

disappear and the solution would show the general absorption as has been found for D-glucose in neutral solution (Fig. 1). Pertinent experiments have revealed the remarkable fact that this is only true if the solution, after treatment with sodium bicarbonate, becomes slightly alkaline. In slightly acidic solution, however, the assumed open-chain form of the sugar persists for a comparatively long period of time. It is seen in Fig. 4 (C) that eight days after "neutralization" the slightly acidic solution still exhibited the characteristic absorption at 2840 Å., although the peak was not as sharp as that measured after the first day (A). It took altogether sixteen days until the absorption band completely disappeared in the slightly acidic solution (D). On the other hand, addition of two drops of dilute alkali to the one-day old solution resulted in the immediate disappearance (B) of the peak at 2840 Å.

In view of the present theory of mutarotation<sup>11</sup> these facts can not be interpreted readily. Further investigations are necessary before the significance of our observations to the true mechanism of mutarotation can be evaluated. We have already made initial attempts to isolate the postulated open-chain form of D-glucose by using super-

(11) T. M. Lowry, "Rapports sur les Hydrates de Carbon," XI<sup>ème</sup> Conférence de l'Union Internationale de Chimie, Liège, 1930.

concentrated hydrochloric acid solutions. So far we have not been able to isolate the free sugar in crystalline form, but we have succeeded in obtaining an amorphous acetate as the single product which, unlike the  $\alpha$ - and  $\beta$ -pentaacetate, in chloroform solution shows strong selective absorption at the wave length of 2840 Å. The investigation is being continued.

### Summary

It has been found that D-glucose and L-arabinose in 50.5% sulfuric acid solutions show strong absorption at wave length 2840 Å. which is characteristic for carbonyl-containing compounds. After neutralization with sodium bicarbonate the slightly acidic solution still exhibits this band which, however, instantaneously disappears on rendering the solution barely alkaline. A possible mechanism is suggested for explanation of the behavior in strong acids of sugars with ring structures by assuming the formation of molecular compounds between the acid molecules and the open-chain forms of the sugars.

From D-glucose in superconcentrated hydrochloric acid solution an amorphous acetate has been obtained which compound, unlike the  $\alpha$ - and  $\beta$ -pentaacetate, in chloroform solution exhibits strong absorption at 2840 Å.

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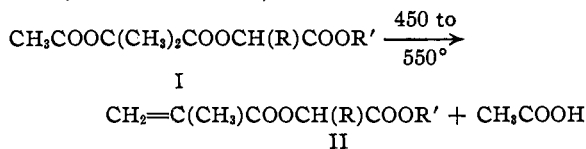
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[CONTRIBUTION FROM THE EASTERN REGIONAL RESEARCH LABORATORY<sup>1</sup>]

## Preparation of $\alpha$ -Carbalkoxyalkyl Methacrylates by Pyrolysis of the Corresponding $\alpha$ -Acetoxyisobutyrate

BY E. M. FILACHIONE, M. L. FEIN, J. H. LENGEL AND C. H. FISHER

In an earlier paper<sup>2</sup> it was demonstrated that  $\alpha$ -carbalkoxyalkyl methacrylates<sup>3</sup> (II) can be made in satisfactory yields by pyrolyzing the corresponding  $\alpha$ -acetoxyisobutyrate (I, R = Me or H; R' = Me or Et).



The present paper records results obtained in a further study of the pyrolysis method of preparing methacrylates (II). The additional esters made pyrolytically are the methacrylates of allyl glycolate and of allyl, methallyl, tetrahydrofurfuryl, benzyl and  $\beta$ -chloroethoxyethyl lactates.

(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) E. M. Filachione, J. H. Lengel and C. H. Fisher, *This Journal*, **66**, 330 (1946).

(3) C. E. Rehberg, M. B. Dixon and C. H. Fisher, *ibid.*, **67**, 208 (1945); I. E. Muskat and F. Strain, U. S. Patent 2,384,119, Sept. 4, 1945.

The  $\alpha$ -acetoxyisobutyrate (I) used in the pyrolysis experiments were prepared in high yields by treating allyl glycolate and several lactic esters (allyl, methallyl, tetrahydrofurfuryl, benzyl and  $\beta$ -chloroethoxyethyl) with  $\alpha$ -acetoxyisobutyryl chloride. The resulting acetoxyisobutyrate (Table I) were decomposed by passage through a Pyrex glass tube heated at about 450° (Table II).

Although the esters (I) pyrolyzed had three esterified carboxyl groups, decomposition occurred preferentially at the acetate group, the principal products being acetic acid and the corresponding methacrylates (II, yields, 65–85%). On the basis of these and earlier<sup>2</sup> findings, it is concluded that the pyrolysis method is generally useful for the preparation of methacrylates (II) of alkyl glycolates and lactates. Presumably the method is useful also for preparing esters (II) in which R is ethyl or higher, but less suitable for esters (II) in which R' is a secondary or tertiary alkyl group. It is anticipated that only relatively low yields of carbalkoxyisopropyl methacrylates ( $\text{CH}_2=\text{C}(\text{CH}_3)\text{COOC}(\text{CH}_3)_2\text{COOR}$ ) would be obtained in the pyrolysis of  $\text{CH}_3\text{COOC}(\text{CH}_3)_2\text{COOC}(\text{CH}_3)_2\text{COOR}$ ,

TABLE I  
 PREPARATION AND PROPERTIES OF ACYLATED HYDROXY ESTERS

$\alpha$ -Acetoxyisobutyrate of	Yield, % of theoretical	B. p.		$d_{20}^4$	$n_{20}^D$	Mol. refraction		Sapon. equiv.		Carbon, %		Hydrogen, %	
		$^{\circ}\text{C}$ .	Mm.			Calcd.	Found	Calcd.	Found	Calcd.	Found	Calcd.	Found
Allyl lactate	89	82	0.2	1.0864	1.4357	62.11	62.11	86.1	86.8	55.80	55.41	7.02	7.04
Methallyl lactate	81	82-85	0.1	1.0713	1.4381	66.73	66.73	90.8	91.2	57.34	57.59 <sup>a</sup>	..	..
Allyl glycolate	79	84-86	0.25	1.1224	1.4407	57.49	57.43	81.41	81.55	54.09	54.27 <sup>a</sup>	..	..
Tetrahydrofurfuryl lactate	87	145	1.5	1.1347	1.4475	71.26	71.29	....	....	55.62	55.59	7.33	7.32
Benzyl lactate	86	136	0.6	1.1285	1.4812	77.45	77.77	102.8	102.7	62.32	62.05	6.54	6.53
$\beta$ -Chloroethoxyethyl lactate	75	145	0.2	1.1798	1.4478	73.71	73.66	....	....	48.07	47.91	6.52	6.59

<sup>a</sup> By wet oxidation (D. D. Van Slyke and J. Folch, *J. Biol. Chem.*, 136, 509 (1940)).

 TABLE II  
 PYROLYSIS OF ACETYLATED HYDROXY ESTERS

Ester pyrolyzed, g. Acetoxyisobutyrate of	Temp., $^{\circ}\text{C}$ .	Feed rate, g./min.	Contact time, sec.	Ester decomposed, %	Methacrylate		Yields, <sup>a</sup> mole per mole of ester destroyed			
					Boiling range, $^{\circ}\text{C}$ .	Press., mm.	Methacrylate	Acetic acid Titr.	Dist.	Acetone
Allyl lactate, 147	446	2.0	10.1	79	54-62	0.45	0.82	0.92	0.76	..
Methallyl lactate, 346	450	2.8	7.5	63	53-60	.2	.85	.97	.84	0.07
Allyl glycolate, 130	450	2.4	7.9	69	51-62	.35	.78	.96	.84	..
Tetrahydrofurfuryl lactate, 288	450	2.3	10.1	68	83-91 <sup>b</sup>	.1	.65	.93	.80	.08
Benzyl lactate, 159	450	2.4	9.9	65	97-104 <sup>c</sup>	.2	.71	.92	.72	.07
$\beta$ -Chloroethoxyethyl lactate, 114	450	2.7	9.5	66	104-116 <sup>d</sup>	.25	.79	.88	.66	.04

<sup>a</sup> Methacrylate yield based on that obtained in first distillation; acetic acid yield based on that obtained in redistillation. <sup>b</sup> Much polymerization occurred during distillation. <sup>c</sup> Hydroquinone distilled with the monomer. <sup>d</sup> Phenyl- $\beta$ -naphthylamine used as inhibitor.

 TABLE III  
 PROPERTIES OF MONOMERIC  $\alpha$ -CARBALKOXYALKYL METHACRYLATES

Methacrylate of	$^{\circ}\text{C}$ .	B. p. Mm.	$d_{20}^4$	$n_{20}^D$	Mol. refraction		Sapon. equiv.		C, %	
					Calcd.	Found	Calcd.	Found	Calcd.	Found <sup>a</sup>
Allyl lactate	52-53	0.15	1.0352	1.4452	50.75	50.98	99.10	99.15	60.59	60.63
Methallyl lactate	57-59	0.25-0.30	1.0160	1.4475	55.37	55.90	106.1	106.6	62.24	61.93
	66	0.55								
Allyl glycolate	73-74	1.0	1.0760	1.4513	46.14	46.12	92.09	92.66	58.69	58.39
	53-54	0.3								
Tetrahydrofurfuryl lactate	91-95	0.15-0.20	1.1015	1.4586	59.94	60.08	121.1	120.5	59.49	59.53
	109-110	0.7								
Benzyl lactate	92-95	0.1	1.0975	1.5002	66.09	66.53	124.1	118.8	67.72	67.68
	96-101	0.2								
$\beta$ -Chloroethoxyethyl lactate <sup>b</sup>	97-99	0.15	1.1528	1.4579	62.35	62.64	....	....	...	...

<sup>a</sup> By wet oxidation (D. D. Van Slyke and J. Folch, *J. Biol. Chem.*, 136, 509 (1940)). <sup>b</sup> Chlorine content, 13.36% (theoretical, 13.40%).

a more probable decomposition product<sup>4</sup> being  $\text{CH}_2=\text{C}(\text{CH}_3)\text{COOR}$ .

The methacrylates (Table III) were mass polymerized at 60 to 65 $^{\circ}$  in the presence of a small quantity (0.1 to 0.15%) of benzoyl peroxide. With the exception of the  $\beta$ -chloroethoxyethyl ester (a soft, flexible, amber polymer that became brittle at approximately -5 $^{\circ}$ ), all the polymeric methacrylates were hard at room temperature and virtually colorless. The polymeric benzyl and tetrahydrofurfuryl esters became soft at approximately

(4) This assumption is based on the pyrolysis behavior<sup>3</sup> of  $\text{CH}_2\text{COOCH}(\text{CH}_3)\text{COOCH}(\text{CH}_3)\text{COOCH}_3$ , which yields methyl acrylate as the principal product.

45 $^{\circ}$  and 35 $^{\circ}$ , respectively. Presumably the polymeric allyl and methallyl esters, insoluble and infusible, were cross-linked.

The authors are indebted to Frances J. Cooper, Mary J. Welsh and C. L. Ogg for the saponification equivalents and ultimate analyses and to W. P. Ratchford and E. J. Schaeffer for pyrolyzing the acetoxyisobutyrate.

### Experimental

**Allyl Glycolate.**—Water was distilled from a mixture of 210 g. of technical-grade glycolic acid (72.4% concentration) and 0.5 ml. of concentrated sulfuric acid for three hours at 100 $^{\circ}$  and 20 to 30 mm. pressure. The resulting

residue (123 g. of polyglycolic acid having an equivalent weight on titration of 64.75; *i. e.*, 117.5% by weight as glycolic acid) was semi-solid.

A mixture of 32.4 g. of the polyglycolic acid (equivalent to 0.5 mole of glycolic acid and containing 0.13 ml. of sulfuric acid) and 2.5 moles of allyl alcohol was heated in a bomb (glass liner) for six hours at 120–139°. Anhydrous sodium acetate (0.6 g.) was added to the reaction mixture and the mixture was distilled to separate allyl glycolate (66% yield; b. p. 60–72° (8 mm.);  $d_{20}^{20}$ , 1.0901;  $n_D^{20}$  1.4418;  $M_D^{20}$  28.09 (calculated, 28.00); saponification equiv., 122.5 (calculated, 116.1)). Muskat and Strain<sup>6</sup> prepared allyl glycolate but did not describe it.

**Allyl Lactate.**—This compound, previously described,<sup>6,7,8</sup> was prepared by the reaction of allyl alcohol with condensation products of glycerol and lactic acid. Water (116 g.) was distilled through a Vigreux column from a mixture of 81.7% edible-grade lactic acid (equivalent to 3 moles), 1 mole of glycerol and 0.75 ml. concentrated sulfuric acid for three hours. This was accomplished by heating the mixture on a water-bath while the system was at a pressure of 40–50 mm. The pale yellow, viscous residue (307 g.) had a saponification equivalent, including free acidity, of 101.4 (glyceryl trilactate, 102.7). A mixture of 50.5 g. of the glycerol-lactic acid product (equivalent to 0.5 mole of lactic acid) and 2.5 moles of allyl alcohol was heated in a bomb (glass liner) for two hours at 120–30°. The reaction mixture was treated with 0.5 g. of anhydrous sodium acetate and distilled in vacuum. Allyl lactate, b. p. 60–61° at 7 mm.,  $n_D^{20}$  1.4363, was isolated in 73% yield.

**Benzyl Lactate.**—A mixture of 1 mole of methyl lactate, 4 moles of benzyl alcohol and 1 g. of aluminum isopropoxide was heated under a Vigreux column fitted with a variable take-off distilling head, methanol being distilled as formed (temperature of the reaction mixture increased gradually from 170 to 192°, and 36 ml. of distillate was collected during four to five hours). The reaction mixture was then distilled under reduced pressure. The yield of benzyl lactate, b. p. 103–104° at 1.3 mm., was 82%. The refractive indices,  $n_D^{20}$ , of two different preparations were 1.5143 and 1.5155. This compound has been made by the alcoholysis of ethyl lactate<sup>9</sup> and by the interaction of benzyl chloride and sodium lactate.<sup>10</sup>

**Methallyl lactate** was prepared by direct esterification of lactic acid with methallyl alcohol, some modifications of the previously reported method<sup>6</sup> being incorporated. A mixture of 110 g. of 81.7% of edible lactic acid, containing 1.0 mole of available lactic acid and 216 g. (3.0 moles) of methallyl alcohol, was refluxed and the vapors were led through a Vigreux column to a Barrett-type Dean-and-Stark moisture trap. The receiver of the moisture trap was jacketed and cooled by circulating cold water. After approximately seven hours removal of water virtually ceased, and 37 ml. of aqueous layer was collected. The reaction mixture was then distilled. There was obtained 86 g., or 60% of the theoretical amount of methallyl lactate, b. p. 85–90° at 15 mm.,  $n_D^{20}$  1.4390.

**Tetrahydrofurfuryl Lactate** was prepared essentially as described previously<sup>11,12</sup> by alcoholysis of methyl lactate.

(5) I. E. Muskat and F. Strain, U. S. Patent 2,370,574, Feb. 27, 1945.

(6) C. H. Fisher, C. E. Rehberg and Lee T. Smith, *THIS JOURNAL*, **65**, 763 (1943); C. E. Rehberg and C. H. Fisher, U. S. Patent 2,367,798.

(7) C. E. Rehberg and C. H. Fisher, *J. Org. Chem.*, **12**, 226 (1947).

(8) I. E. Muskat and F. Strain, U. S. Patent 2,364,126, Sept. 4, 1945.

(9) R. Burns, D. T. Jones and P. D. Ritchie, *J. Chem. Soc.*, 400 (1935).

(10) M. Gomberg and C. C. Buehler, *THIS JOURNAL*, **42**, 2059 (1920).

(11) H. V. Claborn, U. S. Patent 2,222,363, Nov. 19, 1940; U. S. Patent 2,229,997, Jan. 28, 1941.

(12) M. L. Fein, W. P. Ratchford and C. H. Fisher, *THIS JOURNAL*, **66**, 1201 (1944).

The alcoholysis, however, was carried out in the absence of a catalyst. By refluxing a mixture of 10 moles of methyl lactate and 30 moles of tetrahydrofurfuryl alcohol with the continuous removal of methanol, an 85% yield of tetrahydrofurfuryl lactate was obtained; b. p. 84–86° at 0.1 mm.,  $n_D^{20}$  1.4571,  $d_{20}^{20}$ , 1.1360.

**$\beta$ -Chloroethoxyethyl Lactate.**—Direct esterification of  $\beta$ -chloroethoxyethanol (diglycol chlorohydrin) with lactic acid, as previously described,<sup>13</sup> was used to obtain this ester, b. p. 91° at approximately 0.1 mm.,  $n_D^{20}$  1.4567.

**$\alpha$ -Carbalkoxyalkyl  $\alpha$ -Acetoxyisobutyrate.**—Acetoxyisobutyryl chloride was prepared and used to acylate the hydroxy esters as previously described.<sup>2</sup> Physical properties of the acyl derivatives (obtained in 80 to 90% yields) are recorded in Table I.

**Pyrolysis.**—In general the previous procedure was followed.<sup>2</sup> The acylated hydroxy esters were pumped<sup>14</sup> to the top of a vertical Pyrex glass tube (25 mm. diameter) heated electrically over a length of 33 cm. The thermocouple was located 10 cm. from the bottom of the heated zone. The pyrolysis tube was packed with short lengths of Pyrex glass tubing, and the free space of the heated zone was 78 ml.

The pyrolyzates, containing hydroquinone, were distilled in a 56-cm. Vigreux column in an atmosphere of carbon dioxide (Table II). The acetic acid fraction was collected at water pump pressure (in a solid carbon dioxide trap), whereas the methacrylates and undecomposed esters were isolated at pressures below 1 mm.

The constants of the methacrylates (Table III) were obtained on redistilled fractions. The methacrylates of tetrahydrofurfuryl lactate, benzyl lactate and chloroethoxyethyl lactate partly polymerized during distillation, resulting in losses.

**Solubilities of the Polymeric Carbalkoxyethyl Methacrylates.**—The polymeric methacrylates (0.1 g.) were allowed to stand at room temperature with approximately 1 ml. of solvent. The polymethacrylate of tetrahydrofurfuryl lactate swelled in toluene, benzene, ethylene dichloride, nitromethane, dioxane, acetone, methyl lactate and ethyl acetate. The polymethacrylate of benzyl lactate was soluble in nitromethane and partly soluble in toluene, benzene, ethylene dichloride, dioxane, acetone, methyl lactate and ethyl acetate. The polymethacrylate of  $\beta$ -chloroethoxyethyl lactate was soluble in toluene, benzene, ethylene chloride, nitromethane, dioxane, acetone, methyl lactate, and ethyl acetate. The polymers listed above were insoluble in heptane and ethanol, but dissolved slowly at room temperature in 10% sodium hydroxide. The polymers after swelling by acetone dissolved much more rapidly in the sodium hydroxide, presumably by saponification of the esterified lactic acid carboxyl group.

## Summary

The method of producing  $\alpha$ -carbalkoxyalkyl methacrylates ( $\text{CH}_2=\text{C}(\text{CH}_3)\text{COOCHR}\text{COOR}'$ ; R = H or Me) by pyrolysis of the corresponding  $\alpha$ -acetoxyisobutyrate ( $\text{CH}_3\text{COOC}(\text{CH}_3)_2\text{COOCHR}\text{COOR}'$ ) was used satisfactorily to prepare the methacrylates of allyl glycolate and of allyl, methallyl, benzyl, tetrahydrofurfuryl, and  $\beta$ -chloroethoxyethyl lactates. It is concluded that the pyrolytic method is generally useful for making  $\alpha$ -carbalkoxyalkyl methacrylates.

The methacrylates polymerized readily when heated in the presence of benzoyl peroxide. The polymeric methacrylate of  $\beta$ -chloroethoxyethyl lactate was amber and flexible, the allyl and

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

(14) B. B. Corson and W. J. Cerveny, *Ind. Eng. Chem., Anal. Ed.*, **14**, 899 (1942).

methallyl polymers were hard, colorless, insoluble and infusible and the benzyl and tetra-

hydrofurfuryl polymers were hard and colorless. PHILADELPHIA 18, PA. RECEIVED AUGUST 8, 1947

[CONTRIBUTION FROM THE SCHOOL OF CHEMISTRY AND PHYSICS, THE PENNSYLVANIA STATE COLLEGE]

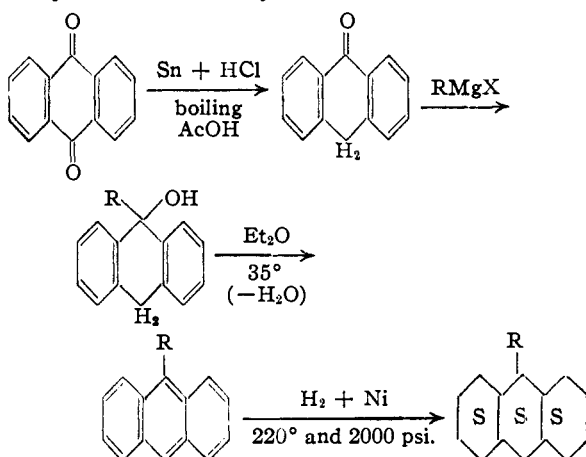
## Higher Hydrocarbons.<sup>1</sup> V.<sup>2</sup> Alkyl Anthracenes and Alkyl Phenanthrenes

BY ROBERT W. SCHIESSLER, ANTON W. RYTINA AND FRANK C. WHITMORE<sup>2a</sup>

As part of our studies of the synthesis, purification and properties of high molecular weight hydrocarbons, the preparations of several alkyl-anthracenes and -phenanthrenes, together with the corresponding perhydro derivatives, have been completed. They are 9-*n*-butylanthracene, 9-*n*-butylperhydroanthracene, 9-*n*-dodecylanthracene, 9-*n*-dodecylperhydroanthracene, 9-*n*-dodecylphenanthrene and 9-*n*-dodecylperhydrophenanthrene.

The alkyl anthracenes were synthesized by a modification of the method of Sieglitz and Marx.<sup>3</sup> The method consists of the addition of anthrone to alkylmagnesium halides, followed by dehydration of the resultant tertiary alcohol to the desired alkyl anthracene:

### Synthesis of 9-Alkylanthracenes.—



9-*n*-Dodecylphenanthrene was prepared by the addition of laurionitrile<sup>2,4</sup> to 9-phenanthrylmagnesium bromide<sup>5</sup> followed by reduction of the resulting 9-phenanthryl hendecyl ketone to the hydrocarbon by the modified Wolff-Kishner method.<sup>6</sup>

(1) American Petroleum Institute Research Project No. 42; Advisory Committee: L. C. Beard, Jr. (Chairman), J. R. Bates, L. M. Henderson, R. G. Larsen, R. F. Marchner, L. A. Mikeska, J. H. Ramser.

(2) Whitmore, Sutherland and Cosby, *THIS JOURNAL*, **64**, 1360 (1942); Whitmore, Cosby, Sloatman and Clarke, *ibid.*, **64**, 1810 (1942); Whitmore, Herr, Clarke, Rowland and Schiessler, *ibid.*, **67**, 2059 (1945); Whitmore, Schiessler, Rowland and Cosby, *ibid.*, **69**, 235 (1947).

(2a) Deceased, June 24, 1947.

(3) Sieglitz and Marx, *Ber.*, **55**, 1619 (1923).

(4) Ralston, Harwood and Pool, *THIS JOURNAL*, **59**, 986 (1937).

(5) Bachmann, *ibid.*, **56**, 1365 (1934).

(6) Whitmore, Herr, Clarke, Rowland and Schiessler, *ibid.*, **67**, 2059 (1945).

The perhydro derivatives of the hydrocarbons were prepared by complete hydrogenation over nickel catalyst<sup>7</sup> at 100–230° and 2300 p.s.i. hydrogen pressure.

It must be emphasized that the perhydro derivatives reported are mixtures of geometric isomers, and the properties given were determined on these mixtures of unknown composition. The difficulty of separating and identifying the various *cis-trans* structures precludes following any other course for our work.

Several of the important physical properties of the hydrocarbons are listed in Table I. The methods of determining the properties and their precision were discussed in the first paper of the series.<sup>2</sup> Numerous other properties have been determined and will be reported in the appropriate journals.

### Experimental

**Intermediates.**—In order to simplify the purification of the final hydrocarbons, great care has been taken with the purification of intermediates. Where possible, distillation of intermediates through fractionating columns of 35–40 theoretical plates was carried out. The boiling points of the intermediates are uncorrected. The properties listed are for the constant boiling, constant refractive index fractions.

(a) *n*-Butyl Bromide.—Edwal Laboratories *n*-butyl bromide was carefully fractionated: b. p. 100° (733 mm.);  $n_{20}^D$  1.4392.

(b) Lauryl bromide<sup>8</sup> was prepared by passing hydrogen bromide (from the direct combination of bromine and hydrogen) into Eastman Kodak Company technical lauryl alcohol at 120°. After washing twice with half its volume of cold (0–5°), concd. sulfuric acid and then with an equal volume of 95% methanol, the crude lauryl bromide was dried over anhydrous potassium carbonate and fractionally distilled through a 35–40 plate all glass column. The yield of pure bromide was 50% from this crude alcohol: b. p. 122° (4 mm.);  $n_{20}^D$  1.4586.

(c) Anthrone.—Eastman Kodak Company anthraquinone (highest purity) was reduced to anthrone in 60% yield,<sup>9</sup> m. p. 156–157°.

(d) 9-Bromophenanthrene.—Reilly Tar & Chemical Co. 90% phenanthrene was purified by the method of Cohen and Carmier<sup>10</sup>; m. p. 99.0–99.5°. A total of 508 g. of 9-bromophenanthrene was prepared in 55% yield following Austin<sup>11</sup>; m. p. 65–66°.

(7) A kieselguhr supported nickel supplied by the Universal Oil Products Co., Chicago, Ill.

(8) Further experience has shown that emulsion difficulty at the sulfuric acid wash step can be obviated by a preliminary simple distillation *in vacuo* of the Eastman Kodak Co. Technical Lauryl Alcohol. A considerable residue is eliminated.

(9) "Organic Syntheses," John Wiley and Sons, New York, N. Y., 1944, Coll. Vol. I, p. 60 (2nd edition).

(10) Cohen and Cormier, *THIS JOURNAL*, **52**, 4363 (1930).

(11) Austin, *J. Chem. Soc.*, **93**, 1762 (1908).