TABLE I

Application of Correction Factors to a Fraction of Menhaden Oil Methyl Esters

Sample	Fatty acid—no. carbons : double bonds													
	14:0	16:0	16:1	18:0	18:1	18:2	18:3	18:4	20:0	20:4	20:5	22:0	22:5	22:6
	%	%	%	%	%	%	%	%	%	%	%	%	%	%
Fraction of menhaden oil (M.O.) methyl esters—uncorrected	0.4	5.3	3.2	5.5	12.8	3.4	5.0	4.9	....	0.9	22.3	....	1.8	34.4
Fraction of M.O. methyl esters—corrected	0.3	3.7	2.2	3.6	8.5	2.3	3.4	3.7	....	0.8	21.5	....	2.2	47.7
Sum of acids of a carbon length M.O. methyl esters—uncorrected	0.4	8.5	....	31.6	....	....	....	....	23.2	....	....	36.2	....	....
Sum of acids of a carbon length M.O. methyl esters—corrected	0.3	5.9	....	21.5	....	....	....	....	22.3	....	....	49.9	....	....
Hydrogenated M.O. methyl esters	....	6.3	....	19.2	....	....	....	....	28.1	....	....	46.4	....	....

mental conditions it has been found that the column has a useful life of approximately 300 hr and that any standardization conducted during this time will give the same correction factors for the various retention times. Therefore, the standardization method outlined need only be carried out once for a column. It should be stressed that this curve and the correction factors for various retention times are not applicable to any other instrument or, in our experience, with another column in this same instrument. However, as a column is used, the retention time usually decreases and, therefore, the correction factor that must be applied from day to day decreases. The use of the relationship between retention time and correction factor makes it possible to eliminate the determining of correction factors each day.

Application of correction factors to an analysis of a fraction from menhaden oil is shown in Table I. The analysis of the same sample completely hydrogenated is also given. Only very small correction factors were required for 22:0 for the hydrogenated sample since it had a retention of 158 spaces. When correction factors were not applied, abnormally high amounts of 16 and 18 carbon fatty acids would appear to be present while the 20 and 22 carbon acids were underestimated. Note the good agreement in the quantities of 22 carbon atom fatty acid in the hydrogenated and corrected sample (46.4 vs. 49.9).

H. M. EDWARDS, JR. and J. E. MARION
Poultry Department
University of Georgia
Athens, Ga.

REFERENCES

1. Messner, A. E., D. M. Rosie, and P. A. Argabright, Anal. Chem., *31*, 230 (1959).
2. Killheffer, J. V., and E. Jungermann, JAOCS, *37*, 456 (1960).
3. Horrocks, L. A., D. G. Cornwell, and J. B. Brown, J. Lipid Research, *2*, 92 (1961).

[Received July 23, 1962—Accepted April 17, 1963]

Errors in the Azelaoglyceride Technique

IN A COMMUNICATION from this laboratory (1), it was pointed out that there are two sources of error in Kartha's method for the determination of glyceride composition (2) *viz.*, formation of incompletely oxidized glycerides containing *presumably* acetylated α-ketols (e.g. $GS_2S°$) and liberation of saturated acids by hydrolysis of azelaoglycerides. In several recent communications (3,4,5), Kartha contends that these observations are based on artifacts of procedures used by us.

While it is true that the presence of ketols was not confirmed by specific keto group reactions, it is certain that some neutral products are formed during the oxidation, which influence the yield, A.V. and S.V. of the insoluble azelaoglycerides (IAG). It is regretted that Kartha has cited only one example in support of his contention. Even so, the data and reasoning presented by him are entirely misleading. In the particular example cited (Tables IV & VI, expt. 6, ref. 1), about 12% of $GS_2S°$ were obtained; and a careful reader will observe that the yield of IAG in this case is 91.3% (theoretical 92.6%, assuming formation of $GS_2S°$) and not 89.3%, as wrongly cited by Kartha. He has also overlooked a number of other points. The yield and S.V. of IAG from GS_2U are not enough proof for the absence of $GS_2S°$, as these are based on the net effect of incomplete oxidation, which increases both of these and hydrolysis, which decreases the same. In the absence of $GS_2S°$ the yield should have been 89.9, however, the experimental value of 91.3 clearly shows that it is in between the values cited above.

As further proof of the absence of $GS_2S°$, Kartha states that the S content of a fat obtained from the azelaoglycerides is the same as that obtained by the oxidation of its mixed fatty acids. However, in the latter there is no saponification with alcoholic KOH prior to its estimation by Bertram separation (6). This agreement is based entirely on the yield of S. A closer examination would show that an increase in yield, due to incomplete oxidation of oleic acid, would be compensated by the loss of stearic acid due to secondary oxidation, as is unequivocally proved (7), but not referred to by Kartha in any of his communications (3,4,5). Furthermore, a sample of methyl oleate containing 2% of methyl stearate, on Kartha's oxidation, carbonate washing and chromatographic adsorption gave 1.6% of neutral products (S° of S.V. 343) after accounting for stearate and unoxidized oleate (8).

Kartha also calculated the S.V. of the scission products lost in Bertram separation by assuming that the α-ketol (from oleins) on saponification with alcoholic KOH yields *quantitatively* azelaic and nonoic acids. However, this was shown to be incorrect (9). On the basis of this calculation, he deduces that the neutral products eluted from the alumina column, contain distearononoin and stearodinonoin formed by ester interchange. Replacement of a half-esterified dicarboxylic acid (azelaic) in azelaoglycerides with a free

monocarboxylic acid (nonoic) by "ester" interchange on a *basic* alumina column has not been reported, nor has Kartha put forward any experimental evidence in support of this. If his reasoning is correct, how could he explain the presence of 5–8.8% of neutral products obtained by the chromatographic adsorption of oxidized GS_2U (containing 2.8% GS_3) and GSU_2 (0.0% GS_3) concentrates, particularly when these were adsorbed *only after removal of low molecular weight acids* (nonoic etc.) by his magnesium salt separation (Tables II and IV, experiments 7,8,9, ref. 1). Also, how could the presence of 9.0% (2.8% GS_3) of neutral products obtained from GS_2U concentrate by a mere carbonate washing procedure (Table III, experiment 6, ref. 1) be explained, even after it was very conclusively demonstrated that GS_2A are not hydrolyzed during this washing procedure. Contrary to Kartha's allegation (4), the chromatographic procedure employed by us was *standardized*, using known mixtures of pure GS_3, GS_2A, stearic acid, oxidized *G. Indica* fat, and GS_2U containing nonoic acid (1,10,11).

As reported earlier (1), the yield of IAG alone is no proof for the absence of hydrolysis of GS_2U and GSU_2 as other data on the IAG (S.V., A.V., percentage S) have also to be taken into consideration. Hydrolysis in a procedure cannot be detected by analyzing fats by the same procedure, which is considered to be in error. Conclusive proof can only be obtained by employing concentrates whose composition is definitely known (1). And if this is not sufficient, then it is suggested that mixtures of 2-oleodistearin and 1-stearodiolein in the proportions of about 70:30 and 30:70 and also pure 1-stearodiolein be analyzed. The analysis of these has been carried out in this laboratory (unpublished work) using the conditions specified by Kartha.

REFERENCES

1. Lakshminarayana, G., and D. Rebello, JAOCS *37*, 274–281 (1960).
2. Kartha, A. R. S., *Ibid. 30*, 280–282 (1953).
3. Kartha, A. R. S., *Ibid. 39*, 478–479 (1962).
4. Kartha, A. R. S., and R. Narayanan, J. Sci. Food Agric. *13*, 411–414 (1962).
5. Kartha, A. R. S., and R. Narayanan, J. Sci. Ind. Research (India) *21B*, 494–498 (1962).
6. Kartha, A. R. S., and R. Narayanan, Indian J. Agric. Sci. *27*, 73–77 (1957).
7. Lakshminarayana, G., and D. Rebello, J. Sci. Ind. Research (India) *19B*, 412-413 (1960).
8. Holla, K. S., (Technology) Thesis, University of Bombay, 1959.
9. Hilditch, T. P., and H. Plimmer, J. Chem. Soc. 204–206 (1942).
10. Lakshminarayana, G., and D. Rebello, J. Sci. Ind. Research (India) *14B*, 189–190 (1955).
11. Lakshminarayana, G., (Technology) Thesis, University of Bombay, 1957.

G. LAKSHMINARAYANA and D. REBELLO
Department of Chemical Technology
Universtiy of Bombay
Bombay, India

[Received December 21, 1962—Accepted March 12, 1963]

Lanolin nee Woolgrease

WE WISH to draw attention to several inaccuracies in the above report by S. T. Goode, which was featured in the February issue of JAOCS.

The work of Weitkamp has been greatly extended by several different workers in the last fifteen years, and the present position is summed up in Table I, taken from Downing (1).

The groups of acids identified by Weitkamp have been extended to include ω-hydroxy acids, and additional compounds in each of the groups have been identified.

Contrary to Goode's statement, the alcohol fraction is almost as fully identified as the acid fraction.

The figures in Table I are for freshly secreted wool wax. The grease on the wool normally contains a high proportion of oxidized material, very little of which has been identified chemically, and which is the source of color and free acidity (2). The oxidized material is recovered in the acid cracking process but not in the centrifugal process, which largely explains the differences between the two products. More oxidized material is recovered centrifugally from neutral nonionic scour liquors than from alkaline soap liquors, and the spread of neutral scouring over the last few years has resulted in a reduction in the quality of crude wool grease, with a consequent increase in refiners' problems.

The statement that the solvent and soap/soda scouring processes have become obsolete because of the advance of science gives a false picture of the situation. In Bradford, England, which is the largest wool scouring center in the world, soap/soda scouring is still used exclusively. The "advance of science" has led to the introduction of at least two new solvent scouring processes in the last 10 years (3,4).

Felting is not "splitting of the woolen fibers."

The use of a suint bowl (i.e. an initial wash in relatively cool water) is by no means as universal in wool scouring as Goode implies.

When nonionic detergents are used for scouring, the main bowls are operated at about 65C. The 49C given by Goode is the temperature used for soap/soda scouring.

TABLE I

Composition of Acid and Unsaponifiable Fractions of Wool Wax

Composition	Weight %
A. Acid Fraction	
Alkanoic acids	60
α-Hydroxy acids	5
ω-Hydroxy acids	30
Undetermined	5
B. Unsaponifiable Fraction	
Hydrocarbons	0.3
Monoalcohols	9.5
α, β-Diols	6.5
Cholesterol	31
Lanosterol	44
Undetermined	8.7

REFERENCES

1. Downing, D. T., Z. H. Kranz, and K. E. Murray, Aust. J. Chem., *13*, 80 (1960).
2. Anderson, C. A., and G. F. Wood, Nature, *193*, 742 (1962).
3. Report No. G10, Division of Textile Industry, C.S.I.R.O. Wool Research Laboratories, Geelong, Victoria, Australia.
4. Technical Bulletin No. 5801E, Alfa Laval Co. Ltd., Brentford, Middlesex, England.

C. A. ANDERSON and G. F. WOOD
Division of Textile Industry
C.S.I.R.O. Wool Research Laboratories,
Geelong, Victoria, Australia

[Received April 22, 1963—Accepted May 17, 1963]