

# Relationship Between Rancimat and Active Oxygen Method Values at Varying Temperatures for Several Oils and Fats

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**ABSTRACT:** Determination of oxidative stability of different edible oils, fats, and typical fat products was made using the Rancimat method and the active oxygen method. Induction periods (IP) were recorded under controlled conditions at 110, 120, and 130 ± 0.1°C for all products and over a range of 100–160°C for selected fats. A general oil stability evaluation (120°C, 20 L/h air flow rate) using the Rancimat method showed industrial shortenings and vanaspati to be the most stable fats, with IP ranging from 10.00 to 15.47 h. Margarine and butter samples (IP, 4.98–6.04 h) were also found to show fair oxidative stability. Among the extracted and open-market salad-grade cooking oils, rapeseed oil (IP, 4.10 h) and soybean oil (IP, 4.00 h) showed the highest oxidative stability, whereas *Salicornia bigelovii* oil (IP, 1.40 h) was the least stable. The induction periods of typical fat products ranged from 2.59 to 9.20 h. CV for four determinations were <5.2% for shortening and vanaspati products and <4.3% for various vegetable oils, margarine, butter, and typical fat products. Rancimat IP values obtained at 110, 120, and 130°C were 40–46, 20–25, and 9–13% of active oxygen method values, respectively, corresponding to a decrease in Rancimat IP by a factor of 1.99 with each 10°C increase in temperature. Similarly, in the temperature range 100–160°C, an increase of 10°C decreased the Rancimat IP by a factor of 1.99.

Paper no. J10159 in *JAACS* 80, 151–155 (February 2003).

**KEY WORDS:** Accelerated temperature, AOM values, break points, induction periods, lipid oxidation, oil and fats, oxidative stability.

The oxidative stability of oils and fats is one of the most important factors determining their shelf life (1–3). Scientific knowledge of the chemistry of lipid oxidation has increased greatly during the last 30 years (4). Lipid oxidation is a degradation process considered to be a major cause of deterioration in the quality of fat products. It imparts rancid and unpleasant flavors to the products and thus decreases their organoleptic value (5–9). Hence, oxidative stability of shortenings and oils is of concern to bakers and snack food fryers. Determination of oxidative stability is of significance whether oils and fats are used in edible or nonedible products.

Measurement of oxidative stability is usually a difficult task because oxidation at ambient or low temperatures is a slow process. Because time is crucial in industry, it is desirable to measure oil stability rapidly during production and prior to shipment of the products under test, and this is generally achieved by employing accelerated temperature conditions. Thus, it is important to establish whether oxidative stability at one temperature can be correlated with that at another temperature and, if so, what the relationship is between them. We explored this question.

Various chemical tests and accelerated methods have been reported for determination of the oxidative stability of oils and fats (10–14). The active oxygen method (AOM) has traditionally been used for such determinations (14). This method is tedious and nonreproducible and involves the use of toxic chemicals and laborious titration.

The present work reports the determination of the oxidative stability of a series of locally available oils and fat composites over a wide range of temperatures, using a programmed Metrohm Rancimat. The results obtained have been compared with AOM values (14). This work also has been extended to study the effect of elevated temperature on induction periods (IP) of selected fats. Efforts also have been made to develop correlations between AOM values and IP at different temperatures.

## EXPERIMENTAL PROCEDURES

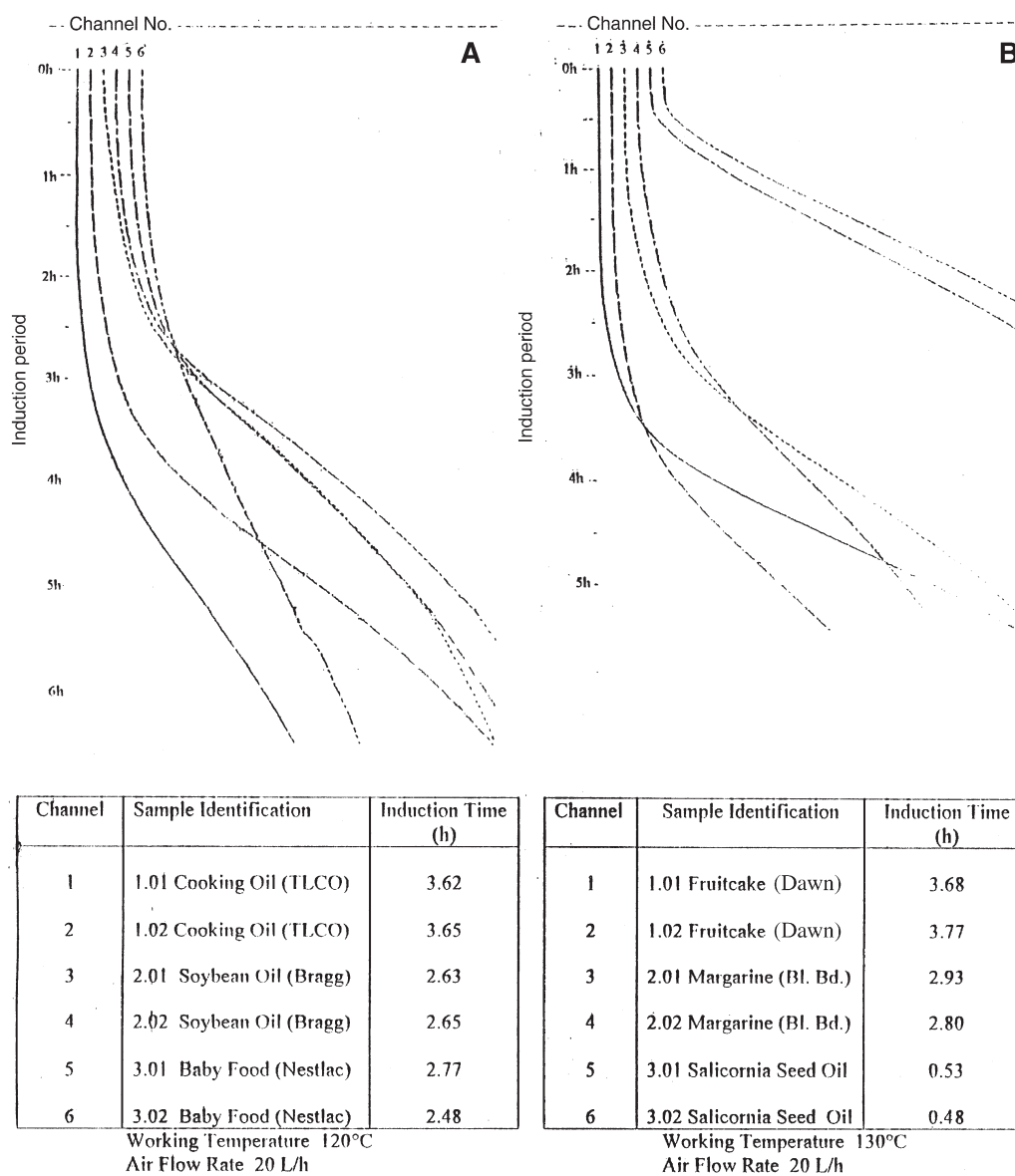
Samples of refined, bleached, and deodorized (RBD) cooking oil (open market oil), vanaspati ghee, shortenings, margarine, butter, and typical fat products were obtained at local retail outlets. Soybean oil (Bragg), sunflower oil (HO-1), cottonseed oil (NIAB-78), *Salicornia bigelovii* seed oil (SOS-10), rapeseed oil (Abasine-95), sarsoon (*Brassica napus*) oil (S-9), and canola oil (Shiralleae) were extracted from seeds procured from the Agriculture Research Institute (ARI) (Tandojam, Pakistan), Nuclear Institute of Agriculture (NIA) (Tandojam, Pakistan), and the Sindh Coastal Development Authority (Karachi, Pakistan). RBD palm oil was obtained from Wazir Ali Industries (Hyderabad, Pakistan). All chemicals and reagents used were from E. Merck (Darmstadt, Germany).

*Sample preparation.* Hydrogenated fats, vegetable oils, and shortenings were melted in a water bath and used directly

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for analytical work. Margarine and butter oils were recovered by melting samples in acid-washed beakers in a water bath at 50°C, allowing phase separation. The upper clear-oil phase was filtered through glass wool coated with sodium sulfate in a drying oven at 50°C (15,16). Fat-containing samples in solid or powdered form, such as powdered milk and baby food, were treated by warm and cold extraction. Each sample (280 g) was weighed in a conical flask and extracted with 500 mL petroleum ether by shaking for 3 h. The mixture was then filtered, and petroleum ether was distilled off at 28°C under vacuum on a rotary evaporator. Samples of products such as puff biscuits, fruitcake, patties, and biscuits (cookies/crackers) were extracted in a Soxhlet apparatus. Recovered fat was dried under nitrogen, stored below 5°C, and melted in a water bath before analysis (15,16).

*Measurement of oxidative stability. (i) Rancimat method.* An automated Metrohm Rancimat apparatus (Model 679; Herisau, Switzerland) capable of operating over a temperature range of 50–200°C was used for the determination of IP of oil and fat samples (12,16,17). Six samples were accommodated in the equipment and analyzed simultaneously. Testing was mainly carried out at 110, 120, and 130 ± 0.1°C. To study the effect of elevated temperatures on IP, the working temperature was also varied over the range of 100–160°C for selected fats (17,18). Oxidative stability was measured following the procedure described elsewhere (15). Briefly, each sample of oil or fat (2.5 g) was weighed into the reaction vessel. The conductometric cells were filled with deionized water up to 70 mL (15). Filtered, cleaned, dried air was allowed to bubble through the hot oil at the rate of 20 L/h. IP of the



**FIG. 1.** Rancimat curves showing induction periods of different samples. (A) Working temperature 120°C, air flow rate 20 L/h; (B) working temperature 130°C, air flow rate 20 L/h. TLCO, Tullio light cooking oil; Bragg, brand of soybean oil; Nestlac, brand of baby food; Dawn, brand of fruitcake; Bl. Bd. (Blue Band), brand of margarine. Samples coded as (e.g.) 1.01 and 1.02 indicate samples run in duplicate.

**TABLE 1**  
**Oxidative Stability of Various Oil and Vanaspati Ghee Samples<sup>a</sup>**

	Rancimat induction period (h) <sup>b,c</sup>						AOM values (h) <sup>c</sup>	
	(110°C)		(120°C)		(130°C)		Mean ± SD	CV%
	Mean ± SD	CV%	Mean ± SD	CV%	Mean ± SD	CV%		
1. Palm oil (RBD commercial)	19.95 ± 0.89	4.46	10.89 ± 0.44	4.04	05.70 ± 0.24	4.21	48.60 ± 3.70	7.61
2. Cooking oil (TLCO)	06.88 ± 0.30	4.36	03.72 ± 0.13	3.49	01.75 ± 0.06	3.42	15.60 ± 1.20	7.69
3. Cooking oil (DCO)	07.00 ± 0.30	4.28	03.61 ± 0.14	3.76	01.80 ± 0.07	3.80	16.20 ± 1.30	8.02
4. Cooking oil (HCO)	05.65 ± 0.22	3.89	03.14 ± 0.10	3.18	01.60 ± 0.06	3.75	13.31 ± 0.93	6.98
5. Cooking oil (SCO)	07.60 ± 0.34	4.47	04.00 ± 0.14	3.50	01.84 ± 0.07	4.34	16.65 ± 1.22	7.32
6. Cottonseed oil (NIAB-78)	05.81 ± 0.23	3.95	03.06 ± 0.12	3.92	01.50 ± 0.05	3.33	12.67 ± 0.88	6.94
7. Rapeseed oil (Abasine-95)	07.38 ± 0.29	3.93	04.10 ± 0.16	3.90	01.85 ± 0.06	3.24	17.00 ± 1.34	7.70
8. Canola (Shiralleae)	07.14 ± 0.28	3.92	03.87 ± 0.16	4.13	02.00 ± 0.08	4.00	15.50 ± 1.28	8.00
9. Soybean oil (Bragg)	05.24 ± 0.21	4.01	02.61 ± 0.11	4.21	01.51 ± 0.05	3.33	12.40 ± 0.90	7.25
10. Sunflower oil (HO-1)	03.70 ± 0.14	3.78	01.89 ± 0.07	3.70	00.88 ± 0.03	3.40	08.60 ± 0.59	6.86
11. <i>Salicornia bigelovii</i> (SOS-10)	03.00 ± 0.02	3.92	01.40 ± 0.06	4.28	00.50 ± 0.02	4.00	05.55 ± 0.40	7.20
12. Vanaspati (Van. Hb.)	29.37 ± 1.28	4.35	14.33 ± 0.75	5.23	06.60 ± 0.29	4.39	64.70 ± 5.50	8.50
13. Vanaspati (Van. Da.)	20.05 ± 0.92	4.58	10.00 ± 0.43	4.30	05.45 ± 0.23	4.22	45.00 ± 3.24	7.20
14. PHVO-a	28.00 ± 1.40	5.00	13.80 ± 0.62	4.49	07.50 ± 0.32	4.40	61.00 ± 4.96	8.13
15. Vanaspati (Van. Ksn.)	25.98 ± 1.35	5.19	14.73 ± 0.63	4.27	07.00 ± 0.30	4.28	62.85 ± 4.75	7.55
16. PHVO-b	27.54 ± 1.33	4.82	15.47 ± 0.71	4.58	06.90 ± 0.33	4.78	66.50 ± 5.50	8.27
17. Powdered milk	17.20 ± 0.76	4.41	09.20 ± 0.36	3.91	04.27 ± 0.18	4.20	40.09 ± 3.50	8.73
18. Fruitcake (Dn)	15.85 ± 0.68	4.29	07.95 ± 0.34	4.27	03.72 ± 0.13	3.49	35.50 ± 2.60	7.32
19. Sandwich biscuits	09.48 ± 0.40	4.21	05.20 ± 0.21	4.03	02.45 ± 0.09	3.67	23.40 ± 1.80	7.69
20. Fruitcake (Vt)	10.99 ± 0.50	4.54	06.00 ± 0.26	4.33	02.64 ± 0.10	3.78	25.80 ± 2.00	7.75
21. Baby food (Nestlac)	05.50 ± 0.21	3.81	02.59 ± 0.10	3.86	01.60 ± 0.06	3.75	12.00 ± 0.73	6.95
22. Patties (C-1)	12.00 ± 0.50	4.16	05.62 ± 0.19	3.38	03.00 ± 0.12	4.04	26.32 ± 2.00	7.59
23. Margarine (Tab.)	10.00 ± 0.43	4.30	04.98 ± 0.20	4.01	02.20 ± 0.08	3.64	22.31 ± 1.70	7.62
24. Margarine (Bl Bd)	12.38 ± 0.55	4.44	06.04 ± 0.25	4.13	02.85 ± 0.11	3.85	30.24 ± 2.45	8.09
25. Shortening (Bac. Bis Sh.)	21.75 ± 1.08	4.96	12.00 ± 0.51	4.25	05.58 ± 0.24	4.30	53.00 ± 4.05	7.64
26. Shortening (Rl. Bn. Sh.)	25.93 ± 1.20	4.62	13.87 ± 0.61	4.39	07.30 ± 0.29	3.97	59.93 ± 4.45	7.42
27. Shortening (Ind. Ch. Sh.)	32.00 ± 1.60	5.00	15.20 ± 0.70	4.60	07.12 ± 0.29	4.07	70.00 ± 5.75	8.21
28. Butter (Pk Pe)	09.15 ± 0.40	4.37	05.43 ± 0.22	4.05	02.75 ± 0.11	4.00	22.52 ± 1.79	7.94
29. Butter (Pk Bd)	12.30 ± 0.51	4.14	06.03 ± 0.24	3.99	02.63 ± 0.11	4.18	27.50 ± 2.30	8.36
30. Butter (Dy Qn)	09.70 ± 0.41	4.22	05.00 ± 0.21	4.20	02.49 ± 0.09	3.61	23.08 ± 1.80	7.79

<sup>a</sup>Comparison between Rancimat and active oxygen method (AOM). Abbreviations: Bac. Bis. Sh., Backman Bisco Shortening; DCO, Dalda cooking oil; HCO, Habib cooking oil; Ind. Ch. Sh., Industrial Champion Shortening; PHVO, partially hydrogenated vegetable oil; PHVO-a and -b, two different PHVO; RBD, refined, bleached, and deodorized; Rl. Bn. Sh., Royal Brown Shortenings; SCO, soy cooking oil; TLCO, Tullo light cooking oil; Van, vanaspati (partially hydrogenated vegetable oils and blended fats); Van. Da., Vanaspati Dalda; Van. Hb., Vanaspati Habib; Van. Ksn., Vanaspati Kisan; tradename products are abbreviated as Dn (Dawn), Vt (Vita), C-1 (Continental); Bl Bd (Blue Band), Pk Pe (Pak Pure), Pk Bd (Pak Brand), and Dy Qn (Dairy Queen); NIAB-78, HO-1, and SOS-10 are codes for different oilseed varieties as provided by the suppliers. Abasine-95, Shiralleae, and Bragg are the identification names of different varieties of rapeseed, canola, and soybean oilseeds, respectively. Tullo, Dalda, and Habib are brands of vanaspati; Royal is a brand of shortening; Nestlac is a brand of baby food; Tab. is a table margarine available on the open market.

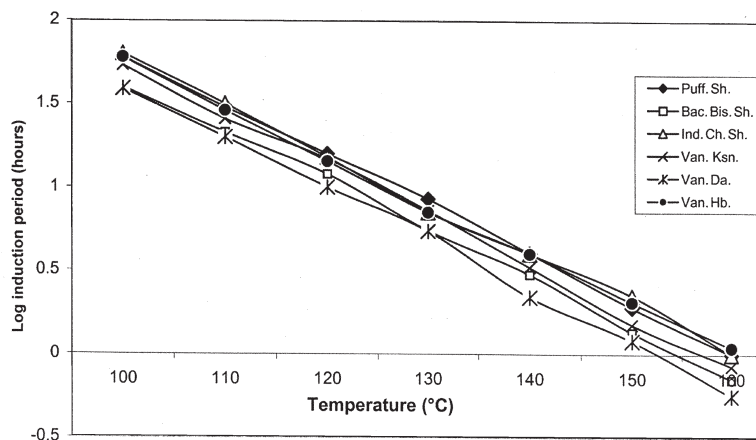
<sup>b</sup>Induction period air flow rate, 20 L/h.

<sup>c</sup>Values are mean of four determinations.

samples were automatically recorded and corresponded to the break point of the plotted curves (the intersection point of the two extrapolated parts of the curve).

**AOM.** A 20-mL portion of a completely melted sample of oil or fat was poured into the reaction tube and placed in a constant-temperature heater kept at  $99 \pm 0.1^\circ\text{C}$ . The aeration assembly was inserted, and air was allowed to flow through the test samples using previously calibrated capillaries. The total flow rate was adjusted to 2.33 mL/tube/min. The time required to reach a PV of 100 meq/kg oil was noted following the standard AOM method (method Cd 12-57; Ref. 14). PV was determined at two time intervals according to AOCS Official Method Cd 8-53 (14), except that different weights were used. A 1.0-g test portion was weighed, and its PV measured a short

time before the expected end point was reached. If this determination indicated a PV between 75 and 175 meq/kg oil, another PV determination was made immediately using a 5-g sample. The test portion was rejected if the PV obtained was greater than 175 meq/kg oil, and another determination was started. When the PV obtained was less than 75 meq/kg oil, the time required to reach this value was estimated. At the end of the estimated time interval, another PV determination was made using a 5-g test portion. PV was determined again on another 5-g test portion from the same reaction tube exactly 1 h later. Thus, two PV values between 75 and 175 meq/kg oil were obtained and plotted against the reaction time. The AOM stability value was the time (in hours) at which the line joining these two points intersected the ordinate for PV of 100.

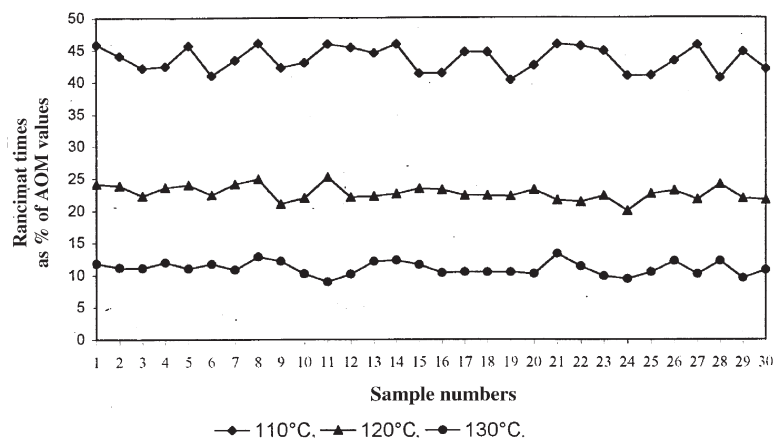


**FIG. 2.** Relationship between induction period and temperature. Abbreviations: Puff Sh., Puff Shortening; Bac. Bis. Sh., Backman Bisco Brand Shortening; Ind. Ch. Sh., Industrial Champion Brand Shortening; Van. Ksn., vanaspati, Kisan brand; Van. Da., vanaspati, Dalda brand (virtually *trans*-free fat); Van. Hb., vanaspati, Habib brand.

## RESULTS AND DISCUSSION

The oxidative stability of different oils and fats was determined by measuring their IP using a programmed Metrohm Rancimat. IP were recorded automatically from the break point of the plotted curves as shown in Figure 1. A comparison of the Rancimat IP obtained at 110, 120, and 130°C and the corresponding AOM values of the same samples is presented in Table 1. For the comparative stability evaluation of different fat composites, 120°C was selected as the standard temperature. The results indicated that industrial shortenings and vanaspati [partially hydrogenated vegetable oils (PHVO) and their blends] were the most stable fats, with IP (120°C, air flow of 20 L/h) ranging from 10.00 to 15.47 h. The corresponding AOM values for these samples were 53.0–70.0 h and 45.0–66.50 h, respectively. This prolonged IP of modified and hardened fats was due to partial hydrogenation, which increases oxidative stability (19,20).

One of the commercial vanaspati (Van. Da.) marketed as virtually *trans*-FA-free was found to show significantly lower oxidative stability (IP, 10.00 h), possibly because it had been hardened by interesterification (21). Rancimat IP and AOM values of margarine and butter oil samples were in the range of 4.98–6.04 h and 22.31–30.24 h, respectively, thus showing a fair oxidative stability. Among the open-market salad-grade, extracted, and commercial vegetable oils, soybean oil (IP, 4.00 h; AOM value 16.65 h), rapeseed oil (IP, 4.10 h; AOM value 17.0 h), and RBD palm oil (IP, 10.89 h; AOM value 48.60 h) showed the highest oxidative stability, whereas *S. bigelovii* seed oil (IP, 1.40 h; AOM value 5.55 h) was found to be the least stable oil. The high oxidative stability of RBD palm oil and the low stability of *S. bigelovii* seed oil as compared with other oils were attributed to their constituent FA. The former is rich in palmitic (C<sub>16:0</sub>) and oleic (C<sub>18:1</sub>) acids (22), which are less susceptible to oxidation (22,23), whereas the latter contains a high proportion of linoleic acid



**FIG. 3.** Relationship of Rancimat induction period as percentage of active oxygen method (AOM) values.

(C<sub>18:2</sub>), which is more prone to oxidation (24,25). Typical fat products (powdered milk, fruitcake, biscuits—cookies/crackers, baby food, patties) were found to show variable oxidative stability, with IP and AOM values in the range of 2.59–9.20 h and 12.00–40.09 h, respectively.

Figure 2 shows the effect of elevated temperature (100–160°C) on IP of selected fat products (shortenings, vanaspati). The IP values are plotted on a logarithmic scale against temperature. Linear least-squares regression of these data yielded correlation coefficients  $r = 0.9979$ – $0.9992$  ( $n = 7$ ). The logarithmic relationship was found to hold between 100 and 160°C for all six hardened fats investigated here. Based on the IP–temperature relationship obtained, each 10°C rise in temperature decreased the IP by a factor of 1.991, in agreement with earlier reports (17).

Rancimat IP at 110, 120, and 130°C as percentages of the AOM values are plotted in Figure 3. Rancimat IP at 110, 120, and 130°C were 40.31–46.06, 19.97–25.22, and 9.00–13.33, of the AOM times, respectively. Our values at 110°C were in close agreement with those reported in the literature (26). However, no data were available with which to compare our results obtained at 120 and 130°C. From these results, it was noted that an increase in temperature of 10°C (i.e., 110–120°C and 120–130°C), decreased the Rancimat times as percentages of AOM values by a factor of 1.93 and 2.06, respectively (mean 1.99). No data were found in the literature to compare with this finding. Analysis by the Rancimat method was four to five times more rapid than by the AOM.

The relationships established during the present work are of significance because they indicate that oxidative stability data at one temperature are interconvertible with those at other temperatures. The use of such relationships makes it possible to extrapolate the temperature-dependent data to predict oil stability at other temperatures at which the determination of oil stability is impeded by time constraints.

## ACKNOWLEDGMENT

The authors would like to express sincere gratitude to Ashfaq Ahmed Waraich and M. Asif Choudhary of Wazir Ali Industries, Hyderabad, Pakistan, for providing research facilities for this work.

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[Received November 19, 2001; accepted November 20, 2002]