

means for continually controlling the variables of time, temperature, and reagent on a scientific, predictable basis. By utilizing the phase diagram as a guide for the correct conditions and employing a continuous process designed to maintain these conditions, it is always possible to obtain high yield refining results on crude coconut oil.

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Estimation of Unsaturation of Fats and Oils, Using Hypochlorous Acid

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HYPOCHLOROUS ACID has been successfully employed by Mukherjee (1) and Choudhury and Mukherjee (2) for the determination of unsaturation of oils containing both nonconjugated and conjugated unsaturation. One of the difficulties in the original method proposed by the above workers is the preparation of the hypochlorous acid reagent. This is usually done by passing a stream of washed chlorine through a 4N solution of sodium hydroxide, maintaining a temperature below 10°C., then diluting to 0.1N. It is a little difficult for ordinary commercial laboratories to prepare the above reagent.

It has been recently found by the present worker that commercial bleaching solutions sold in grocery stores, such as Clorox, serve very satisfactorily as a reagent in the determination of the unsaturation of oils. When Clorox is acidified with acetic acid, hypochlorous acid is liberated, which adds on to the double bonds quantitatively. The other substances present in Clorox do not interfere. By using Clorox, the most difficult step in the hypochlorous acid method for the determination of unsaturation of oils can be eliminated. The present method is much easier than the Wijs method, and it is believed that it is particularly suitable for commercial and industrial laboratories.

Experimental

A. Fats and Oils Containing Nonconjugated Unsaturation

Reagents

Clorox (or similar commercial bleaching agent); Clorox which was found to be 1.5N was diluted to 0.1N
Potassium iodide—15% solution
Sodium thiosulfate—0.1N solution
Starch indicator—1% solution

Procedure

Weigh 0.1-0.2 g. of oil into a 500-ml. glass-stoppered bottle and add 10 ml. of glacial acetic acid. Shake well to dissolve the oil, or disperse it thoroughly in the acid and add 25 ml. of 0.1N Clorox solution. Shake well again, and allow to stand for 10 min. at room temperature. Add 20 ml. of 15% potassium iodide solution, and after 1 min. titrate the liberated iodine in the usual manner, using 0.1N starch as an indicator. Prepare a blank simultaneously, containing no oil.

Calculation

$$\text{Iodine value} = \frac{(\text{ml. blank} - \text{ml. sample}) \times 0.127 \times \text{normality of thiosulfate}}{\text{wt. of sample}}$$

The iodine values of the following oils were determined by Wijs method, by using pure hypochlorous acid and also by using Clorox. The results are shown in Table I. It is seen that the Clorox method agrees very well with the Wijs method, also with the original hypochlorous acid method.

TABLE I
Iodine Numbers of Some Oils by Different Procedures

Oil used	Iodine number		
	Wijs method	Pure HO Cl	Clorox
Cottonseed.....	108.8	108.5	108.7
Soybean.....	129.5	129.2	129.3
Peanut.....	93.2	92.8	93.0
Sesame.....	110.9	111.2	110.9
Corn.....	120.7	120.8	120.5
Linseed.....	180.5	181.0	180.5
Beef tallow.....	42.8	43.1	43.0
Lard.....	65.2	65.1	65.2

B. Fats and Oils Containing Conjugated Unsaturation

Reagents

Clorox solution made to a 0.3N strength
Mercuric acetate, 2.5% in glacial acetic acid
Other reagents as above

Procedure

Weigh 0.1-0.2 g. of oil or fat into a 500-ml. glass-stoppered bottle, and add 10 ml. of a 2.5% solution of mercuric acetate in glacial acetic acid. Shake well to disperse the oil thoroughly. Add 25 ml. of 0.3N Clorox solution, and shake well again. Allow to stand for 1 hr. at approximately 20°C. Add potassium iodide and titrate as above, running a blank simultaneously.

Calculation

As above.

The iodine number of tung oil was determined by the following methods, as shown in Table II. The result by the Clorox method, also by the hypochlorous acid method, agrees fairly well with the catalytic hydrogenation procedure (3) and is comparable with the other accepted methods for the determination of total unsaturation of tung oil. The results are shown in Table II.

TABLE II
Iodine Number of Tung Oil by Various Procedures

Method	Iodine number
Wijs.....	162.0
Woburn.....	239.8
Benham-Klee.....	249.3
Plank-Pack-Goldblatt.....	241.8
Hypochlorous acid.....	242.1
Clorox.....	241.9
Catalytic hydrogenation.....	241.6

TABLE III
Iodine Number of Fatty Acids with Conjugated Double Bonds

Acid	Theoret-ical	HO Cl	Clorox	Hydro-genation method
Beta-eleostearic acid.....	273.8	273.7	273.5	272.3
9:10, 11:12 Linoleic acid.....	181.03	181.3	181.6	181.6

The iodine numbers of β -eleostearic acid and isomerized 9:10, 11:12 linoleic acid were determined by the hydrogenation method, by the hypochlorous acid method, and

by using Clorox. β -Eleostearic acid was prepared by the method of Thomas *et al.* (4), and linoleic acid was isomerized with 7.5% KOH in glycerine by heating in a constant temperature isomerization bath at 180°C. for 1 hr. The isomerized acid was extracted with ether from the acidified reaction product, the ethereal layer was washed and dried, the solvent was evaporated off in a current of nitrogen, and the iodine number was determined by the above-mentioned method. Results are shown in Table III.

The results indicate that the present method of using Clorox for the determination of unsaturation of fats and oils is a very satisfactory one.

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• Fats and Oils

DEHYDRATION OF CASTOR OIL WITH CATALYSTS CONTAINING MORE REACTIVE GROUPS. V. A. Saraf and K. K. Dole (Ferguson Coll., Poona). *Indian J. Appl. Chem.* **22**, 1-5 (1959). The rate of dehydration of castor oil increases with the number of acidic groups in the catalyst, the order of increase being: benzene-, phenol-, and naphthalene-monosulfonic<disulfonic<trisulfonic<heteropoly acids. A lower temperature of dehydration (200-230°) and a reduction of side reactions occurred with increase in reactive groups. The introduction of hydroxyl in 1,3,5-benzenetrisulfonic acid mitigated hydrolysis and decomposition of the oil, while the naphthalene nucleus decreased the activity of the catalyst. Of the three heteropoly acids studied, phosphomolybdic was the least active. (*C. A.* **54**, 1897)

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