# USE OF THIOBARBITURIC ACID NUMBER AS A MEASURE OF THE DEGREE OF OXIDATION OF ROASTED MACADAMIA NUTS

The Macadamia nut, which contains approximately 75–80 per cent. oil, is prone to rancidity development, and increased production of roasted locally grown Macadamia nuts in transparent regenerated cellulose film bags has aggravated the problem of stabilization of the nuts against oxidative fat rancidity which occurs during shelf storage in retail stores.

Present knowledge of oxidative fat rancidity indicates that carbonyl compounds are responsible for the rancid flavours and odours. Methods based on the 2-thiobarbituric acid (TBA) reaction with carbonyl compounds and other products of oxidative fat rancidity have been found by Tappel, Knapp, and Urs (1957) to be a simple, reliable means of measuring the oxidative deterioration of walnuts. In addition, the results from TBA tests were found to give good correlation with storage time and organoleptic scores of walnuts, but not of almonds, pecans and filberts.

Studies by Younathan and Watts (1960) on the oxidative changes in fats revealed no correlation between palatability ratings and peroxide numbers, and showed also that determinations such as iodine number and Kries colour reaction of fats frequently are unsatisfactory due to the fact that the fat has deteriorated beyond the point of palatability by the time these tests show significant change. These investigations were therefore conducted to determine whether the TBA number can be used as a measure of rancidity for processed Macadamia nuts and whether this number shows any relation to organoleptic scores.

## **Methods and Materials**

A 24 lb random sample of raw Macadamia nuts (*Macadamia tetraphylla*) was obtained from a commercial source. The sample was subdivided into three 8 lb batches and three separate cooking treatments were carried out, as follows:—

- (a) Dry heat,  $260^{\circ}$ F for 25 min.
- (b) Coconut oil (new), 260°F for 15 min.
- (c) Coconut oil (used), 260°F for 15 min.

After cooking, nuts from treatments (b) and (c) were cooled and salted immediately. The nuts from the dry roasting treatment were coated with hydrogenated oil ("Hydrul") prior to salting. All treatments were then divided into 4 oz batches and packed in regenerated cellulose bags and stored at room temperature to simulate conditions experienced on retail store shelves. Samples consisting of 3 bags of nuts were withdrawn bi-weekly, the TBA number was measured and an organoleptic rating was made.

#### TECHNICAL NOTES

Samples of 50 g were taken at random from each bag for fat extraction. Each sample was finely ground by placing in a Waring blendor for 2-3 min. This method was found to be far quicker and more convenient than conventional mechanical or hand-grinding methods. The ground nut sample was transferred to a 250 ml Erlenmeyer flask with 100 ml of peroxide-free diethyl ether and shaken for one hour. After this time sufficient fat had been extracted for analysis. Longer extraction times, though producing larger quantities of fat, did not give any significant difference in TBA values. The supernatant ether layer containing the extracted fat was decanted and centrifuged for 5–10 min at 2500 r.p.m.

Storage Time (days)	Mean Max. Temp. (°F)	Mean Min. Temp. (°F)	Absorbence at 532 mµ	TBA Value	Organoleptic Rating
0			·00	5 0.04	Raw nuts
7	82	72	N ·03 O ·03 D ·02	$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	Firm texture; no rancidity
21	80	72	N •02 O •03 D •04	9 0·25 2 0·27 4 0·38	Firm texture; no rancidity
25	80	72	N •03 O •03 D •03	4 0·29 6 0·28 3 0·30	Texture softening; no rancidity
33	80	72	N •04 O •04 D •05	2 0·36 8 0·41 5 0·46	Soft texture; no rancidity
35	80	72	N •05 O •05 D •06	1 0·44 5 0·47 0 0·51	Soft texture; no rancidity
39	84	76	N ·08 O ·09 D ·06	2 0.70 0 0.76 6 0.57	Soft texture; all slightly rancid
42	88	82	N ·32 O ·31 D ·22	$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	Soft texture; all rancid. D less rancid than N or O
46	88	82	N O ·40 D ·35	$ \begin{array}{c c}                                    $	Soft texture; all rancid

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## THIOBARBITURIC ACID ASSAY AND ORGANOLEPTIC RATING

N = Nuts roasted in new coconut oil.

O = Nuts roasted in old coconut oil.

 $\mathbf{D} = \mathbf{N}\mathbf{u}\mathbf{t}\mathbf{s}$  dry roasted.

#### TECHNICAL NOTES

The clear solution was placed in a vacuum oven to remove ether traces from the oil, which was then used for the determination of the TBA number by a modification of the Schmidt (1959) method. The reason for modification of this method was that after boiling, cooling, and addition of trichloracetic acid to precipitate protein matter, an emulsion still remained. It was therefore necessary to "break" this emulsion by centrifuging for 5 min at 3000 r.p.m. The centrifuged solution was clear with an oil phase on the surface. This oil was removed and the remaining solution filtered through 10 x 4 sintered glass crucibles into test-tubes. Optical densities were read on an Optica CF4 spectrophotometer at 532 m $\mu$ . This wavelength agrees with data reported by Patton (1960) for malonic dialdehyde, which was shown by Bernheim, Bernheim, and Wilbur (1948) to be a measure of oxidation products of unsaturated fatty acids.

## **Results and Discussion**

The results are summarized in Table 1 and shown graphically in Figure 1. The TBA number increased with an increase in length of the storage period and a good relationship exists between this increase in TBA number and increase in rancidity detected by organoleptic means. The results also show that method of cooking did not have any effect on the development of rancidity. Figure 1 indicates that roasted Macadamia nuts have a general induction period during which the TBA value does not increase to any marked degree. At the end of this induction period there is a very rapid increase in TBA value which corresponds closely to the development of rancid flavour within the processed nuts. From Figure 1 it is clear that the maximum length of shelf storage before the development of rapid deterioration takes place.





#### TECHNICAL NOTES

## Conclusion

From the results of this experiment it can be seen that the TBA number provides a simple, reliable and objective means of measuring oxidative deterioration of processed Macadamia nuts.

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