL beaker. About 400 mL of acetic acid was used in successive fractions of 20 mL to dissolve the iodine. The iodine was stirred each time with a glass rod, and the clear solution was decanted into a 500-mL volumetric flask. Chloramine-T, 15.0 g, was dissolved in the same beaker in about 80 mL acetic acid. It was transferred into the same volumetric flask that contained the iodine solution, and the contents were made up to 500 mL with acetic acid. The volumetric flask was stoppered and shaken well. The brown-colored iodine solution then turned a characteristic orange-brown without liberating heat or gas. The whole process of preparing the reagent required less than 1 h. The solution was clear. However, it was put aside for about 1 h and later decanted into a brown bottle, which was stoppered and stored under diffuse light. The reagent was found to be stable for more than 1 wk. In large-scale or commercial preparations, a magnetic stirrer may be employed to reduce risk factors, if any, in handling the chemicals in bulk.

To determine the iodine values of the oils, six iodine flasks were arranged bearing numbered labels. Different weighed amounts of clean oil were added to five flasks in the range 50–150 mg. The sixth flask was treated as a blank. Ten mL, solvent I was added to each of the six flasks, which were then swirled so as to completely dissolve the oil. To each was added 25 mL of the prepared reagent. The flasks were stoppered and swirled briefly to ensure intimate mixing before storage under diffuse light at laboratory temperature. After 30–45 min, they were removed from storage and to each was added 20 mL of 10% potassium iodide solution followed by 100 mL distilled water. Each was separately titrated against 0.1 M sodium thiosulfate solution, previously standardized with 0.1 N potassium dichromate solution. Initially sodium thiosulfate was added gradually with constant and vigorous shaking by hand until the solution became pale yellow, when 1–2 mL of freshly prepared starch indicator was added and the titration continued until the blue color just disappeared.

The calculation of iodine values of the oils follows the normal difference approach as detailed in our earlier paper (1). The same procedure was repeated with solvents II and III and with a variety of vegetable edible oils. A note of caution may be added here. It is important to employ only the specified quantity of 10 mL in the case of solvents I and III. When a larger volume like 20 mL or more is employed, a biphasic system is formed with cyclohexane forming the upper layer. Then the oil is expected to dissolve differentially (14), possibly leading to unexpected results. The color of the biphasic system is grey (12), which makes it difficult to determine the starch blue end point. Such difficulties are avoided when we use a fixed quantity, 10 mL, of each solvent. The results obtained with different solvents presented in Table 1 are in good agreement with each other. Iodine values of the oils determined following the "official" Wijs method (5) are also indicated in Table 1 for comparison. The values agree within experimental error, are reproducible with a standard deviation

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TABLE 1
Iodine Value (IV) of Edible Oils Determined by the New Chloramine-T[a]

	a		b		c		
Oil	Experimental ($n = 5$)	Average IV	Experimental ($n = 5$)	Average IV	Experimental ($n = 5$)	Average IV	IUPAC
Coconut	8.40–8.70 (1009–1820)	8.56 ± 0.11	7.98–9.20 (1028–1780)	8.58 ± 0.49	8.50–9.20 (1056–1700)	8.85 ± 0.27	8.5
Ginjelly[b]	109.40–112.40 (78–135)	110.84 ± 1.09	108.84–111.32 (88–134)	110.04 ± 1.01	108.98–113.86 (78–136)	111.23 ± 1.95	110.0
Groundnut	92.80–95.40 (71–170)	93.70 ± 1.10	92.98–94.45 (68–163)	93.80 ± 0.58	92.82–95.24 (80–156)	93.95 ± 0.89	94.0
Mustard	103.40–106.20 (96–140)	104.64 ± 1.15	103.88–105.05 (89–141)	104.36 ± 0.53	102.96–106.24 (77–137)	104.60 ± 1.37	105.0
Palm	56.80–58.20 (80–170)	57.44 ± 0.61	55.86–57.49 (72–165)	56.78 ± 0.61	56.58–58.54 (59–180)	57.40 ± 0.76	58.0
Safflower	138.80–141.20 (72–140)	139.90 ± 0.95	139.48–141.45 (69–138)	140.45 ± 0.79	138.92–142.28 (86–136)	140.08 ± 1.35	140.0
Soybean	134.20–136.40 (84–140)	135.20 ± 0.81	133.85–136.26 (80–140)	134.80 ± 0.93	134.82–136.82 (69–137)	135.66 ± 0.82	134.5
Sunflower	134.60–137.20 (86–140)	135.98 ± 0.98	134.48–136.48 (78–136)	135.29 ± 0.76	134.84–136.20 (61–137)	135.29 ± 0.55	136.0

[a] With iodine method using 10 mL, (a) cyclohexane + acetic acid (1:1, vol/vol), (b) acetic acid alone, and (c) cyclohexane alone along with IUPAC method. Figures in parentheses represent weight (mg) of the oil taken for estimation.
[b] Ginjelly oil is not common outside India.

around two, are in agreement with results obtained following the “official” method (5). All these lie within the range of values quoted in the literature (3,4).

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REFERENCES

1. Nagendrappa, G., S.S. Raj Urs, and S. Rajalakshmi, Chloramine-T with Iodine: A New Reagent to Determine the Iodine Value of Edible Oils, *J. Am. Oil Chem. Soc. 75*: 1219–1221 (1998).
2. Babu, S., R.V. Sudharshan, R.K. Sharma, and R.V. Bhat, A Simple and Rapid Polarimetric Method for Quantitative Determination of Castor Oil, *Ibid. 73*:397–398 (1996).
3. *The Prevention of Food Adulteration Act 1954,* 13th edn., EBC Publishing Pvt. Ltd., Lucknow, 1991, pp. 155–160.
4. Beckett, A.H., and J.B. Stenlake, *Practical Pharmaceutical Chemistry,* 3rd edn., 1, Part 1, C.B.S. Publishers and Distributors, New Delhi, 1986, pp. 204–205.
5. Pocklington, W.D., IUPAC, Determination of the Iodine Value of Oils and Fats, *Pure Appl. Chem. 62*:2339–2343 (1990).
6. *Official Methods and Recommended Practices of the American Oil Chemists' Society,* 4th edn., AOCS Press, edited by David Firestone, Champaign, Method Cd1b-87, 1996.
7. *Ibid*., Method Cd1d-92, 1995.
8. *Ibid.*, Method Cd1-25, 1993.
9. Powell, M.D., F. Haman, G.M. Wright, and S.F. Perry, Response of Rainbow Trout *(Oncorhynchus mykiss)* to a Graded Hypoxia Following Repeated Intermittent Exposure to Chloramine-T, *Aquaculture Amsterdam 165*:27–39 (1998).
10. Fu, Q., P. Mcphie, and D.C. Gowda, Methionine Modification Impairs the C5-Cleavage Function of Cobra Venom Factor-Dependent C3/C5 Convertase, *Biochem. Mol. Biol. Intern. 45*:133–144 (1998).
11. Ackman, R.G., E. Macpherson, and A. Timmins, Anomalous High Iodine Value of Squalene and the Impact on Iodine Values of Shark Liver Oils, *J. Am. Oil Chem. Soc. 75*:1223–1225 (1998).
12. Barlow, S.M., A.P. Bimbo, E.L. Miller, S. Thorisson, and D.E. Walters, Collaborative Test Determination of Iodine Value in Fish Oils. 1. Reaction Time and Carbon Tetrachloride or Cyclohexane as Solvents, *Ibid. 74*:1077–1083 (1997).
13. Barlow, S.M., A.P. Bimbo, E.L. Miller, S. Thorisson, and D.E. Walters, Collaborative Test Determination of Iodine Value in Fish Oils. 2. Carbon Tetrachloride or Cyclohexane/Acetic Acid as Solvents, *Ibid. 74*:1085–1088 (1997).
14. Certik, M., P. Andrasi, and J. Sajbidor, Effect of Extraction Methods on Lipid Yield and Fatty Acid Composition of Lipid Classes Containing Gamma (γ)-Linolenic Acid Extracted from Fungi, *Ibid. 73*:357–365 (1996).

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