

# Comparison of Oxygen Bomb Method to Other Methods for Measuring Oxidative Stability of Peanuts and Peanut Products

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## ABSTRACT

The active oxygen method of the American Oil Chemists' Society has been used extensively to evaluate the oxidative stability of fats and oils. The AOM lacks versatility, however, in that it can be used for only a few products such as lard and vegetable oils. Experience in our laboratory has shown that results also can differ widely, even on the same sample. Recent work with the oxygen bomb at the National Peanut Research Laboratory has shown that it is both reliable and accurate when compared to other methods for measuring the oxidative stability of peanuts and peanut products. Results with the oxygen bomb were compared to the active oxygen method and iodine value of the peanuts.

## INTRODUCTION

Predicting the potential oxidative stability in oils and fatty products has been the object of many experiments and papers over the past 40 years (1-8). The active oxygen method (AOM) of the American Oil Chemists' Society (9) has been used extensively to evaluate the stability of fats and oils, but the AOM lacks versatility in that it can only be used on a few products such as lard and oils.

One of the earliest tests developed to determine the keeping quality of products was the Schaal oven test (10). This method was originated by the biscuit and cracker industry for measuring the relative stabilities of the various shortenings purchased for use in their products. Joyner and McIntyre (10) later adapted the Schaal test for use by the shortening industry and, because of simplicity and minimum equipment requirement, it is still used today. In recent years, however, the oxygen bomb method (OBM) has evolved as one of the quickest and most precise

methods available for predicting the oxidative stability of fatty materials. Gearhart et al. (1) were one of the first groups to adapt the ASTM oxygen bomb method, used by the petroleum industry for determining the stability of gasolines to gum formation, to measuring the oxidative stability of fatty materials. Stuckey et al. (8) proposed an improved OBM, which was based on using a dispersant to give the sample a larger surface area, thereby improving the oxygen absorption of the sample. This procedure decreased time for a test and improved the end point, but difficulty was encountered with the dispersant methods. Pohle et al. (6) compared the OBM to the AOM and an oxygen absorption method. He found that the OBM and the absorption method were more precise than the AOM. Pohle et al. (11) also developed a method based on the OBM, in which a copper catalyst was used to shorten the oxidation time on highly stable products. Bennett and Byer (12) compared the OBM to the AOM and found the OBM to be twice as precise.

This study describes a rapid and precise procedure, based on the oxygen bomb technique, which was compared with the AOM and the iodine value for predicting the oxidative stability for peanuts and peanut products.

## EXPERIMENTAL PROCEDURES

### Materials and Equipment

Oxygen bomb apparatus: Precision Scientific Co., type used in test for measuring tendency of gasoline to form gum in storage, in accordance with ASTM D525.

Pressure recorder: two-pin, 12 in., incorporating a 12 in. circular chart with range of 0 to 200 psi in subdivisions of 2 lb with separate sensing systems for simultaneous recording of two bomb pressures, and having threaded male connectors 1/8 in. N.P.T. for attachment to bomb by flexible tubing.

Glass liner and cover: Pyrex glass for fitting inside bomb to hold sample.

TABLE I

Comparisons of Precision and Sensitivity between AOM, OBM and among Sample Types for OBM<sup>a</sup>

Type of analysis	AOM		OBM		
	Peanut oil	Peanut oil	Peanut butter	Raw blanched peanuts	Roasted blanched peanuts
Standard deviation of differences between sample duplicates	9.10	6.86	6.06	4.39	4.24
Coefficient of variability, %	43.72	5.44	3.76	3.95	4.75
Sample mean	20.81	126.10	161.25	111.00	89.38
Two way analysis of variance <sup>b</sup>					
Variance among 8 samples - <i>F</i> value	0.91	14.57 <sup>c</sup>	61.04 <sup>d</sup>	90.31 <sup>d</sup>	76.84 <sup>d</sup>
Variance between 2 replications - <i>F</i> value	0.37	1.05	0.80	0.02	3.61

<sup>a</sup>AOM, active oxygen method; OBM, oxygen bomb method.

<sup>b</sup>Degrees of freedom for samples = 7, for replications = 1, for error = 7.

<sup>c</sup>Significant at the 1.0% level.

<sup>d</sup>Significant at the 0.1% level.

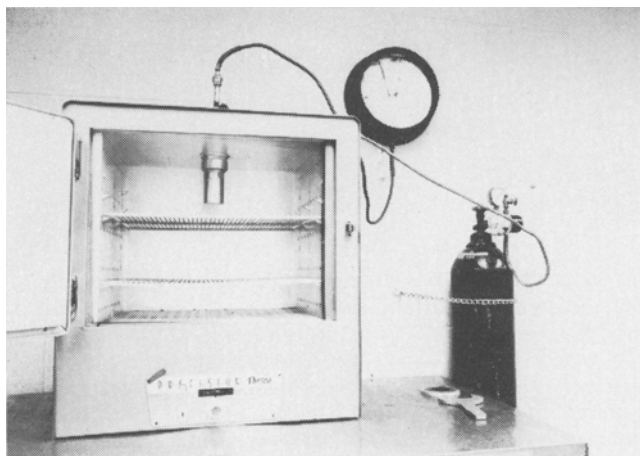


FIG. 1. Oxygen bomb equipment showing oxygen bomb suspended in oven.

Flexible seamless tubing: two 5 ft lengths with protective armor braid. Each end of one tubing has a 1/8 in. male coupling to connect the oxygen tank to the bomb. The other tubing has a 1/8 in. male coupling one end and a 1/8 in. female coupling on the other to connect the bomb to the recorder. The flexible seamless tubing is superior to rigid tubing with regard to leaks.

Forced draft oven: Precision Thelco Model 28. The opening in top of oven was enlarged to accommodate oxygen bomb.

Wrench: for use in tightening the top of the oxygen bomb.

Table socket: accommodates oxygen bomb gripping its octagonal portion.

Oxygen: tank equipped with regulator and pressure gauges.

### Method

A 50 g sample (peanuts, peanut butter or peanut oil) is weighed into the glass liner and covered with a glass lid. If the sample is of raw or roasted kernels, it should be blanched before testing. The liner containing sample is placed inside oxygen bomb and the top screwed on tightly with wrench while bomb is resting in table socket. Bomb is purged twice with oxygen to displace air and then pressurized to 110 psi with oxygen. Bomb and fittings are checked for leaks by checking all joints and connections with a soap solution, and placed in oven heated to  $135\text{ C} \pm 2$ . Recorder pen is lowered onto recorder chart and pressure monitored until a 2 lb drop in pressure is observed. This is the point at which the sample begins to absorb oxygen. Time required for this drop in pressure is related to the oxidative stability of the sample. Figure 1 shows the oxygen bomb and accessory equipment; the oxygen bomb is shown suspended from the oven. Figure 2 shows a typical pressure vs. time chart of raw peanuts.

### RESULTS AND DISCUSSION

Series of eight peanut oil, peanut butter, blanched raw, and blanched roasted peanut samples were tested with the OBM, and the results compared with the AOM tests made on the oil cold-pressed from an aliquot of the raw peanuts from the same eight samples. The peanuts were of known history and had widely varying genetic differences. Samples were stored at 5 C for 3 months before testing.

The peanuts were roasted uniformly in a cylindrical wire basket placed inside a home-type rotisserie. The basket was made of stainless steel and had four compartments.

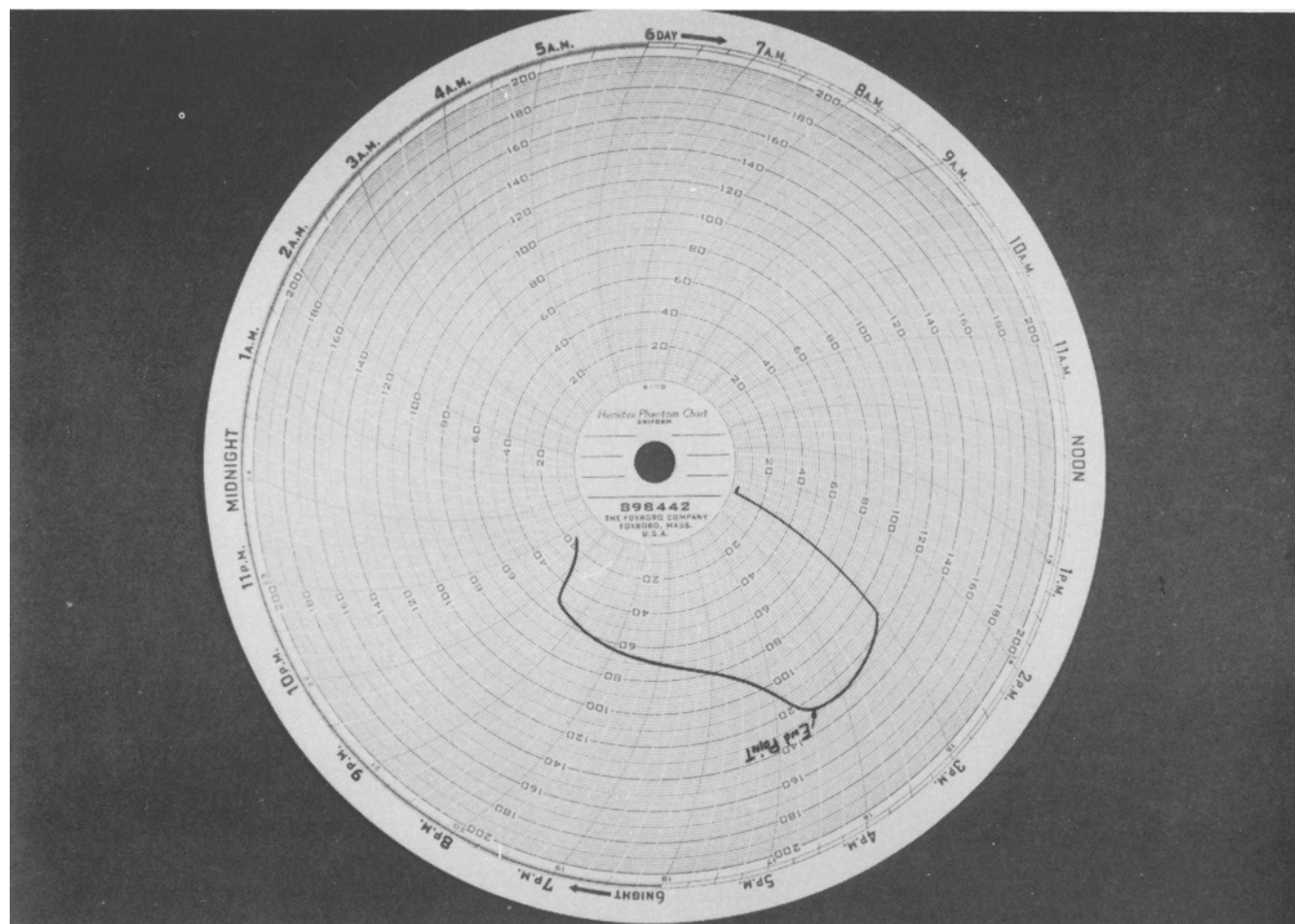


FIG. 2. A typical time vs. pressure chart of raw peanuts showing end point as sample begins absorbing oxygen.

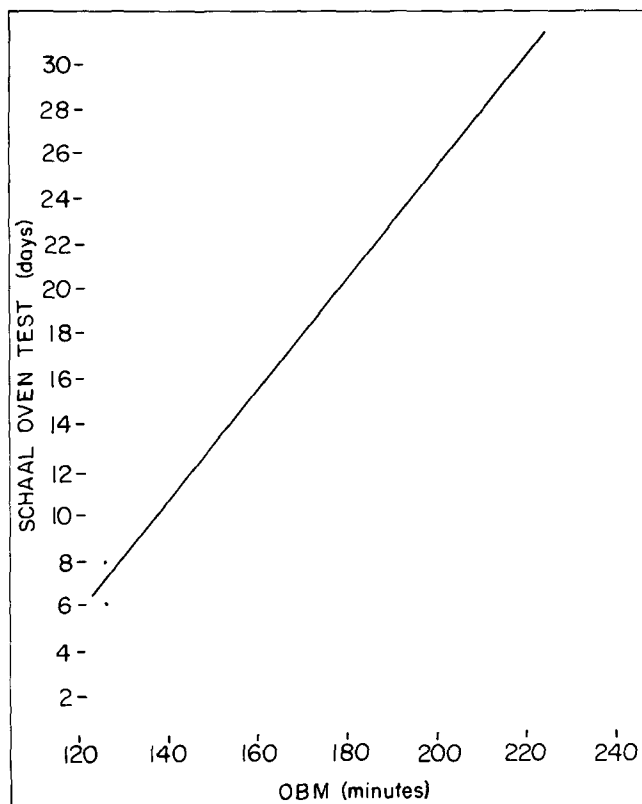
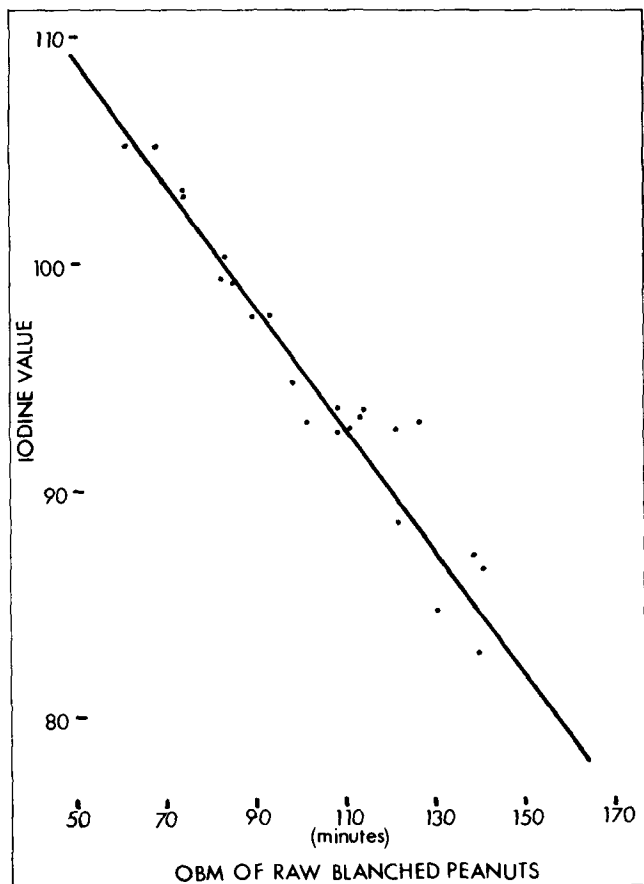


FIG. 4. Correlation curve of Schaal oven test results and oxygen bomb method of roasted blanched peanuts.  $r = 0.9473$ ;  $T = 7.2451$ ;  $DF = 6$ ;  $Sig. = 1.0\%$ .

FIG. 3. Correlation curve of iodine value and oxygen bomb method of raw blanched peanuts.  $r = -.9576$ ;  $T = -15.5914$ ;  $DF = 22$ ;  $Sig. = 0.1\%$ .

One-fourth of each of four samples was placed in a different compartment during successive roastings. This technique provided a very uniform roast. The roasting conditions were: temperature, 205 C; time, 30 min. The peanut butter samples were prepared in a Bauer attrition mill with plates set at ca. .02 in spacing. The peanut butter contained no additives. The oil samples were cold-pressed from raw peanuts. The raw and roasted samples were blanched on a pneumatic sample blancher developed by Barnes et al. (13). The AOM tests were made in accordance with the official AOCs method (9). The iodine values were determined by the refractometric method of the Peanut Research and Education Association, Inc. (14).

The four variables in the OBM tests are temperature, pressure, sample size and surface area. Temperature and pressure changes gave similar results. Increasing either one decreased the length of the test and increased the sharpness of the end point. The temperature, however, seemed to cause a greater effect than pressure. Pressures above ca. 130 psi caused the glass liner to fracture. Particle size or the surface area of the sample also had a significant effect on both the sharpness of the end point and the length of the test. The greater the surface area, the shorter the time for a test and the sharper the end point. Since peanut butter and peanut oil provide comparatively small surface areas as compared to peanut kernels, the length of the test for these two materials is longer and the end point is less sharp. Sample size had little effect on either end point or time of tests.

The OBM tests on the types of material and the AOM tests were made in duplicate in order to compare the precision and sensitivity of each method. As shown in Table I, both the standard deviation of differences between sample duplicates and the coefficient of variability were considerably lower on the OBM results than on the results

from the AOM. A two way analysis of variance showed that the variance among the eight samples was not significant on the AOM but was highly significant on the OBM for all four types of materials. On the other hand, the variance between two replications was not significant in either the AOM or the OBM.

The precision of the OBM was tested further by comparing the OBM results to the iodine values of the oil cold-pressed from the same eight samples used above and 16 additional samples with widely varying genetic properties and known histories.

The 16 additional samples also had been stored for 3 months at 5 C before testing. Figure 3 shows the correlation curve of the OBM results vs. the iodine values for the 24 samples as shown; the correlation coefficient was  $-.9576$  and T value was  $-15.5914$ , which is significant at 0.1% level.

The precision of the OBM was also tested by comparing the OBM results to the Schaal oven test (10) on the same eight samples used above. The peanuts were roasted and blanched before testing. Although the Schaal oven test inherently has large errors, by careful control these errors were reduced to a minimum. Figure 4 shows the correlation curve of the OBM results vs. the Schaal oven tests results. As shown, the correlation coefficient was  $.9473$  and the T value was  $7.2451$ , which is significant at the 1.0% level.

The OBM was found to be not only precise but versatile, in being able to determine oxidative stability on both raw and roasted peanuts, peanut butter and peanut oil. On the other hand, the other methods such as the AOM and the iodine value can be used only on the oil. These other methods also require longer times to carry out, more elaborate equipment and trained personnel; whereas the OBM is a simple and direct approach requiring less time, a minimum of equipment and no specially trained personnel.

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