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## REFRACTIVE INDICES OF SOME SATURATED AND MONOETHENOID FATTY ACIDS AND METHYL ESTERS

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*Canadian Journal of Chemistry*, 1953, 31(5): 499-504, <https://doi.org/10.1139/v53-068>

### ABSTRACT

The refractive indices for a number of saturated and monoethenoid fatty acids and their corresponding methyl esters have been determined. Equations have been calculated to obtain the refractive index at any given temperature. Significant differences have been found for the temperature coefficients of refractive index within an homologous series and between the free fatty acid and the corresponding methyl ester.

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# REFRACTIVE INDICES OF SOME SATURATED AND MONOETHENOID FATTY ACIDS AND METHYL ESTERS<sup>1</sup>

BY B. M. CRAIG<sup>2</sup>

## ABSTRACT

The refractive indices for a number of saturated and monoethenoid fatty acids and their corresponding methyl esters have been determined. Equations have been calculated to obtain the refractive index at any given temperature. Significant differences have been found for the temperature coefficients of refractive index within an homologous series and between the free fatty acid and the corresponding methyl ester.

The literature contains few references to systematic studies on the refractive indices of the fatty acids and their esters, particularly the variation in refractive index with temperature. Dorinson, McCorkle, and Ralston (1) have reported refractive indices for the saturated fatty acids from caproic to stearic at five degree intervals of temperature. Wyman and Barkenbus (8) determined the refractive indices of the methyl esters of this series of fatty acids at 45°C. and this work was extended by Mattil and Longenecker (5) who determined the variation in refractive index as a function of temperature. The latter workers computed equations to calculate the refractive indices at any intermediate temperatures. More recently Krewson (3) has published data on the refractive indices of the methyl esters of the saturated acids up to and including methyl octacosanoate at temperatures of 50°C. and 80°C.

Data on refractive indices of unsaturated acids and esters are more meager than for the corresponding saturated series. Wood *et al.* (7) have reported on the refractive indices of oleic, elaidic, linoleic, and linolenic acids at 50°C. McCutcheon (4) determined the refractive indices of ethyl linoleate and ethyl linolenate over the range 20°C. to 60°C.

In the course of some work on rapeseed oil the saturated and monoethenoid acids and esters of the C<sub>16</sub> to C<sub>22</sub> series were purified, and a refractive index study was made on these materials. Equations were calculated from the data expressing the refractive index as a function of temperature.

## MATERIALS AND METHODS

Crude commercial stearic acid was used as a source for methyl palmitate and methyl stearate. The crude acid was hydrogenated to a negligible iodine value and converted to methyl esters by conventional procedures. The crude esters were distilled in a Podbielniak "Heli-Grid" distillation column. The methyl palmitate and methyl stearate fractions were redistilled and fractionally crystallized to yield pure esters. Methyl oleate was obtained by converting olive oil to the methyl esters and distilling the esters. The C<sub>18</sub> fraction was repeatedly crystallized from acetone until the precipitates and filtrates agreed in refractive index. The purified methyl oleate was then redistilled in

<sup>1</sup> Manuscript received January 30, 1953.

Contribution from the Prairie Regional Laboratory, Division of Applied Biology, National Research Council, Saskatoon, Sask. Issued as N.R.C. No. 2961.

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the Podbielniak column. Methyl eicosenoate and methyl erucate were prepared from rapeseed oil in the same manner. Methyl behenate and methyl arachidate were prepared from the corresponding monoethenoid esters by hydrogenation with Raney nickel followed by fractional crystallization.

The pure fatty acids were obtained by saponification of the corresponding pure methyl esters and fractional crystallization. The physical and chemical constants of the esters and acids are given in Table I. Iodine values were

TABLE I  
PHYSICAL AND CHEMICAL CONSTANTS FOR FATTY ACIDS AND METHYL ESTERS

	Acids				Esters			
	I.V.	Theor. I.V.	M.P.	Reported M.P.	I.V.	Theor. I.V.	M.P.	Reported M.P.
Oleic	89.90	89.87	—	—	85.63	85.62	—	—
Eicosenoic	81.62	81.75	24.2	23-24	78.15	78.22	—	—
Erucic	74.81	74.98	33.0	33.5 (6)	71.91	71.99	—	—
Palmitic	0	0	63.3	62.9 (2)	0	0	29.9	30.6 (2)
Stearic	0	0	70.1	69.6 (2)	0	0	39.1	39.1 (2)
Arachidic	0	0	75.4	75.35 (2)	0	0	46.9	46.6 (2)
Behenic	0	0	80.7	79.95 (2)	0	0	53.3	53.3 (2)

determined by the Wijs method, one hour reaction time and 0.1 *N* solution. Melting points were measured by the Wiley method using a Bureau of Standards thermometer and applying stem emergence corrections. Diene and triene acid contents of the unsaturated esters and acids were determined according to the standard A.O.C.S. procedure. No measurable quantities were found in any of the monoethenoid esters or acids.

A Bausch and Lomb Abbe type refractometer equipped with a circulating water bath controlled to  $\pm 0.005^\circ\text{C}$ . was used to measure the refractive indices. A thermometer calibrated against a Bureau of Standards thermometer was used to read the temperature in the prism and stem emergent corrections were applied to the thermometer reading. No prism corrections were applied to the refractive index values. The refractive indices were measured at five degree intervals over the temperature range at which the material under study was in the liquid state from  $20^\circ\text{C}$ . to  $85^\circ\text{C}$ . The refractometer was carefully calibrated with the test piece supplied by the manufacturer and checked with purified ethyl oxalate and ethyl citrate.

#### RESULTS

Equations for calculation of refractive indices of the esters and acids at any given temperatures are listed in Table II. The equations were calculated by the method of least squares and the error of estimate for the equation and the standard error of the regression coefficient are included. The errors of estimate of the equations listed in Table II are of the same order as the error of measurement of refractive index credited to the instrument used in the

TABLE II  
EQUATIONS FOR CALCULATION OF REFRACTIVE INDICES

		Standard error of estimate	Standard error of regression coefficient
Methyl oleate	$R.I.t = 1.45968 - 0.000377t$	0.00005	0.000,001
Methyl eicosenoate	$R.I.t = 1.46134 - 0.000372t$	0.00005	0.000,001
Methyl erucate	$R.I.t = 1.46288 - 0.000369t$	0.00009	0.000,002
Methyl palmitate	$R.I.t = 1.44830 - 0.000379t$	0.00008	0.000,002
Methyl stearate	$R.I.t = 1.45149 - 0.000375t$	0.00006	0.000,001
Methyl arachidate	$R.I.t = 1.45363 - 0.000366t$	0.00005	0.000,001
Methyl behenate	$R.I.t = 1.45554 - 0.000358t$	0.00007	0.000,002
Oleic acid	$R.I.t = 1.46677 - 0.000354t$	0.00009	0.000,002
Eicosenoic acid	$R.I.t = 1.46805 - 0.000351t$	0.00008	0.000,001
Erucic acid	$R.I.t = 1.46892 - 0.000346t$	0.00008	0.000,002
Palmitic acid	$R.I.t = 1.45589 - 0.000355t$	0.00006	0.000,004

*R.I. t = Refractive index at temperature t°C.*

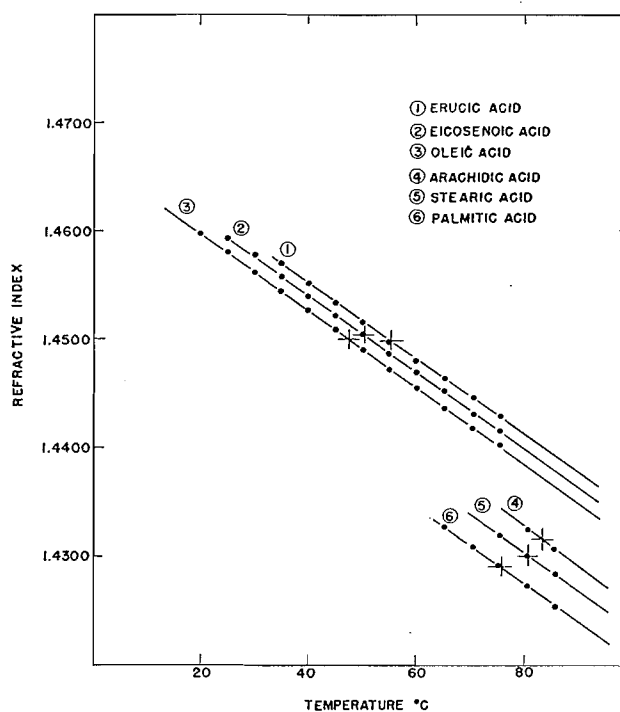


FIG. 1. Refractive indices of fatty acids at different temperatures.

study. Lines representing the equations and the experimental values for refractive indices are presented graphically in Figs. 1 and 2.

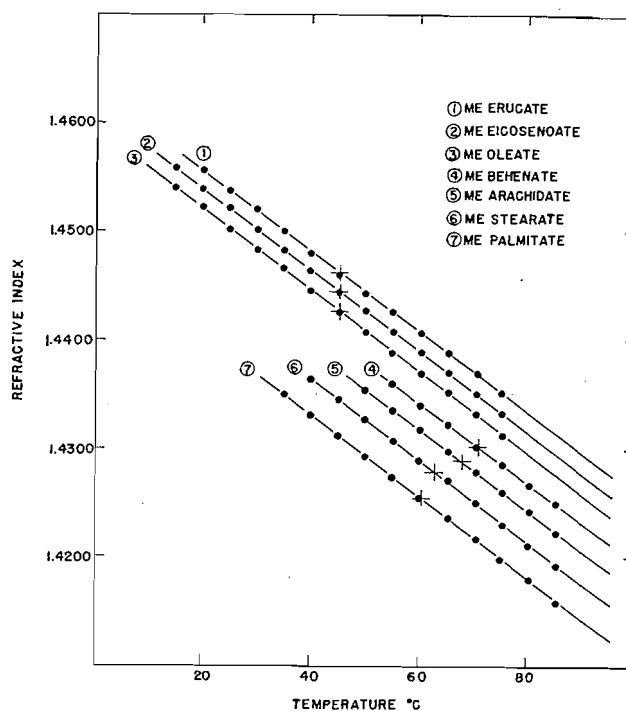


FIG. 2. Refractive indices of methyl esters of fatty acids at different temperatures.

Table III shows a comparison of the values obtained experimentally and those calculated from the equations given in Table II. Experimental values are included for stearic, arachidic, and behenic acids where there were insufficient data to calculate the necessary equations.

The refractive indices for acids and esters given in this study agree quite well with those reported by other workers. The equations which are given in Table II represent an accurate and convenient means for calculating refractive indices at any temperature and are a definite advantage where temperatures other than those reported in the literature are used to measure refractive index. It is interesting to note that the temperature coefficients of refractive index given in the equations in Table II vary in a regular manner within an homologous series of acids or esters. Statistically the difference may not be significant between any two consecutive members of the series, e.g. methyl palmitate (0.000379) and methyl stearate (0.000375), but the difference is significant between two members such as methyl palmitate (0.000379) and methyl behenate (0.000358). It must also be noted that there are significant differences between the temperature coefficients of refractive index for the methyl esters and the corresponding free fatty acids. The practice of using the general figure 0.00038 as the temperature coefficient of refractive index will lead to an error, the magnitude of which will depend on the temperature being used.

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