A Distillation Method for the Quantitative Determination of Malonaldehyde in Rancid Foods[1]

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OBJECTIVE TESTS for following organoleptic deteriorations in food products are highly desirable. One such test is the reaction of 2-thiobarbituric acid (TBA) with the oxidation products of unsaturated fatty acids to give a red pigment. The spectrophotometric determination of this red pigment has been used to follow rancidity in a wide variety of food products (1, 2, 4–9, 11, 13–16, 18–20, 22, 24, 25, 28). Several of these references (6, 9, 11, 12, 14, 22, 23, 27) review the earlier literature of this reaction.

The chemistry of the reaction producing the red pigment has been at least partially elucidated. A three-carbon fragment derived from the oxidation of mono-or-polyenoic fatty acids was early postulated as the active color-producing compound (10, 13, 17, 27). Patton *et al.* (13) tentatively identified this fragment as malonaldehyde on the basis of spectral curves. Sinnhuber *et al.* (22, 23, 29) prepared the pure pigment both from malonaldehyde and from rancid oil. On the basis of elemental analyses, absorption spectrophotometry, and paper chromatography they concluded that pigments from the two sources were identical and suggested a structure in which two molecules of TBA condense with one of malonaldehyde.

Various procedures have been employed for performing the TBA test on food products. It is usually performed on the whole food rather than on extracted fat. It would therefore be expected to measure oxidation products of protein bound lipids and phospholipids which would not be extracted by ordinary fat solvents. All test procedures involve heating the food with a strong acid. This step appears to be essential for the liberation of malonaldehyde from some precursor (17, 20, 22, 25) as well as for the condensation of malonaldehyde with TBA.

The TBA may be added with the acid directly to the food and the whole mixture heated for periods of 30 to 50 min. to obtain maximum color development. The red pigment formed during the heating is then extracted with a suitable solvent. For example, Turner *et al.* (25) reacted pork tissue with TBA in the presence of phosphoric and trichloro-acetic acids and extracted the color with an iso-amyl alcohol-pyridine mixture. Yu and Sinnhuber (22, 29) heated fishery products with TBA in the presence of hydrochloric and trichloro-acetic acids and pyridine and used petroleum ether to extract the chromogen.

Sidwell *et al.* (20) described a steam-distillation procedure for dried milk in which the malonaldehyde was distilled from the acidified milk. A fraction of the distillate was then reacted with TBA, and the color was read directly. The distillation procedure offers several advantages over other methods. The malonaldehyde is obtained in a clear aqueous solution so that its reaction product with thiobarbituric acid does not need to be extracted with solvents. The acid heat treatment necessary to effect the liberation and distillation of malonaldehyde from the sample is less drastic than that required for maximum color development with the TBA reagent, therefore there is less likelihood of fat oxidation occurring during the test itself. Also the relation of the rancid odor to thiobarbituric acid-reactive material and to other volatile compounds can be more readily studied in the clear distillates.

Most workers reported values for the TBA reaction in arbitrary absorbance units, which in view of the diversity and empirical nature of the methods employed cannot be compared from one laboratory to another. Sinnhuber and Yu (22) proposed the use of 1,1,3,3-tetra-ethoxypropane as a standard. Acid hydrolysis of this acetal yields malonaldehyde. It was therefore possible to express their results in terms of the "TBA number," defined as mg. of malonaldehyde per 1,000 g. of sample.

This value would be expected to have real significance as a comparable measure of fat oxidation in the various modifications of the TBA method applied to different foods only if a) significant oxidation of fatty components of the foods does not occur during the test itself and b) all of the malonaldehyde is extracted from the tissue by the test procedure.

It seems unlikely that either of these conditions is met. With the Turner method, significant readings are obtained even with fresh raw meats (24, 25) and with oysters (18), in which prior fat oxidation would not be expected. The fact that the thiobarbituric acid reaction has been used to distinguish fresh horse meat from other meats, based on the greater content of linolenic acid in the former (7), is presumptive evidence that considerable oxidation is brought about by the test itself. It follows that the test can be useful as a measure of prior fat oxidation of rancidity only when applied to material for which the "fresh" base line has been established.

It is equally clear that, for the Turner method at least, complete extraction of malonaldehyde is not obtained in the test procedure. The meat sample is ground to a specified size rather than homogenized in a blender in order to prevent the development of too much color for convenient reading in the spectrophotometer.

The research reported in this paper describes the application of the distillation procedure to meat and compares this method with Turner's extraction method. The distillation technique has been simplified somewhat by direct heating of meat slurries in Kjeldahl distillation racks rather than by passage of live steam through the samples, as suggested by Sidwell *et al.* (20). This allows the simultaneous distil-

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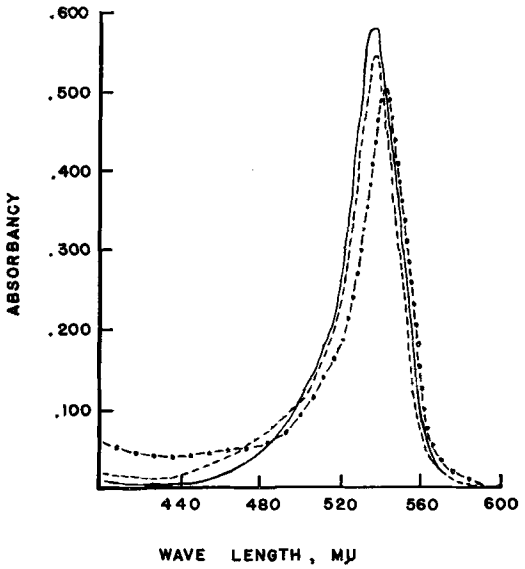

FIG. 1. The absorption of the colored complexes produced with the TBA reagent and: TEP standard solution by the distillation method (———); TEP standard solution by Turner's method (—·—·); cooked meat by the distillation method (– – – –).

lation of multiple samples with equipment generally available in food laboratories. Malonaldehyde standards have been used with both methods, as suggested by Sinnhuber and Yu (22).

Experimental

Reagents

TBA Reagent. 0.02 M 2-thiobarbituric acid in 90% glacial acetic acid. Bring into solution by warming slightly in a boiling water bath.

TEP Standard. 1×10^{-3} M 1,1,3,3,-tetra-ethoxypropane in distilled water. This solution can be kept for about a week if stored in the refrigerator and diluted as needed.

HCl Solution. 1 part of concentrated HCl to 2 parts of distilled water (approximately 4 N).

Procedure

Blend 10 g. of meat with 50 ml. of distilled water in a Waring Blendor for 2 min. Transfer the mixture quantitatively into a Kjeldahl flask by washing with an additional 47.5 ml. of distilled water. Add 2.5 ml. of HCl solution to bring the pH to 1.5. Place a small amount of Dow antifoam A onto the lower neck of the flask, and add a few saddle stones to prevent bumping. Assemble apparatus and heat flasks at the highest heat obtainable on the Kjeldahl distillation apparatus. With the electric heating elements available, in this laboratory approximately 10 min. from the moment boiling begins are required to collect 50 ml. of the distillate.

Mix the distillate, pipette 5 ml. into a 50-ml. glass-stoppered tube, and add 5 ml. of TBA reagent. Stopper the tubes, mix the contents, and immerse in a boiling water bath for 35 min. A distilled water-TBA reagent blank should be prepared and treated like the samples.

After heating, cool in tap water for 10 min., transfer a portion to a cuvette, and read the optical density of the sample against the blank at a wavelength of 538 mμ. (A Beckman DU spectrophotometer was used). Multiply the reading by the factor 7.8 to convert to mg. of malonaldehyde per 1,000 g. of meat.

The details of the distillation procedure given above differ in several respects from the recommendations of Sidwell *et al.* (20) for milk powder. The experimental justification for the recommendations given will be clear from the observations in the following sections.

Results

Absorption Curves. Absorption spectra of the color produced with TBA and the hydrolysis product of 1,1,3,3,-tetra-ethoxypropane (malonaldehyde), using Turner's method and the method described above, are plotted in Figure 1. In addition, a spectrum of the chromogen produced by reacting the TBA reagent with a distillate from rancid cooked meat is included. The concentration of malonaldehyde used for Turner's method was 5×10^{-8} moles and that for the distillation method 4×10^{-8} moles. The shapes of the absorption curves are similar to those shown by Sinnhuber and Yu (22). The position of maxima differed slightly in the two methods, probably because of differences in the solvent and pH.

Sinnhuber and Yu (22) reported a maximum at 535 mμ. Maxima of 543 and 538 were found for Turner's method and the distillation procedure, respectively.

Malonaldehyde Standard Curves. A standard curve was prepared by making appropriate dilutions of the 1×10^{-3} M TEP standard solution to give amounts ranging from 1×10^{-8} to 7×10^{-8} moles of malonaldehyde in 5 ml. Determinations were run directly on 5-ml. portions both by Turner's method and the method described for testing the distillate. The results are plotted in Figure 2. It is evident that the pigment follows Beer's law over the entire concentration range when measured on the Beckman DU Spectrophotometer. However deviations from a linear relationship between absorbancy and concentration were obtained when the Evelyn colorimeter with a 540 mμ filter was used in place of the spectrophotometer, in both methods. All results reported in this paper were obtained with the spectrophotometer.

Since these standard curves were obtained by reacting the solutions directly with the TBA reagent, they do not, of course, give any measure of the recovery of malonaldehyde upon distillation. Data on this point are shown in the following section.

Factors Affecting Distillation of Malonaldehyde

1. *pH of the Material to Be Distilled.* As found by Sidwell *et al.* (20) for milk, acidification was necessary to release malonaldehyde either from TEP standards or meat. Table I shows the optical densities

TABLE I

Effect on Optical Densities of the pH of the Material to Be Distilled[a]

pH	Optical density	
	TEP solution	Cooked meat slurry
2.00	.391	.....
1.90	.412	.....
1.75	.461	.....
1.70	.449	.542
1.60	.463	.....
1.50	.462	.600
1.30	.426	.....
1.10	.428	.....
.90	.422	.560
.70	.415	.....
.50	.405	.455

[a] Distillation time: 10 min.

obtained on distilling at various pH values. The distillation flask contained 5×10^{-7} moles of 1,1,3,3,-tetra-ethoxypropane, standard or 10 g. of cooked meat. In each case 50 ml. of distillate were collected, and 5 ml. were used for the TBA test. Maximum optical density, representing a recovery of 69%, was obtained at pH 1.5 both with TEP standard and with the meat samples. The 2.5 ml. of HCl solution produced a pH within the range of 1.5–1.7 in meat. In TEP standard solutions 2.0 ml. were required.

2. *Effect of Time of Heating During Distillation.* Time required to obtain 50 ml. of distillate was varied by heating samples with same pH, using the three heat speeds and several positions available on the Kjeldahl apparatus. The distillation flask contained 5×10^{-7} moles of TEP or 10 g. of cooked meat. Results are shown in Table II.

TABLE II

Effect on Optical Densities of Varying the Time Necessary to Collect 50 ml. of Distillate[a]

Time of heating (min.)	Optical density	
	TEP solution	Cooked meat slurry
10	.490	.580
11	.480	.610
12	.465	.650
14	.451	.670
18	.438	.755
20	.415	.780

[a] pH: 1.5.

The greatest amount of malonaldehyde was obtained in 50 ml. of distillate when the distillate was collected in the shortest time possible, *i.e.*, the highest heat available. The heating period was 10 min. When however 10 g. of cooked meat slurry were distilled in the same way, color development increased with the time of heating.

It is thought that fat oxidation may occur during the test itself, especially if the heating period is prolonged and the pH is very low. To test this hypothesis, a sample of fresh raw pork was assayed both by Turner's method (which requires heating the tissue for 30 min. at a pH of 0.5) and also by several variations of the distillation procedure. The results given in Table III demonstrate the production of malonaldehyde in nonrancid material by prolonged heating at low pH. Rapid distillation at the recommended pH of 1.5 produced no malonaldehyde from fresh raw meat.

3. *Effect of Varying the Amount of Distillate.* A preliminary experiment in which an acidified meat sample was distilled almost to dryness and the distillate collected in three equal fractions demonstrated the presence of malonaldehyde in all three fractions. Since it was not possible to extract all malonaldehyde from meat by partial distillation and since prolonged heating may cause further oxidation, the following experiment was devised to determine the point at which the distillation should be stopped.

TABLE III

Production of Malonaldehyde in Test Procedures Used with Fresh Raw Pork

Method	pH	Time of heating (min.)	Optical density
Turner's ground meat	.5	30	.201
Turner's meat slurry	.5	30	.387
Distillation	1.5	10	.000
Distillation	1.5	20	.014
Distillation	.5	10	.025
Distillation	.5	20	.048

TABLE IV

Effect on Optical Densities of Varying the Amount of Distillate[a, b]

Ml. of distillate collected	Optical density	
	TEP solution	Cooked meat slurry
40	.450	.540
50	.460	.578
60	.370	.480

[a] Distillation time: 10 min.
[b] pH: 1.5.

A solution containing 5×10^{-7} moles TEP or 10 g. of cooked meat slurry were distilled, and samples were collected at various intervals. The maximum optical density was obtained when 50 ml. of distillate were collected, as shown in Table IV. Results are expressed on the same volume basis.

Observations on the Distillate

1. *Odor.* The distillate from TEP had a characteristic odor, somewhat suggestive of apples. The distillates from rancid meat had a stronger, more unpleasant odor, similar to that of the meat itself. The malonaldehyde appears to contribute a relatively small fraction of the total odor complex of rancid meats.

2. *Separation of Oily Layer.* It was observed that some oily spots appeared at the surface of the distillates from both TEP and meat samples when the distillates were allowed to stand a few hours. To determine whether this represented a separation of some polymer of malonaldehyde which might interfere with proper sampling for the TBA test, the TBA reaction was performed on 5 ml. of distillate from the surface layer, 5 ml. from the bottom, and 5 ml. from a duplicate distillate just after mixing and shaking. The original solution contained 5×10^{-7} moles of TEP. The values agreed within the experimental error of the method (Table V). The same test run on a cooked meat distillate gave similar results.

TABLE V

Recovery of Reactive Material in Various Portions of the Distillate

Portion used	Optical density	
	TEP solution	Cooked meat slurry
Top layer	.440	.567
Bottom layer	.445	.575
Mixed distillate	.460	.582

3. *Effect of Heating Time on Color Development with the TBA Reagent.* The distillates from 5×10^{-7} moles of TEP and from cooked meat were heated with the TBA reagent in a boiling water bath for times varying from 25 to 55 min. The results are shown in Table VI. Although color formation was still increasing slowly at the end of 55 min., it was decided to use a 35-min. heating period routinely since the rate falls off after that time.

4. *Stability of the Distillate and of the Colored Complex.* Distillates collected one day can be held over 24 hrs. at room temperature with very little change in subsequent color development with the TBA reagent. After the color complex is formed, optical density increases about 10% if the solution is held for 24 hrs.

5. *Recovery of Malonaldehyde in the Distillate.* The average recovery of malonaldehyde obtained in 50 ml. of distillate from known concentrations of TEP was 68%. The usual range was from 66 to 70%.

Expression of Results. Sinnhuber and Yu (22) proposed the use of the term "TBA number" as the mg. of malonaldehyde per 1,000 g. of sample. In the distillation method the "TBA number" may be calculated by multiplying the absorbancy by a constant K, the value of which may be obtained from the standard curves and the known dilutions as follows:

$$K\ (\text{distillation}) = \frac{\text{conc. in moles/5 ml. of distillate}}{\text{optical density}} \times \text{mol. wt. of malonaldehyde} \times \frac{10^7}{\text{wt. of sample}} \times \frac{100}{\%\ \text{recovery}}$$

The value of the first term from the standard curve is 7.4×10^{-8}. With a sample of 10 g. and 68% recovery: K (distillation) = 7.8.

The constant for the method of Turner *et al.* (25) has a numerical value of 1.4. The conversion of optical densities obtained by this method to mg. of malonaldehyde is however rather meaningless in view of the oxidation of the sample during the test procedure and the incomplete extraction of malonaldehyde.

Correlation of Sensory Scores with mg. of Malonaldehyde per 1,000 g. of Meat. Fresh pork ham was purchased locally, ground in a meat grinder, and thoroughly mixed by hand. The meat was divided in two lots. One served as a control, and the other was treated with a mixture of 0.5% sodium tripolyphosphate and .22% sodium ascorbate as antioxidants (24). It was packed in 307 × 113 cans, sealed, and cooked in a water bath to an internal temperature of 70°C. Some of the cans were kept in the refrigerator and some in the freezer.

TABLE VI

Effects of Heating Time on Color Development of the TBA-Malonaldehyde Chromogen [a]

Time of heating (min.)	Optical density	
	TEP solution	Cooked meat slurry
25	.367	.890
35	.462	1.050
45	.470	1.120
55	.495	1.150

[a] pH: 1.5.

At intervals cans were opened, the meat was ground again and presented to a panel of six to eight trained judges for odor evaluation. The judges rated the intensity of rancid odor on a six-point scale ranging from not detectable to very strong. Values from 1 to 6 were computed for each sample. The TBA number (using the distillation method described above) was performed on duplicate samples. The sensory values were compared with the average TBA numbers, using the Spearman Rank Correlation Coefficient rs (21).

The results, representing a total of 147 judgments on 21 different samples of the stored meat, gave a correlation coefficient (rs) of 0.89, which is highly significant. The threshold range of TBA numbers for detection of off-odor in pork was approximately 0.5 to 1.0.

Discussion

Although thiobarbituric acid methods for the determination of malonaldehyde in rancid foods are less precise than peroxide determinations on rancid fats,

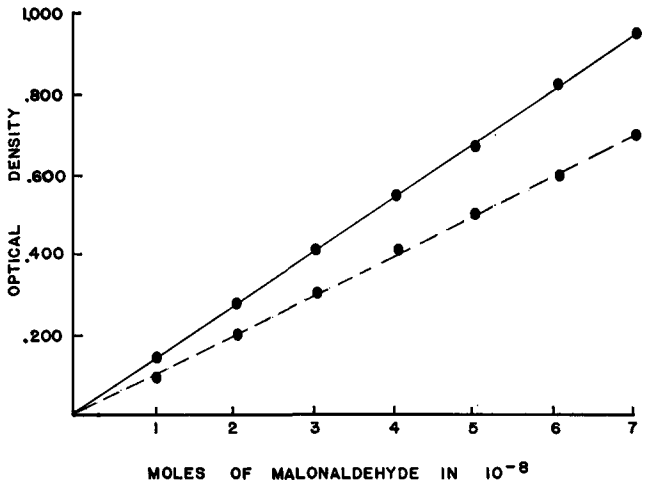

FIG. 2. Malonaldehyde standard curves: Distillation method (————); Turner's method (– – – – –).

it is nevertheless important that these methods be explored thoroughly. They offer at present the only available measure of the oxidation of tissue lipids not extracted with ordinary fat-solvents. This fraction is largely composed of phospholipids and protein bound lipids characterized by a high degree of unsaturation. The oxidation of this fraction is undoubtedly responsible for types of odor and flavor deterioration in meat which have not previously been characterized chemically or measured objectively (26).

The distillation procedure described is superior to other current methods of carrying out the thiobarbituric acid reaction in that there may be less oxidation of fatty acids during the test itself. At the present time identical "TBA numbers" obtained by various methods do not represent equal degrees of rancidity. Nevertheless the expression of results in terms of TEP standards (malonaldehyde) will at least allow comparison of the results obtained with the same material, using different methods and should facilitate the improvement and standardization of procedures.

It is realized that 2-thiobarbituric acid also reacts with other compounds which may be present in distillates from rancid foods. Glyoxal has been shown to form a colored complex absorbing at 525 and 550 mμ (3). This may be present in irradiated meats, but there is no evidence that it is a product of the oxidation of unsaturated fatty acids. It does not interfere with the malonaldehyde-TBA readings at approximately 540 mμ.

The distillate from meats may also contain a compound which reacts with TBA to give a peak at 450 mμ. This peak is not observed with pure malonaldehyde solutions. The compound responsible has not been identified.

It should also be pointed out that there is no necessary relationship between the thiobarbituric acid test and the total carbonyls as measured by 2,4-dinitrophenylhydrazine. Malonaldehyde is, of course, a dicarbonyl. It reacts with 2,4-dinitrophenylhydrazine to give a peak at 380 mμ (26). However, in the concentrations present in the distillates from meats, malonaldehyde does not contribute a significant fraction of the value for total carbonyls.

The relationship between malonaldehyde and rancid odor needs further investigation. Comparison of the odor of distillates from TEP standards *versus* rancid meats indicates that malonaldehyde contributes only

a small part of the total odor complex. Nevertheless it appears to accompany the odor very closely. The relationship between malonaldehyde, other carbonyls, and odor, in distillates from pure fatty acids, is under investigation at the present time in an effort to throw light on these questions.

Summary

An improved distillation method is described for the quantitative determination of malonaldehyde in foods containing oxidized fats. The procedure is compared with other methods in current use for the determination of malonaldehyde. A high correlation of TBA numbers with rancid odor in cooked meats was established.

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Search for New Industrial Oils. II. Oils with High Iodine Values

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AMONG THE FIRST 87 samples of seed oils analyzed in a program to find new oils of industrial importance (1), 12 were found to have iodine values above 180.

Only three plant families, the *Cruciferae, Euphorbiaceae,* and *Labiatae,* are represented, and all three have previously been known (2, 5) to contain members producing oils with high iodine values. Eight of the 12 species however have not had oil composition reported. Failure of other plant families to appear in this tabulation of oils with iodine values above 180 may result partly from the limited number of samples thus far analyzed. It may be expected that additional oils with high iodine values will be discovered and that other plant families will be represented as additional seed materials are examined.

The *Cruciferae* (mustard family) include some 300 genera and 3,000 species (3). The rapeseed and mustard seed oils from this family are familiar items of commerce. Oil composition has been reported in the literature for some 30 species of *Cruciferae,* and the presence of erucic acid is characteristic of the family. The two representatives of the family in this report belong to *Hesperis* and *Matthiola,* genera which contain some 25 and 50 species, respectively. The only previously analyzed oil from these genera was from *H. matronalis.* There are almost 75 other species which may be explored with the expectation of finding some with improved oil composition, increased seed yield, more desirable plant form, and wider climatic adaptability.

The *Euphorbiaceae* (spurge family) are a large family of some 280 genera and 8,000 species. The plant types are quite varied, ranging from prostrate herbaceous weeds to cactus-like trees. The best known commercial oils from this group are tung and castor oils, which have special value because they contain structures not present in the more common oils. Only about 60 species from the entire family and about 15 of the 1,000 species in the genus *Euphorbia* have been analyzed for oil composition.

The *Labiatae* (mint family) include some 3,000 species, of which about 60 are grown in gardens in this country as ornamentals or as kitchen herbs. Perilla is the principal representative of the family among industrial seed oils, but published analyses for some 15 other species (2, 5) indicate that several should produce oils of similar drying quality.

Materials and Methods

The source of seeds, the method of preparing oils, and the methods of analysis have been described previously (1). Methods presented by Gardner (4) were used to determine film hardness and drying time. Films of oil modified by the addition of 0.015 g. of mixed drier (24% lead, 6% cobalt, and 6% manganese naphthenates) were put on microscopic slides. Those for the drying-time test were touched repeatedly with the finger to determine when the film had set to touch. The films for the hardness test were aged two days, then tested with a series of drawing pencils of graduated hardness to determine the softest one which would scratch the film. Viscosities were determined by using the Gardner-Holdt Bubble Viscometer.

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