TABLE VI

Natural Antioxidant Contents of Refined Oils

	Tocopherol (%)	Oryzanol (%)
Degummed oil	0.761	1.670
Caustic refined oil	0.537	0.977
Solvent refined oil	0.601	1.553
Steam refined oil	0.744	1.532

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⁂Determination of Peroxide Value by Conventional Difference and Difference-Derivative Spectrophotometry

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ABSTRACT

The spectrophotometric behavior of the system iodide-iodine-iodate-oxygen was studied, and a simple iodometric method to determine peroxide value by conventional, difference or difference-derivative spectrophotometry was developed. The procedures are carried out inside standard photometric cells containing iodide solution to which the sample is added. Absorbance or its first derivative with respect to wavelength is measured to determine iodine concentration by means of calibration curves. No special care to avoid iodide oxidation by atmospheric oxygen is necessary.

The method is applicable even to lipids that give emulsions with water, such as phosphatides, and precise results are obtained even at low peroxide values.

The use of a digital spectrophotometer with data processing and programming facilities allowed development of an interactive program for automatic operation, which is outlined.

INTRODUCTION

The autoxidation of lipids is a major cause of their deterioration and hydroperoxides formed by reaction between oxygen and the unsaturated fatty acid moieties are the primary products of this process. The peroxide concentration, usually expressed as peroxide value, gives therefore a measure of the early steps of lipid oxidation (1). The peroxide value (PV) is the concentration of substances, in terms of milli-equivalents of peroxide per 1000 g of sample, that oxidize potassium iodide to iodine.

The numerous methods described in the literature to determine PV were reviewed by Gray (2) together with other procedures to evaluate lipid oxidation.

Iodometric Determination of PV

The most widely used methods to determine PV are those based on the volumetric measurements of the iodine produced from potassium iodide by peroxide at room temperature in acetic acid-chloroform medium (3). According to Mehlenbacher (4), one of the principal sources of error in these methods is the oxidation of iodide by atmospheric oxygen, leading to high results (5,6). Besides variations in the reaction conditions such as temperature and time, it also has been established that other possible sources of error in the titrimetric methods include variation in the weight of sample, the type and grade of solvents used and the nature of the sample (2).

The iodometric AOCS Official Method Cd 8-53 (7), as it states itself, is highly empirical; any variation in procedure may result in variation in results. Besides, it fails for very low PV because of difficulties in the titration end point determination. Furthermore, it is inadequate for samples like phosphatides which give emulsions by shaking, also causing trouble with the end point observation.

Very low PV can be determined by a modification (8) of the titrimetric methods that involves the replacement of the titration step with an electrochemical technique in which the iodine liberated is reduced at a platine electrode maintained at a constant potential, while purified nitrogen is passed for deaeration.

Some colorimetric versions of the iodometric methods have been reported (9-13) in which the liberated iodine is measured either directly by means of the absorbance of the triiodide ion in the UV, or by measurement of the blue color of the starch-iodine complex. It has been suggested (12) that a main factor which has heretofore prevented the wide use of colorimetric methods is the deviation of the absorbance data due to air oxidation of the excess iodide. To avoid this deviation the reaction mixture must be protected from light, and deaeration of solutions by purging with carbon dioxide and exclusion of atmospheric oxygen with the same or other inert gas are necessary. Furthermore, in the procedures of Swoboda and Lea (11) and of Takagi et al. (12), the excess iodide is converted into the more stable cadmium-iodide complex ion. The use of colorimetric cells especially constructed to avoid autoxidation of iodide was proposed (14,15), but they are troublesome and not readily available (12).

The measurement of iodine concentration in the volumetric methods is carried out in an aqueous phase in the presence of an immiscible organic solvent in which the

sample is dissolved, while in the colorimetric methods, in order to allow the absorbance measurement, the aqueous phase must be separated by sedimentation or centrifugation. In a modified volumetric procedure (16), however, a single phase solvent is used.

In order to investigate the possibilities of spectrophotometry to determine PV, a series of experiments was conducted in an attempt to develop a method easier to carry out than those colorimetric and electrochemical methods mentioned above, and capable of giving precise and accurate results even at low PV, regardless of the nature of the sample. The leading idea in this work has been allowing free autoxidation of iodide instead of trying to avoid it.

SPECTROPHOTOMETRY OF THE SYSTEM $I^-/I_2/O_2/IO_3^-$

The effect of peroxides upon iodide was simulated by means of potassium iodate, which oxidizes iodide to iodine through the reaction

$$IO_3^- + 5I^- + 6H^+ \rightarrow 3I_2 + 3H_2O$$

A mixture of acetic acid, chloroform and absolute ethyl alcohol was used as solvent, in which the spectrum of triiodide ion (Fig. 1) presents two bands of absorbance in the UV region with maxima at 292 and 359 nm.

Conventional Spectrophotometry

With a double beam spectrophotometer, the procedure in which the sample solution is placed in the sample cell and the solvent or other blank in the reference cell will be called conventional spectrophotometry (CS).

Provided enough reactants are available in the oxidation of iodide by atmospheric oxygen, the rate of increase in iodine concentration, as well as the rate of increase in absorbance, will be constant: $dC/dt = k$; $dA/dt = k'$. Thus, when the absorbance of an iodide solution undergoing autoxidation is measured at different times, it increases steadily and its plot is a straight line with positive slope (Fig. 2B). In order to observe this phenomenon it is necessary to keep the solution stirred to avoid gradients or local concentrations of iodine which would give a "noisy" or aleatory recording (Fig. 2A). When iodate solution is added, more iodine is liberated and the absorbance suddenly increases (Fig. 3). Once the reaction with iodate is over, a new straight line starts to develop; its slope is higher than that of the first line, probably because of the change in solvent composition due to the small amount of water incorporated with the reagent solution. The difference in absorbance R_A at the time the reagent was added is a measure of the iodine liberated by iodate. If absorbance A_0 (Fig. 3) is measured at time t_0 immediately before the reagent addition, and absorbances A_1 and A_2 are measured at times t_1 and t_2 once the reaction with iodate is over, R_A can be calculated by

$$R_A = A_2 - A_0 - (A_2 - A_1) \cdot (t_2 - t_0)/(t_2 - t_1)$$

By means of aliquots of a standard iodate solution it was found that R_A values keep a linear relation with the iodine concentration.

Difference Spectrophotometry

In difference spectrophotometry (DS) the same solution is placed in both sample and reference cells of a double beam spectrophotometer. Other experimental conditions are controlled in such a way that the difference in absorbance between cells is measured. This difference (induced change)

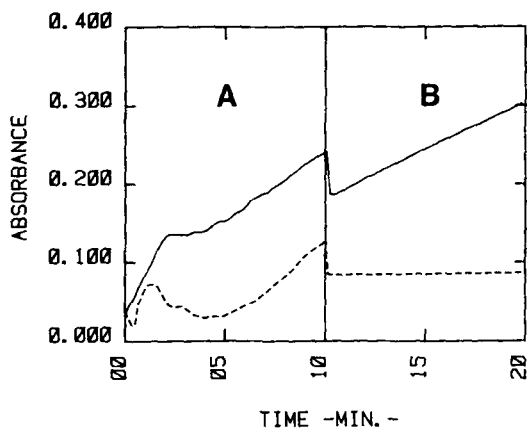

FIG. 2. Absorbance against time in the autoxidation of iodide without (A) and with (B) agitation, by conventional (—) and difference (- - -) spectrophotometry.

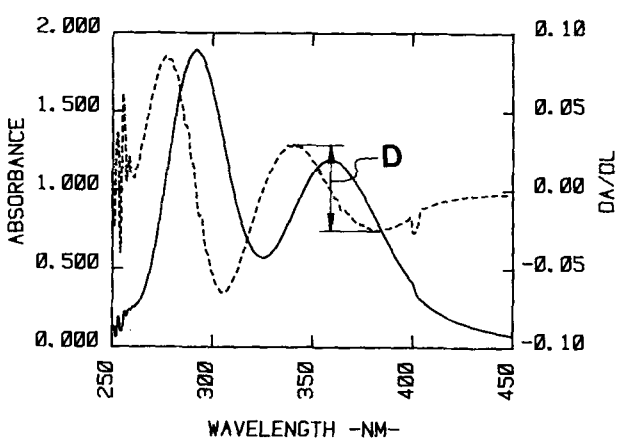

FIG. 1. Spectra of triiodide ion in absorbance (—) and first derivative of absorbance (- - -) in acetic acid-chloroform-absolute ethyl alcohol.

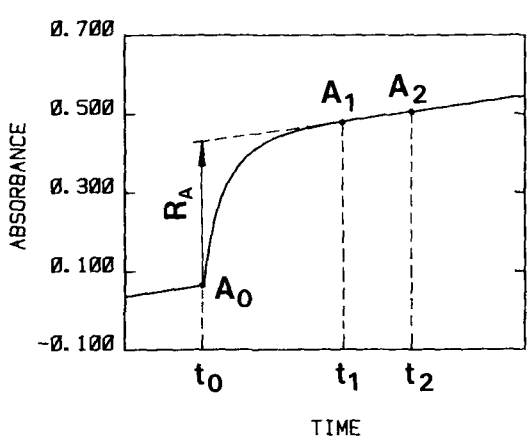

FIG. 3. Absorbance of triiodide ion against time in conventional spectrophotometry showing increase by autoxidation of iodide and by addition of iodate or peroxide.

occurs when a reagent is added to one cell, while unwanted or non-induced changes in absorbance, as they take place simultaneously in both cells, are rejected.

Thus, when iodide solution is placed in both cells and iodate solution is added to one of them, the fraction amount of iodine liberated by iodate produces the induced change in absorbance while the non-induced one, originated by autoxidation, is compensated in the other cell. Recording absorbance against time (Fig. 4), the plot before the addition of iodate is a straight line with zero slope and zero intercept. When iodate is added into one cell, the absorbance suddenly increases and returns to give another straight line once the reaction is over. The difference in absorbance R'_A at the time the reagent was added is a measure of the iodine liberated by iodate. It can be calculated by

$$R'_A = A_2 - (A_2 - A_1) \cdot (t_2 - t_0)/(t_2 - t_1)$$

where t_0 is the time of reagent addition and A_1 and A_2 are absorbances measured at times t_1 and t_2 once the reaction with iodate is over. As in CS it was found that R'_A values keep a linear relation with iodine concentration.

Difference-Derivative Spectrophotometry

The graphic representation of the quotient $dA/d\lambda$ (A = absorbance; λ = wavelength) over the wavelength of interest is called a derivative spectrum, and the technique is referred to as derivative spectrometry (17,18). Theoretical aspects and performance of this method have been reviewed by O'Haver and Green (19,20). If several conditions are fulfilled, a useful characteristic of the first order derivative generated by an absorbance band is that the difference (D in Fig. 1) between the maximum and minimum remains constant even if the absorbance itself is affected by a background absorbance due to an interference or turbidity.

Differential and derivative spectrophotometry (DDS) were combined to render a third method to scan iodide oxidation. For the derivative spectrum of triiodide ion, the difference between the maximum and minimum $dA/d\lambda$ values due to the band at 359 nm (Fig. 1), i.e., $D = (dA/d\lambda)_{340} - (dA/d\lambda)_{382}$ where the subscripts indicate wavelength, will be considered.

When iodide solution is placed in both cells of a double beam spectrophotometer able to measure $dA/d\lambda$, the D value will be zero because of mutual compensation between cells, and will stay unchanged even if iodide is undergoing autoxidation (non-induced change). A sudden increase in D takes place when iodate is added to the sample cell (induced change). The difference R_D (Fig. 4) at the time the reagent was added is a measure of the iodine concentration fraction due to iodate. It can be calculated by

$$R_D = D_2 - (D_2 - D_1) \cdot (t_2 - t_0)/(t_2 - t_1)$$

where D_1 and D_2 are measures of D at times t_1 and t_2 once the reaction with iodate is over, and t_0 is the time of reagent addition. As in CS and DS it was found that R_D values in DDS keep a linear relation with iodine concentration.

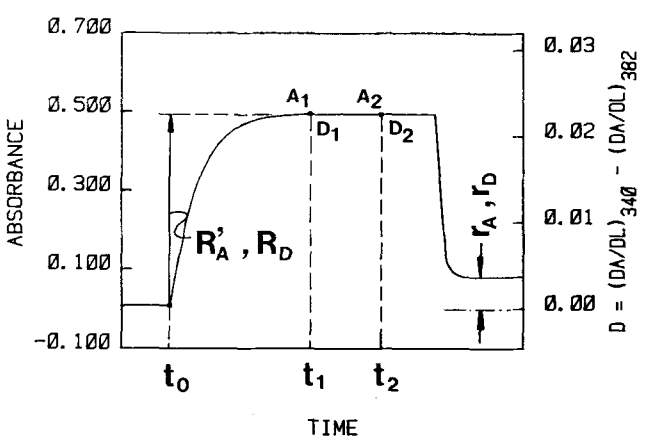

FIG. 4. Absorbance and D values of triiodide ion against time in difference and difference-derivative spectrophotometry showing increase by addition of iodate or peroxide, and correction by addition of thiosulfate.

DETERMINATION OF PEROXIDE VALUE

The model experiments described above have proven to be useful methods to determine PV since lipid peroxides behave, although at a slower rate, like iodate under the outlined experimental conditions. The procedures should include therefore the following steps: iodide solution is placed in the sample cell (CS) or both cells (DS and DDS) of a double beam spectrophotometer and sample solution is added to the sample cell. Absorbances at 359 nm (CS and DS) or $dA/d\lambda$ at 340 and 382 nm (DDS) are measured at known times and R values are calculated. The iodine concentration is obtained from calibration curves and PV is calculated.

Calibration

To obtain calibration curves giving the relation between iodine concentration and R values, known amounts of iodine are generated by aliquots of a standard potassium iodate solution; absorbances or their derivatives are measured and R values calculated. Potassium iodate was chosen because it is a primary standard and gives with iodide no other products than iodine and water. Potassium bromate gives the same results as iodate but potassium dichromate, although it was used as a standard to liberate iodine from iodide (12), was found to give higher absorbances than iodate.

Solvent

The mixture acetic acid, chloroform and absolute ethyl alcohol (4:4:1, v/v/v) was chosen to avoid the need of phase separation. It dissolves most lipids as well as iodine and small amounts of potassium iodide, allowing the determination without removing the cells from the spectrophotometer. Nevertheless, it fails to fully dissolve phosphatides, yielding hazy or turbid solutions, but this is circumvented by employing DDS.

The use of citric acid in a mixture of *tert*-butyl alcohol and carbon tetrachloride (21) was found unsuitable.

Interferences

Samples themselves can give some absorbance at the wavelength of measurement, and this interference depends on the PV of the sample, due to the amount necessary for the determination being larger at lower PV.

Interferential absorbances of samples can be compensated for in CS by adding equal volumes of sample solution to both cells, but this is not possible in DS because the reference cell contains iodide as well. In this case the contribution of the sample absorbance (r_A in Fig. 4) to the total absorbance can be determined by a simple procedure; once the measurements are made, a drop of sodium thiosulfate solution is added to both cells, the iodine is reduced back to iodide and thus, a new measurement of absorbance will correspond to the interference, which is subtracted from the total.

The absorbance of the sample will not interfere in DDS, provided the spectrum shows a gentle slope with no maxima or minima between 340 and 382 nm. If the sample spectrum do not meet these conditions, or to avoid checking them, a correction (r_D in Fig. 4) must be applied as in DS.

Reaction Time

Reaction times from 1 to 60 min are specified in different iodometric methods for PV determination. The two criteria taken into account are fixing a standard time for all samples or allowing enough time for reaction completion. The second criterion is favored in this study because variations in results will be less likely and results will approximate more closely the actual concentration of peroxides, giving more realistic figures related to the oxidative status of the samples.

It was found that the rate of iodide oxidation depends on the concentration of alcohol in the solvent. A high rate was obtained in acetic acid-chloroform with no alcohol, but the autoxidation of iodide in this solvent became uncontrolled for practical measurements in DS and DDS. On the other hand, in the solvent of Dahle and Holman (16), which contains 50% absolute ethyl alcohol, the oxidation of iodide by lipid peroxides is so slow that it takes about one hr for completion. In the solvent chosen, the reaction is over in less than 10 min.

EXPERIMENTAL

All experiments and determinations were carried out at 25 C.

Apparatus

Spectrophotometric measurements of absorbance and $dA/d\lambda$ values were made with a Hewlett-Packard 8450A UV/Vis spectrophotometer equipped with cell holders (HP 89101A Temperature Stations) able to keep constant temperature and to move small magnetic stirrers placed inside the cells. Spectral plotting was made with a Hewlett-Packard 7225B Graphics Plotter, and a Hewlett-Packard 9895A Flexible Disk Memory was added to allow automatic running.

Quartz cells of 1 cm optical path were used.

Solutions

The following solutions were prepared with analytical grade reagents and solvents.

A. Solvent: Glacial acetic acid-chloroform-absolute ethyl alcohol (4:4:1, v/v/v).

B. Potassium iodide: 0.6 g dissolved in 0.5 ml of water.

C. Potassium iodate: About 100 mg accurately weighed, dissolved and made up to 50 ml with water. Two ml of solution made up to 25 ml with water.

D. Sodium thiosulfate: 100 mg dissolved in 10 ml of water.

E. Sample: An amount dissolved in chloroform to give a solution containing approximately 0.004 milli-equilvalents (meq) of peroxides per ml.

CS Procedure

Solution A is pipetted into two cells (3.0 ml each) and solution B (one drop) is added to the sample cell. The cells are placed in the spectrophotometer and agitation is started. At time t_0 the absorbance A_0 at 359 nm is measured and immediately afterward equal volumes (20-80 μl) of solution E are added to both cells from a microsyringe, without removing the cells from the spectrophotometer. Measurements of absorbances A_1 and A_2 at 359 nm are made at 10 and 15 min after t_0 and R_A is calculated by

$$R_A = 3A_1 - 2A_2 - A_0$$

The iodine concentration is obtained from R_A by means of the calibration curve, and PV is calculated by PV = 1000·C/g where C and g are the concentration of iodine in meq/3 ml and the sample weight in the cell in g, respectively.

DS and DDS Procedures

Solution A (about 7 ml) and solution B (one drop) are mixed thoroughly by shaking in a small tube with a glass stopper. This mixture is pipetted into two cells (3.0 ml each), they are placed in the spectrophotometer and agitation is started. Solution E (20-80 μl) is added to the sample cell from a microsyringe, and measurements of absorbances A_1 and A_2 at 359 nm (DS) or D_1 and D_2 [$D = (dA/d\lambda)_{340} - (dA/d\lambda)_{382}$] (DDS) are made at 10 and 15 min. Solution D is added to both cells (one drop each) and absorbance r_A or D value r_D is measured again two min later. R'_A and R_D are calculated by

$$R'_A = 3A_1 - 2A_2 - r_A \quad \text{and} \quad R_D = 3D_1 - 2D_2 - r_D$$

The iodine concentration and PV are calculated as in the CS procedure.

Calibration

The above described CS, DS and DDS procedures are carried out with increasing amounts of solution C (from 20 to 80 μl) instead of solution E, and the relations between iodine concentration and R_A, R'_A and R_D are obtained.

AUTOMATION

The method described is particularly suitable for automation because the whole procedure is carried out inside the photometric cells without removing them from the spectrophotometer. By using an apparatus with data processing and programming facilities, it is possible to program the operational sequence for automatic running.

The interactive program developed contains 78 commands in five subroutines, called methods, by which measurements of absorbance (method 1), data input (method 2), calculations (method 3) and printing a report showing results and warnings (methods 4 and 5) are performed interactively.

Under the program command, the apparatus carries out the following operations:

- Resets default conditions and clears the memory.
- Prompts the operator to place the cells with reagent in the cell holders.
- Starts agitation and sets temperature.
- Performs the balance to compensate for differences in absorbance between cells.
- Prompts the operator to add an aliquot of sample solution to the sample cell.
- Measures and records absorbance spectra (200-800 nm) at 600 and 900 sec after sample addition.
- Prompts the operator to add a drop of sodium thiosulfate solution to both cells.
- Measures and records absorbance spectrum at 120 sec after addition.
- Gives the operator instructions to enter the data of concentration and volume of sample solution.
- Calculates and records: $(dA/d\lambda)_{340} - (dA/d\lambda)_{382}$ values; concentration of iodine by means of a function derived from the least squares method, and peroxide value.
- Prints a report (Fig. 5) with results in which the last spectrum recorded before the addition of thiosulfate is plotted to check for any possible abnormality, and wavelength and absorbance of peaks found are shown. Warnings also are printed to invalidate the determination run if im-

proper measurements (because of too high concentration of sample solution or abnormal spectrum) have been made.

RESULTS AND DISCUSSION

Taking into account the stoichiometry of the reaction iodide-iodate, the calibration data shown in Fig. 6 and the following linear relations, by the least squares method, were obtained:

$$\text{meq } I_2/3 \text{ ml} = 2.22\times10^{-4}\ R'_A - 3.5\times10^{-7}$$

$$\text{meq } I_2/3 \text{ ml} = 4.86\times10^{-3}\ R_D + 4.8\times10^{-7}$$

The correlation coefficients were 0.999 in both cases. Departures from linearity occur at R_A or R'_A higher than 1.4 and R_D higher than 0.065.

The proposed method uses the same principle as the routine procedures, although the titration step is replaced by a spectrophotometric assay. The comparison of CS, DS and DDS procedures with regard to applicability, correction of interferences and other characteristics is summarized in Table I.

As R_A or R'_A values from 0.025 and R_D from 0.001 can be well differentiated from the background, concentrations of iodine from 5×10^{-6} meq/3 ml can be determined which correspond, assuming a maximum of 25 mg of sample in the cell, with PV from 0.2.

The results obtained with samples of several commercial products are given in Table II. While no significant differences were found comparing the data obtained by the CS, DS and DDS procedures, PV of lipid samples were lower when determined by the AOCS method. This disagreement can be explained by considering the reaction time in both methods. It has been shown (8,12) that one min, as in the

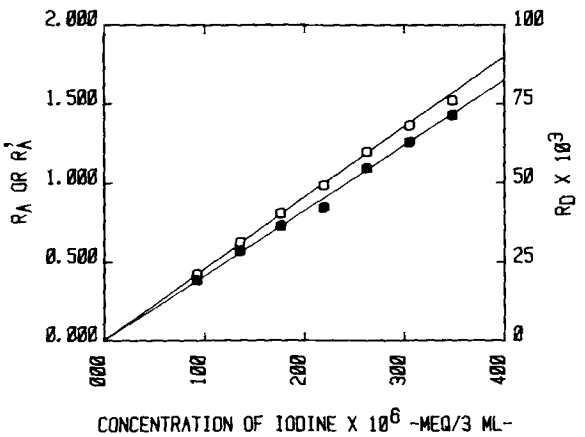

FIG. 6. Calibration curves showing concentration of iodine against R_A or R'_A (○) and R_D (●).

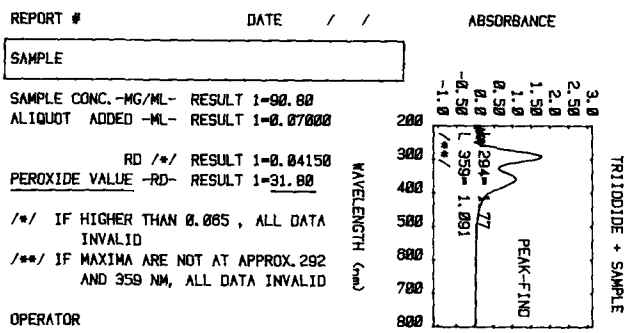

FIG. 5. Facsimile of report issued.

TABLE I

Characteristics of the Spectrophotometric Method to Determine Peroxide Value by the Different New Procedures

	Spectrophotometric procedure		
	Conventional	Difference	Difference-derivative
Measurement of	Absorbance	Absorbance	Derivative of absorbance
Content of reference cell	Solvent plus sample	Iodide sol.	Iodide sol.
Correction of interferences	Not required	With $Na_2S_2O_3$	With $Na_2S_2O_3$
If sample gives hazy solution	Not applic.	Not applic.	Applicable

TABLE II

Peroxide Values[a] Obtained with Different Methods

Sample	Method			
	Spectrophotometric			Titrimetric (AOCS)
	CS	DS	DDS	
Soybean oil	4.7 ± 0.2	4.7 ± 0.04	4.8 ± 0.2	4.5 ± 0.25
Soybean lecithin			3.1 ± 0.07	1.2 ± 0.1
Sunflower oil	18.2 ± 0.1	17.8 ± 0.3	18.0 ± 0.25	14.2 ± 0.08
Castor oil		63 ± 1	65 ± 2	50.1 ± 0.13
Autoxidized soybean oil	106 ± 3	106 ± 3	105 ± 2	77.9 ± 0.2
n-Hexane[b]		0.63 ± 0.03	0.64 ± 0.02	
n-Hexane[c]			33 ± 1	25.6 ± 0.2
Dibenzoyl peroxide[d]		8357 ± 99	8259 ± 123	8461 ± 22

[a]Average of triplicate analyses. Results expressed as PV ± standard deviation.

[b]Spectroscopic grade stored in a dark bottle.

[c]Extraction grade stored in a clear bottle.

[d]Expected PV 8257.

AOCS method, is not enough to reach reaction completion between lipid peroxides and iodide. The Japan Oil Chemists' Society method (22), in the same medium and taking precautions to avoid iodide autoxidation, gives, after five min reaction, higher results than the AOCS method (12).

Dibenzoyl peroxide, as diacyl peroxides in general, reacts with iodide at a higher rate than hydroperoxides present in lipids (15). When the PV of a dibenzoyl peroxide sample was determined, excellent agreement between results by the proposed and AOCS methods was obtained, with recoveries of 101% (DS), 100% (DDS) and 102% (AOCS). This indicates that no determinate excess errors occur in the proposed method, and the AOCS method behaves adequately when the peroxide reacts with iodide quickly enough to allow reaction completion in one min or less.

The data obtained on standard deviation confirm a previous finding (12) showing that titrimetric methods for PV determination are more precise than spectrophotometric ones. The reproducibility in this case was acceptable, with coefficients of variation lower than 5%.

An advantage of the developed method is that no trouble occurs with samples of low PV and those that give emulsions with water, such as lecithin. Deaeration of solutions and protection from atmospheric oxygen, use of special cells and complexation of the excess iodide are not required as in other spectrophotometric methods. Another major advantage is the possibility of automation. Although several commercial devices able to perform automatic or robotic analysis are available, no special piece of equipment was required in this case.

Running time of the procedure in the automatic mode is about 25 min, most of which is spent waiting for reaction completions and printing instructions and report. The demand for operator attention is no longer than in routine methods.

The list of commands, available from the author, can be adapted easily by translation to the language of any apparatus similar to the one used.

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