was made basic with 20% sodium hydroxide solution. The base was taken up in ether and the solution was dried over anhydrous magnesium sulfate. The solvent was removed by vacuum distillation at 30–35°. Immediately before using the residual oil in the condensation reaction, a sample was withdrawn and converted to the picrate, (m. p. 158–160°). After crystallization from alcohol, the melting point was 166–167°. Therefore, the chloroamine added to the reaction was 2-dimethylamino-1-chloropropane.

Summary

1-Dimethylamino-2-chloropropane and 2-dimethylamino-1-chloropropane were prepared from the corresponding dimethylaminopropanols by the action of thionyl chloride.

2-Dimethylamino-1-chloropropane, either as the free base or as the hydrochloride, rearranges on heating into 1-dimethylamino-2-chloropropane which is thermally stable.

Both chloroamines reacted with diphenylacetonitrile to give the same mixture of isomeric aminonitriles.

Glenolden, Pa. Received July 7, 1947

[Contribution from the Eastern Regional Research Laboratory[1]]

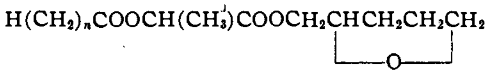
n-Alkanoyl Derivatives of *n*-Butyl and Tetrahydrofurfuryl Lactates

By M. L. Fein and C. H. Fisher

Several alkyl lactates have been acylated[2] with the lower acid anhydrides and chlorides, but little is known about acylated lactic esters of relatively high molecular weight. The present paper describes several homologous acyl derivatives (I and II, *n*-1, 2, 3, 6, 8 and 11) of *n*-butyl lactate and tetrahydrofurfuryl lactate, prepared in high yields by treating the corresponding lactic esters with various aliphatic acid chlorides or anhydrides.

$$H(CH_2)_nCOOCH(CH_3)COO(CH_2)_4H$$

I



$$H(CH_2)_nCOOCH(CH_3)COOCH_2\underbrace{CHCH_2CH_2CH_2}_{O}$$

II

The *n*-alkanoyl derivatives (I and II) were purified carefully by fractional distillation. The resulting colorless liquids were used in the determination of density (*d*), refractive index (*n*), viscosity (η), and boiling points (T) under different pressures. These physical properties of the two homologous series (I and II) were related to the number of carbon atoms, *x* (or molecular weight) by plotting certain functions of the physical properties (x/d, x/n, $\log \eta$, and T^2) against *x*. The resulting straight line relationships are useful in estimating properties of other homologous members of I and II by interpolation and extrapolation, comparing the properties of I and II with those of other homologous series, and correlating x/d, x/n, $\log \eta$ and T^2 with one another (within a homologous series, *i.e.*, I or II).

Experimental

Materials.—Redistilled *n*-butyl lactate (b.r. 79–80° at 12.4 mm.) from a commercial source and tetrahydrofurfuryl lactate[3] (b.p. 92° at 1 mm.) prepared by alcoholysis of methyl lactate were used. Commercial grades of acetic, propionic, and butyric anhydrides and pelargonyl chloride were used; the *n*-heptanoyl and *n*-dodecanoyl chlorides were Eastman Kodak Co. White Label grade.

Acylation: Acid Anhydrides.—The previously described[2b] method was used to acylate the lactic esters.

Acid Chlorides.—The acid chloride (1 mole per mole of lactic ester) was added slowly with stirring to a mixture of the lactic ester, reagent-grade pyridine (10% excess), and benzene (100 ml. per mole of lactic ester) contained in a 3-necked flask fitted with a condenser, mechanical stirrer, and glass-stoppered funnel. In most instances, the temperature of the reaction mixture was kept at 10 to 20° by external cooling. Cooling is recommended, although it was omitted in some experiments (temperature rose to 80°). When the reaction was complete (shortly after the addition of all the acid chloride), a small quantity of water was added to dissolve the pyridine hydrochloride. The oil layer was separated and washed in succession with dilute hydrochloric acid, saturated brine, and dilute sodium bicarbonate. In some instances, water was removed by drying over anhydrous sulfate; in others this treatment was omitted, and the water was distilled azeotropically with the benzene already present. The low- and high-boiling materials were then distilled under reduced pressures with a water aspirator and oil pump, respectively. Vigreux columns approximately 50 cm. high were used for most of the distillations. Yields and properties of the products are given in Tables I and II.

Determination of Physical Constants.—The boiling points used for the construction of Fig. 1 were determined, as in a previous study,[4] by carefully distilling narrow-boiling fractions either through a 60-cm. Vigreux column or with an alembic flask[5]; pressures were read from a Dubrovin gage.[6] When the same equipment and technique were used to determine boiling points of butyl phthalate at several pressures between 2 and 10 mm., data agreeing with those of Hickman[7] were obtained. The boiling points of the acylated lactic esters (I and II) were plotted on a Cox chart[8]; boiling points at 10 mm. were taken from the chart (Fig. 1).

Refractive indices and specific gravities (Table I) were determined with an Abbe type refractometer and a 10-ml.

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(1) One of the Laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, United States Department of Agriculture. Article not copyrighted.

(2) (a) R. Burns, D. T. Jones, and P. D. Ritchie, *J. Chem. Soc.*, 400 (1935); (b) M. L. Fein and C. H. Fisher, *Ind. Eng. Chem.*, **36**, 235 (1944).

(3) M. L. Fein, W. P. Ratchford and C. H. Fisher, This Journal, **66**, 1201 (1944).

(4) M. L. Fein and C. H. Fisher, *ibid.*, **68**, 2631 (1946).

(5) J. C. Cowan, L. B. Falkenburg and H. M. Teeter, *Ind. Eng. Chem.*, *Anal. Ed.*, **16**, 90 (1944).

(6) J. Dubrovin, *Instruments*, **6**, 194 (1933); F. E. E. Germann and K. A. Gagos, *Ind. Eng. Chem., Anal. Ed.*, **15**, 285 (1943).

(7) K. C. D. Hickman, *J. Franklin Institute*, **221**, 383 (1936); G. E. Barker, G. E. Alter, Jr., C. E. McKnight, J. R. McKlveen and D. M. Hood, A. S. T. M. Bulletin No. 139, 25 (Mar. 1946).

(8) E. R. Cox, *Ind. Eng. Chem.*, **15**, 592 (1923); G. Calingaert and D. S. Davis, *ibid.*, **17**, 1287 (1925).

TABLE I

ACYL DERIVATIVES OF *n*-BUTYL AND TETRAHYDROFURFURYL LACTATES[a]

Acyl deriv.	B. p., °C.	B. p., Mm.	d^{20}_4	n^{20}_D	M^{20}_D Found	M^{20}_D Calcd.	Ester equiv. Found	Ester equiv. Calcd.	Analyses, % Carbon Found	Analyses, % Carbon Calcd.	Analyses, % Hydrogen Found	Analyses, % Hydrogen Calcd.
					n-Butyl lactate							
Acetyl[b]	90	7.8	1.0036	1.4163	47.09	47.07	95.2	94.1	...	...	...	...
Propionyl[b]	118	18.7	0.9864	1.4178	51.65	51.69	100.9	101.1	...	...	...	...
n-Butyryl[b]	90	2	.9731	1.4215	56.40	56.31	108.9	108.1	60.91	61.09	9.07	9.32
n-Heptanoyl[c]	122	2	.9477	1.4290	70.27	70.16	129.3	129.2	64.84[d]	65.08	...	...
n-Nonanoyl[e]	143	3	.9353	1.4339	79.70	79.40	142.3	143.2	66.92	67.09	10.05	10.56
n-Dodecanoyl[c]	142	0.6	.9218	1.4388	93.68	93.25	163.8	164.2	69.54	69.47	11.12	11.05
					Tetrahydrofurfuryl lactate							
Acetyl[b]	109	1.2	1.1234	1.4445	51.18	51.13	108.2	108.1	...	...	...	...
n-Butyryl[b]	92	0.2	1.0768	1.4448	60.33	60.39	122.5	122.1	59.07	59.00	8.32	8.25
n-Heptanoyl[c]	171	5.4	1.0300	1.4478	74.38	74.22	143.1	143.2	62.89	62.91	9.14	9.15
n-Nonanoyl[e]	202	10.0	1.0121	1.4498	83.45	83.46	154.8	157.2	64.75	64.94	9.44	9.62
n-Dodecanoyl[c]	205	2.7	0.9843	1.4522	97.74	97.31	180.2	178.3	67.92	67.38	10.41	10.18

[a] Acylation yields of product distilling over approximately 3° range were approximately 90% of the theoretical, except for the nonanoates (15 to 20° range). The authors are indebted to Mary J. Welsh, Mildred Gaspar and C. O. Willits for analytical data. [b] Acid anhydride used for acylation. [c] Acid chloride (Eastman Kodak Co. White Label) used. [d] Carbon determined by wet oxidation (D. D. Van Slyke and J. Folch, *J. Biol. Chem.*, 136, [2] 509 (1940)). [e] Commercial grade of pelargonyl chloride used.

TABLE II

VISCOSITIES[a] OF ACYL DERIVATIVES OF *n*-BUTYL LACTATE AND TETRAHYDROFURFURYL LACTATE

Acyl deriv.	Viscosity at 20° Centistokes	Viscosity at 20° Centipoises	I at 20°[b] Found	I at 20°[b] Calcd.	I/M[c]
		Butyl Lactate			
Acetyl	3.19	3.20	577.2	571.9	12.25
Propionyl	2.92	2.88	629.0	627.5	12.19
n-Heptanoyl	5.21	4.94	853.1	849.9	12.14
n-Nonanoyl	6.74	6.30	966.2	961.1	12.12
n-Dodecanoyl	10.84	9.99	1140.4	1127.9	12.17
		Tetrahydrofuryl Lactate			
Acetyl	12.09	13.58	621.4	627.8[d]	12.14
n-Butyryl	10.30	11.09	728.1	739.0[d]	12.07
n-Heptanoyl	13.06	13.45	897.6	905.8[d]	12.07
n-Nonanoyl	17.27	17.48	1009.8	1017.0[d]	12.10
n-Dodecanoyl	25.03	24.64	1184.5	1183.8[d]	12.12

[a] A.S.T.M. procedure using modified Ostwald tubes. [b] Souders' viscosity function, I (M. Souders, Jr., THIS JOURNAL, 60, 154 (1938); A. N. Planovskii and V. V. Kafarov, *Khimicheskaya Prom.*, No. 8, 19 (1944)(*C.A.*, 40, 2048 (1946))). [c] According to R. T. Lagemann (THIS JOURNAL, 67, 498 (1945)), I/M should be approximately 12. [d] In calculating I from group and structural values, −24 (recommended by Souders for 5-membered carbon rings) was used for the 5-membered tetrahydrofuran ring.

specific gravity bottle (fitted with a ground glass thermometer and cap), respectively. Viscosities (Table II) were determined by A.S.T.M. procedure,[9] using modified Ostwald tubes and a constant temperature bath[10] capable of maintaining the temperature within ± 0.02° at 20°.

Determination of Boiling Water Stability.—A mixture of 5 g. of ester and 100 ml. of distilled water was refluxed for twenty-four hours and allowed to cool. Approximately 200 ml. of acetone and several drops of cresol red–thymol blue mixed indicator[11] were added, and the resulting solution was titrated with 0.1 *N* sodium hydroxide. This method appeared to have certain advantages (one-phase system and less fading of end-point) over the similar method of Fordyce and Meyer.[12] Water solubilities were determined by a method similar to that of Fordyce and Meyer[12], using Sudan Red No. III dye.

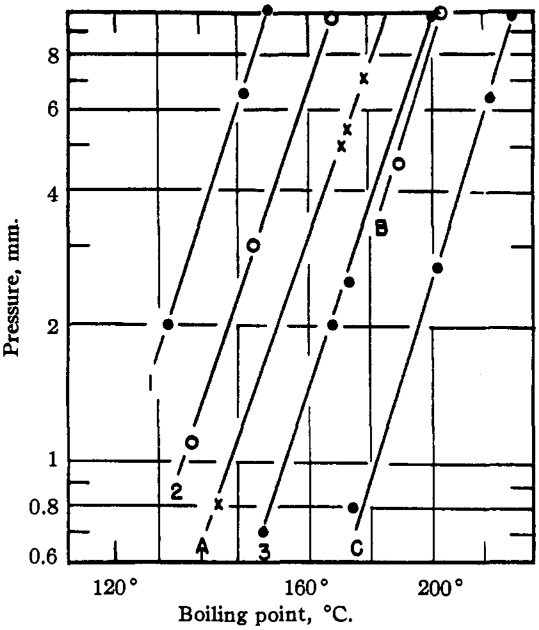

Fig. 1.—Boiling points at various pressures of acyl derivatives of *n*-butyl and tetrahydrofurfuryl lactates: acyl derivatives of *n*-butyl lactate: 1, heptanoate; 2, nonanoate; 3, dodecanoate; acyl derivatives of tetrahydrofurfuryl lactate: A, heptanoate; B, nonanoate; C, dodecanoate.

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(9) A. S. T. M., D445 and Committee D-2 Report, 1936; M. R. Cannon and M. R. Fenske, *Oil Gas J.*, 33, 52 (1935); *ibid.*, 34, 45 (1936).

(10) M. L. Fein, *Chem. Analyst*, 34, 94 (Nov. 1945).

(11) A. Kleinzeller and A. R. Trim, *Analyst*, 69, 241 (1944).

(12) C. R. Fordyce and L. W. A. Meyer, *Ind. Eng. Chem.*, 32, 1053 (1940).

TABLE III

PROPERTIES OF ACYL DERIVATIVES OF *n*-BUTYL LACTATE AND TETRAHYDROFURFURYL LACTATE

Acyl deriv.	Vapor press. at 120°., mm.[a]	Solubility in water, g./liter, less than	Boiling water stability[b]	Compatibility[c] Ethyl cellulose, 1:1[d]	Cellulose acetate, 4:1[d]	Cellulose acetate butyrate, 4:1[d]	Polyvinyl chloride, 1:1[d]
		Butyl lactate					
n-Heptanoyl	1.7	0.07	..	C	I	C	C
n-Nonanoyl	0.7	.03	12.3	C	I	CI	C
n-Dodecanoyl	.15	.04	7.8	C	I	CI	C
		Tetrahydrofurfuryl lactate					
n-Heptanoyl	.34	.16	..	C	I	C	C
n-Nonanoyl	.22	.04	8.9	C	I	C	C
n-Dodecanoyl	< .1	.04	5.0	C	I	C	C

[a] Estimated from Fig. 1 by extrapolation; vapor pressure of butyl phthalate at 120° = 0.26 mm. (ref. 7). [b] Volume 0.1 *N* sodium hydroxide (ml.) to neutralize sample (5 g. in 100 ml. of water) that had been refluxed for twenty-four hours; values obtained by this method with methyl and butyl phthalate, respectively, were 1.0 and 0.2 ml. [c] C, compatible; I, incompatible; and CI borderline compatibility (commercially available plastics used for tests). [d] Ratio of polymer to plasticizer.

Compatibilities.—The acylated lactic ester and certain commercially available resins (Table III) were dissolved in a suitable solvent (acetone for ethyl cellulose, cellulose acetate, and cellulose acetate butyrate and dioxane for polyvinyl chloride). The solution was placed in a Petri dish and covered with a watch glass, and the solvent was allowed to evaporate. The resulting film was examined to determine compatibility. The esters were recorded as being compatible (Table III) only when transparent and dry films were obtained.

Correlation of Properties with Number of Carbon Atoms

The experimental data of the present work (Tables I to III) characterize many members of two homologous series (α-carbobutoxyethyl *n*-alkanoates, I, and α-carbotetrahydrofurfuryloxyethyl *n*-alkanoates, II), but several members of both series were not prepared or examined. To provide methods for estimating the properties of the missing members and to facilitate comparison of the *n*-alkanoyllactic esters (Table I) with certain previously-described homologous series, relationships between physical properties and number of carbon atoms (or molecular weight) of the homologous *n*-alkanoyl lactates were developed. Instead of plotting the physical constants as such against the number of carbon atoms and obtaining curved lines, certain functions of the properties that gave straight lines were selected. In agreement with earlier work, it was found that the straight line relationships usually were unsatisfactory for the first two or three members of the homologous series.

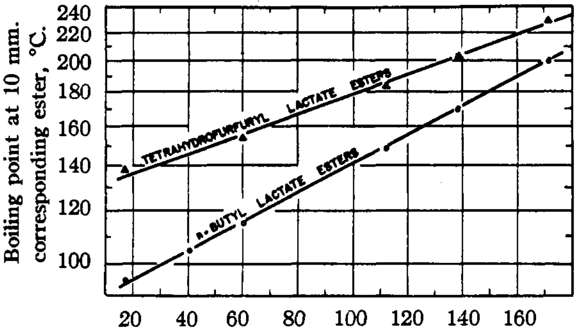

Fig. 2.—Relation of boiling points at 10 mm. of aliphatic acids and the corresponding acyl derivatives of tetrahydrofurfuryl and *n*-butyl lactates.

Boiling points at 10 mm. were taken from Fig. 1 and related[13] to the number of carbon atoms (x) and molecular weight (M) by equations 1, 2, 3 and 4 (T = b.p. at 10 mm., °K.). Observed boiling points vary 2° or less from the values calculated by equations 1 and 3. In the case of equations 2 and 4, the variation is 1° or less.

Butyl esters:
$$T^2 10^{-4} = 0.061M + 1.96 \quad (1)$$
$$T^2 10^{-4} = 0.85x + 5.8 \quad (2)$$

Tetrahydrofurfuryl esters:
$$T^2 10^{-4} = 0.061M + 3.42 \quad (3)$$
$$T^2 10^{-4} = 0.85x + 8.2 \quad (4)$$

Figure 2, constructed by plotting boiling points at 10 mm. of aliphatic acids[14] against the boiling points of the corresponding carbobutoxyethyl (I) and carbotetrahydrofurfuryloxyethyl esters (II) on semilog paper, also can be used to estimate boiling points of the missing homologous esters. Boiling points (°K.) of the acids ($H(CH_2)_{x-1}COOH$) at 10 mm.[14] were used also to derive equations 5 and 6. Boiling points reported[14] in the literature vary 1 degree or less from the values calculated by equations 5 and 6.

$$H(CH_2)_{x-1}COOH: \ T^2 10^{-4} = 1.28x + 6.06 \quad (x = 1 \text{ to } 7, \text{ inclusive}) \quad (5)$$
$$T^2 10^{-4} = 0.93x + 8.60 \ (x = 8 \text{ to } 18, \text{ inclusive}) \quad (6)$$

The boiling points of the lactic esters (1 and II) are compared with those of other homologous

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[13] This method for relating the boiling point at 760 mm. and number of carbon atoms or molecular weight has been used by E. Boggia-Lera, *Gazz. chim. ital.*, **29**, 441 (1899); A. H. W. Aten, *J. Chem. Phys.*, **5**, 260 (1937); and F. Klages, *Ber.*, **76**, 788 (1943).

[14] Boiling points were estimated from the vapor pressure data of W. O. Pool and A. W. Ralston (*Ind. Eng. Chem.*, **34**, 1104 (1942)) by the use of Cox charts.[8]

series in Fig. 3, in which boiling points at 10-mm. pressure (°C.) are plotted against the square root of the number of carbon atoms. The n-butyl (I) and tetrahydrofurfuryl esters (II) boil lower and higher, respectively, than the diethyl esters[15] ($(CH_2)_n(COOC_2H_5)_2$) having the same number of carbon atoms. The lateral methyl group of the lactic esters and the tetrahydrofurfuryl group are relatively ineffective and effective, respectively, in raising the boiling point.[16] Both homologous series of lactic esters boil higher than the methyl alkanoates ($H(CH_2)_nCOOCH_3$) having an equal number of carbon atoms (with the exception of some of the higher n-alkanoyl derivatives of n-butyl lactate). The lines in Fig. 3 representing the homologous series having two or more functional groups have less slope than the n-paraffin[17] and methyl alkanoate lines.

The boiling points at 760 mm. of the n-butyl lactate derivatives (I) were calculated (Table IV) by the methods of Kinney,[16a] Burnop[16b] and Klages.[18] Boiling points at 760 mm. computed by Kinney's method[16a] were used to estimate[19] boiling points at 10 mm. Some of the calculated boiling points at 10 mm. (Table IV) are in close agreement with the experimental values.

The densities (d^{20}_4) and refractive indices (n^{20}_D) of the n-alkanoyl derivatives of n-butyl lactate (I) are given in Table I. They are related to the number of carbon atoms (x) by equations 7 and 8. Observed densities differ 0.0009 or less from the values calculated by equation 7. With the exception of the first member of the series, the observed refractive indices vary 0.0009 or less from the values calculated by equation 8.

$$\text{Butyl esters: } x/d = 1.164x - 1.509 \qquad (7)$$

$$x/n = 0.6845x + 0.2082 \qquad (8)$$

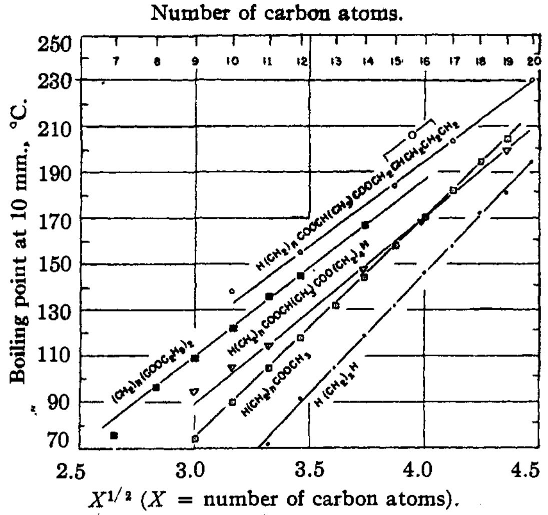

Fig. 3.—Relation between boiling point at 10 mm. and carbon atoms (X) of homologous compounds.

TABLE IV

CALCULATED BOILING POINTS OF THE ACYL DERIVATIVES OF n-BUTYL LACTATE

No. of carbon atoms	9	10	11	14	19
Boiling point (760 mm.), °C.					
Kinney's method[a]	222	235	254	304	377
Burnop's method[b]	232	249	266	312	375
Klages' method[c]	230	250	269	323	404
Boiling point (10 mm.), °C.					
Found[d]	95	105	115	148	201
Calcd.[e]	97	107	122	161	219

[a] Ref. 16a. [b] Ref. 16b. [c] Ref. 18. [d] From Fig. 1 or similar charts. [e] On the assumption[19] that esters have the temperature–vapor pressure relationship of group 3 compounds, using the equation: b. p. 10 mm. = (0.786) (b. p. 760 mm.) − 77.6.

Similarly, equations 9 and 10 may be used to calculate d^{20}_4 and n^{20}_D of the tetrahydrofurfuryl esters (II) from the number of carbon atoms (x). With the exception of the first member of the series, the average deviation of the observed densities from those calculated by equation 9 was 0.002. The average deviation for the refractive indices (equation 10) was 0.0004.

$$\text{Tetrahydrofurfuryl esters: } x/d = 1.142x - 2.563 \qquad (9)$$

$$x/n = 0.6845x + 0.0892 \qquad (10)$$

The butyl acyloxypropionates (I) and tetrahydrofurfuryl acyloxypropionates (II) are similar to other homologous series,[20] in that linear relationships (Fig. 4) exist (excluding the first few members) between the logarithm of viscosity and the number of carbon atoms (Table II). The n-butyl (I) and tetrahydrofurfuryl esters (II) had lower and higher viscosities, respectively, than the diethyl esters[21] ($(CH_2)_n(COOC_2H_5)_2$) having an

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(15) Boiling points of the diethyl esters ($(CH_2)_n(COOC_2H_5)_2$) at 10-mm. were estimated from boiling point data of A. I. Vogel, *J. Chem. Soc.*, 333 (1934).

(16) These conclusions are in agreement with the boiling points of somewhat related compounds at 760 mm. (a) C. R. Kinney, THIS JOURNAL, **60**, 3032 (1938); Lange's "Handbook of Chemistry," 5th ed., 1944, Handbook Publishers, Inc., Sandusky, Ohio, p. 853, (b) V. C. E. Burnop, *J. Chem. Soc.*, 826 (1938).

(17) Boiling points of $H(CH_2)_nCOOCH_3$ and $H(CH_2)_nH$ at 10 mm. may be calculated by the equations: (esters) $T^2 10^{-4} = 1.09x + 2.23$; ($n$-paraffins) $T^2 10^{-4} = 1.19x - 1.0$ (x = 1 to 12, inclusive), and $T^2 10^{-4} = 1.07x + 0.35$ (x = 12 to 20, inclusive); where T = °K., and x = number of carbon atoms. Boiling point data used in deriving these equations were taken from (esters) P. M. Althouse and H. O. Triebold, *Ind. Eng. Chem., Anal. Ed.*, **16**, 605 (1944); F. A. Norris and D. E. Terry, *Oil & Soap*, **22**, 41 (1945); A. W. Weitkamp, THIS JOURNAL, **67**, 447 (1945); "International Critical Tables," Vol. III, p. 217, and (n-paraffins) C. B. Willingham, W. J. Taylor, J. M. Pignocco and F. D. Rossini, *J. Research Natl. Bur. Standards*, **35** (31) 219 (1945); M. P. Doss, "Physical Constants of the Principal Hydrocarbons," 4th ed., The Texas Co., New York, N. Y., 1943. Boiling points reported in the literature differ 2° or less from the values calculated by the n-paraffin equations.

(18) F. Klages, *Ber.*, **76**, 788 (1943).

(19) In estimating the boiling points at 10 mm., it was assumed (following the suggestion of C. Bordenca, *Ind. Eng. Chem., Anal. Ed.* **18**, 99 (1946), and S. B. Lippincott and M. M. Lyman, *Ind. Eng. Chem.*, **38**, 320 (1946)) that esters fall into Group 3 with respect to temperature–vapor pressure properties.

(20) A. E. Dunstan and F. B. Thole, *J. Chem. Soc.*, **103**, 127 (1913).

(21) Viscosity data of the diethyl esters were taken from R. Cedar, *Ann. Univ. Fennicae Aboensis Series*, **A2**, No. 4, 14 pp. (1926) (*C. A.*, **22**, 3137 (1928)).

equal number of carbon atoms. The lactic esters (*I* and *II*) were more viscous than the corresponding ethyl alkanoates[22] and *n*-paraffins[23] (Fig. 4).

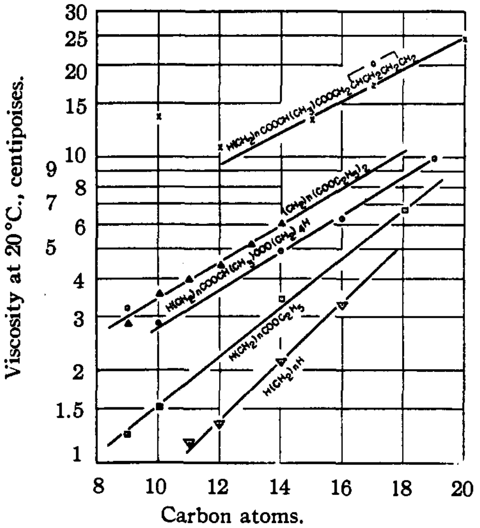

Fig. 4.—Relation between viscosity and carbon atoms of homologous compounds.

The viscosities of the *n*-butyl lactate (I) and tetrahydrofurfuryl lactate (II) derivatives are plotted in Fig. 5 as a function of the boiling points at 10 mm. The viscosities of the higher *n*-alkanoyl derivatives of butyl lactate were similar to those of the diethyl esters ($(CH_2)_n(COOC_2H_5)_2$) and *n*-paraffins of the same boiling point, but the tetrahydrofurfuryl esters (II) had viscosities considerably higher than those of the corresponding linear compounds (Fig. 5).

The values of Souders' viscosity function[24] were calculated (Table II) for the esters (I and II) of lactic acid; the I values calculated from the formula

$$I = \frac{M}{d}\log_{10}(\log_{10} n) + 2.9$$

where M = molecular weight, d = density and n = viscosity agreed moderately well with those estimated from the atomic and group increments.[24] In agreement with Lagemann,[25] the ratios between the I values and molecular refraction were approximately 12.

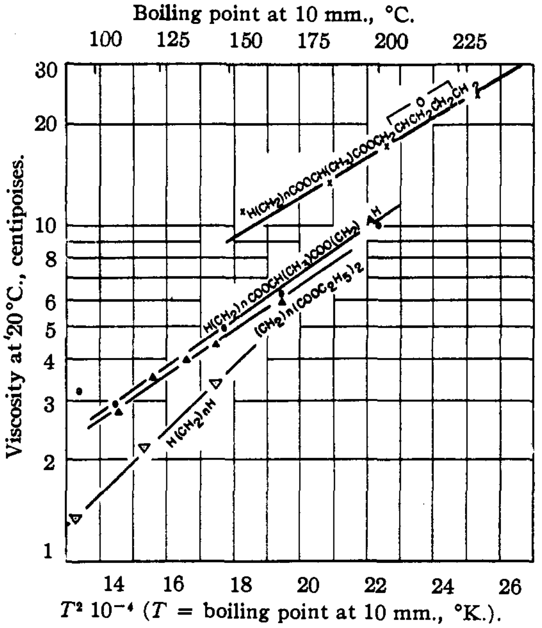

Fig. 5.—Relation between boiling points at 10 mm. and viscosity of homologous compounds.

Several of the compounds of Table I had vapor pressures (Table III and Fig. 1) sufficiently low for use as plasticizers.[26] The suggestion that some of the esters of the present work should be suitable as plasticizers is supported by their relatively low viscosity, moderate stability in the presence of boiling water, and compatibility with certain commercially important plastics (Table III).

Acknowledgment.—W. P. Ratchford demonstrated that the alembic type still is suitable for determining boiling points at relatively low pressures and verified the boiling points of Figure 1. The authors are grateful for this major contribution.

Summary

Several homologous *n*-alkanoyl derivatives of *n*-butyl lactate ($H(CH_2)_nCOOCH(CH_3)COO(CH_2)_4H$) and of tetrahydrofurfuryl lactate ($H(CH_2)_nCOOCH(CH_3)COOCH_2\underbrace{CHCH_2CH_2CH_2}_{O}$) were prepared in high yields by treating the corresponding lactic esters with acid chlorides or anhydrides. Relationships between physical properties and molecular weight are given, from which the density, refractive index, viscosity and boiling point at 10 mm. of the missing members can be calculated.

Philadelphia 18, Pa. Received April 25, 1947

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[22] Viscosities of ethyl *n*-alkanoates were obtained from A. E. Dunstan, F. B. Thole and P. Benson, *J. Chem. Soc.*, **105**, 782 (1914).

[23] "Landolt-Bornstein Tabellen," 3rd Supplement, p. 162, 1935, Julius Springer, Berlin; E. C. Bingham and H. J. Fornwalt, *J. Rheol.*, 1, 372 (1930).

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[25] R. T. Lagemann, This Journal, **67**, 498 (1945).

[26] This statement is based on a comparison of the esters with *n*-butyl phthalate, a widely used plasticizer having a vapor pressure of 10 mm. at 195°.[7]