fraction, b.p. 133°/16 mm. (reported[3] b.p. 133–134°/16 mm.), 123°/10 mm., 109°/5 mm., n_D^{20} 1.5157.

Anal. Calcd. for $C_{10}H_{12}O_3$: C, 66.65; H, 6.71. Found: C, 66.73; H, 6.76.

The low-boiling fraction (10 g., b.p. 97–121°/10 mm.) consisted of a mixture of unknown by-products. Infrared analysis indicated the presence of alcoholic (2.9 μ) and acetal functions (8.9 and 9.4 μ) as well as a compound absorbing at 4.73 μ. Upon redistillation of this material, a sirupy residue was obtained which showed a band in the infrared at 5.77 μ and appeared to be a nonaromatic ester with an acetal. A very similar material was obtained as a high-boiling (150–211°/2–3 mm.) fraction, 23.9 g., following the dimethyl acetal cut. It consisted of a viscous orange sirup with infrared characteristics similar to those of the residue described above, including carbonyl absorption at 5.77 μ. Most of this substance boiled at 175–185°/2 mm. The following analyses were obtained for this unknown.

Anal. Calcd. for $C_{18}H_{21}O_4$: C, 71.50; H, 7.33; mol. wt., 302.4. Found: C, 70.84; H, 7.10; mol. wt., 319.

The aqueous hydrolysate following the sodium hydroxide treatment was acidified and worked up in the manner previously described, yielding 38.4 g. (12.7% yield) of crude DL-mandelic acid and 52.2 g. (21.4% yield) of benzoic acid.

2-(4-Methyl-1,3-dioxolan-2-yl)-2-phenyl-4-methyl-1,3-dioxolane (V).[4] Acetophenone (120 g., 1.0 mole) was partially dissolved in a mixture of 1,2-propanediol (456 g., 8.1 moles) and 1000 ml. of benzene. The resulting mixture was stirred vigorously and 2.39 moles of nitrosyl chloride (110 ml. at −30°) was added dropwise at 20–30° over a period of 2.25 hr. During the addition, an exothermic reaction occurred, and cooling was required to keep the temperature below 30°. At the end of the feed period, stirring was continued for 1 hr. at 25–35° and then for 1.5–2 hr. at 60°. During the reaction, approximately 1 mole of gas was liberated and found to consist entirely of nitrous oxide, nitric oxide, and nitrogen.

The cool reaction mixture consisted of two liquid layers. The upper layer was extracted five times with 500-ml. portions of water and freed of benzene on the steam bath, leaving 200 ml. of an oil. The lower layer was diluted with 1000 ml. of water and extracted with six 500-ml. portions of ether. These extracts were combined, dried (sodium sulfate), and evaporated free of ether to give approximately 60 ml. of yellow oil.

The organic oils from the two layers were combined and heated under reflux with 2000 ml. of 2*N* sodium hydroxide for a 1.5-hr. period. The mixture was then cooled and extracted with ether to obtain (after evaporation of ether) 113 g. of an oil. Fractional distillation of the oil at reduced pressure gave 90.4 g. of 2-(4-methyl-1,3-dioxolan-2-yl)-2-phenyl-4-methyl-1,3-dioxolane (36% yield) as a heavy colorless sirup. The main fraction boiled at 147–152°/3 mm., n_D^{20} 1.5169.

Anal. Calcd. for $C_{14}H_{18}O_4$: C, 67.18; H, 7.24, mol. wt. 250.3. Found: C, 66.37; H, 7.15, mol. wt. 250.

Infrared analysis revealed no bands in the carbonyl region and showed numerous bands in the 8–10 μ region in accord with the ketal-acetal structure. Surprisingly, no aromatic absorption occurred in the 6.2–6.35 μ region. The presence of a benzene ring was, however, confirmed by dichromate oxidation to benzoic acid. The material proved surprisingly inert toward permanganate oxidation.

The aqueous layer from the sodium hydroxide reaction was acidified to *p*H 1 to precipitate 58.6 g. of a gummy white solid having a neutralization equivalent of 205. The material was recrystallized from water to give 22.7 g. of benzoic acid, m.p. 121–122°. The balance of the material was not identified.

Following precipitation of the solid, the aqueous layer was extracted with ethyl ether to recover 35.3 g. of a viscous orange oil (neutralization equivalent 222). This material was distilled at reduced pressure to give 21.5 g. of oil, b.p. 110°–160°/3.5 mm. and a dark residue. The oil contained a small amount (1–3 g.) of benzoic acid and unknown, nonacidic, carbonyl compounds.

Acknowledgment. The authors are indebted to Mr. H. R. Harless for the mass spectrometric analyses and to Mr. C. R. Lovell for the infrared studies performed.

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[CONTRIBUTIONS FROM THE RESEARCH DEPARTMENT, UNION CARBIDE CHEMICALS CO.]

Some Esters of 1,2,4-Butanetricarboxylic Acid

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The preparation of several saturated, unsaturated and epoxyalkyl esters of 1,2,4-butanetricarboxylic acid is described.

Higher trialkyl esters of 1,2,4-butanetricarboxylic acid are known to be effective plasticizers for poly(vinyl chloride) resins.[1] A variety of such materials were prepared (Table I), most of which are new compounds, for evaluation in this application. In general, these plasticizers exhibit excellent low-temperature properties and low volatility.

A series of unsaturated esters of 1,2,4-butanetricarboxylic acid was prepared by esterification with unsaturated alcohols or by vinylation with acetylene (Table II). The allyl and vinyl esters were evaluated as polymerizable plasticizers and found to be effective. The higher unsaturated esters in general proved to be effective plasticizers, but their major utility was as intermediates in the preparation of a new class of epoxides.

A wide variety of epoxyalkyl esters of 1,2,4-butanetricarboxylic acid was prepared by the epoxidation of the respective unsaturated esters by peracetic acid in ethyl acetate[2] (Table III). In general, high yields of materials with high oxirane content were obtained. The epoxyalkyl esters proved to be excellent plasticizer-stabilizers for poly(vinyl chloride) resins and could be poly-

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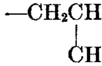
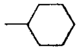
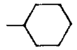
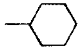
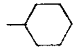
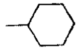
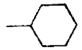
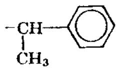
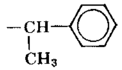
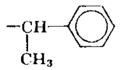
(1) British Patent 688,344 (1953), N. V. De Bataafsche Petroleum Maatschappij (The Netherlands).

(2) F. C. Frostick, B. Phillips, and P. S. Starcher, *J. Am. Chem. Soc.*, 81, 3350 (1959).

TABLE I

SATURATED ESTERS OF 1,2,4-BUTANETRICARBOXYLIC ACID

$[RO_2C—CH_2CH(COOR')CH_2CH_2COOR'']$

R	R′	R″	Formula	Yield, %	B.P.[a]/Mm.	n_D^{30}	d_{20}^{20}	Purity,[b] %	Calcd., %		Found, %	
									C	H	C	H
$—CH_3$	$—CH_3$	$—CH_3$	$C_{10}H_{16}O_6$	18[c]	110/1[d]	1.4407	—	100	—	—	—	—
$—C_2H_5$	$—C_2H_5$	$—C_2H_5$	$C_{13}H_{22}O_6$	87	143/2[e]	1.4350	1.0746	100	56.92	8.08	56.85	7.96
$—n\text{-}C_5H_9$	$—n\text{-}C_5H_9$	$—n\text{-}C_5H_9$	$C_{22}H_{40}O_6$	98	—[f]	1.4442	0.9800	100	65.97	10.07	66.42	10.23
$—n\text{-}C_6H_{13}$	$—n\text{-}C_6H_{13}$	$—n\text{-}C_6H_{13}$	$C_{25}H_{46}O_6$	99	—[f]	1.4448	0.9668	99	66.93	10.15	67.84	10.48
$—CH_2\underset{\substack{\vert\\ CH_3}}{CH}—C_3H_7$	$—CH_2\underset{\substack{\vert\\ CH_3}}{CH}—C_3H_7$	$—CH_2\underset{\substack{\vert\\ CH_3}}{CH}—C_3H_7$	$C_{25}H_{46}O_6$	98	138/2.3[g]	1.4447	0.9655	99	67.84	10.48	67.46	10.29
$—CH_2\underset{\substack{\vert\\ CH_3}}{CH}—C_3H_7$	$—CH_2\underset{\substack{\vert\\ C_2H_5}}{CH}—C_4H_9$	$—CH_2\underset{\substack{\vert\\ CH_3}}{CH}—C_3H_7$	$C_{25}H_{46}O_6$	99	—[f]	1.4462	0.9658	99.7	68.90	10.70	68.22	10.56
$—CH_2\underset{\substack{\vert\\ C_2H_5}}{CH}—C_4H_9$	—H	$—CH_2\underset{\substack{\vert\\ C_2H_5}}{CH}—C_4H_9$	$C_{23}H_{42}O_6$	90	—[f]	1.4452	0.9983	97	66.63	10.21	66.11	9.93
$—CH_2\underset{\substack{\vert\\ C_2H_5}}{CH}—C_4H_9$	$—CH_2\underset{\substack{\vert\\ CH_3}}{CH}—CH_3$	$—CH_2\underset{\substack{\vert\\ C_2H_5}}{CH}—C_4H_9$	$C_{27}H_{49}O_6$	98	—[f]	1.4470	0.9625	98	69.04	10.52	68.80	10.72
$—CH_2\underset{\substack{\vert\\ C_2H_5}}{CH}—C_4H_9$	$—CH_2\underset{\substack{\vert\\ CH_3}}{CH}—C_3H_7$	$—CH_2\underset{\substack{\vert\\ C_2H_5}}{CH}—C_4H_9$	$C_{29}H_{54}O_6$	—	130–68°/8[g]	1.4482	0.9560	99.4	69.84	10.91	69.92	10.97
$—CH_2\underset{\substack{\vert\\ C_2H_5}}{CH}—C_4H_9$	$—CH_2\underset{\substack{\vert\\ C_2H_5}}{CH}—C_4H_9$	$—CH_2\underset{\substack{\vert\\ C_2H_5}}{CH}—C_4H_9$	$C_{31}H_{58}O_6$	98.5	159–62°/0.1[g,h]	1.4500	0.9510	99	70.68	11.10	70.98	10.82
-"oxo" $C_{10}H_{21}$	-"oxo" $C_{10}H_{21}$	-"oxo" $C_{10}H_{21}$	$C_{37}H_{70}O_6$	94	—[f]	1.4537	0.9362	97	72.74	11.55	72.38	11.92
$—CH_2\underset{\substack{\vert\\ CH_3}}{CH}—CH_3$		$—CH_2\underset{\substack{\vert\\ CH_3}}{CH}—CH_3$	$C_{21}H_{36}O_6$	89	—[f]	1.4538	1.0345	95	—	—	—	—
	$—CH_2\underset{\substack{\vert\\ CH_3}}{CH}—CH_3$		$C_{23}H_{38}O_6$	95	—[f]	1.4673	1.0664	96	—	—	—	—
	$—CH_2\underset{\substack{\vert\\ C_2H_5}}{CH}—C_4H_9$		$C_{27}H_{46}O_6$	96	—[f]	1.4668	1.0265	98.7	69.49	9.94	68.86	9.85
$—CH_2\underset{\substack{\vert\\ C_2H_5}}{CH}—C_4H_9$		$—CH_2\underset{\substack{\vert\\ C_2H_5}}{CH}—C_4H_9$	$C_{29}H_{52}O_6$	98	—[f]	1.4574	0.9842	99.5	69.04	10.52	69.57	10.48
-"oxo" $C_{10}H_{21}$		-"oxo" $C_{10}H_{21}$	$C_{33}H_{60}O_6$	99	—[f]	1.4600	0.9683	98	—	—	—	—
			$C_{31}H_{34}O_6$	88	—[f]	1.5220	1.1215	93	—	—	—	—
$—CH_2CH_2CN$	$—CH_2CH_2CN$	$—CH_2CH_2CN$	$C_{16}H_{19}N_3O_6$	89	—[f]	1.4686	—	—	55.01	5.48	54.34	5.24

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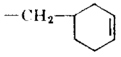
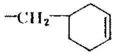
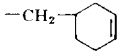
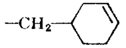
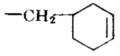
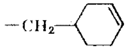
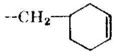
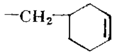
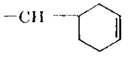
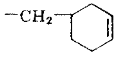
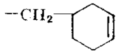
[a] All temperatures are uncorrected. [b] By saponification. [c] From crude oxidation mixture. [d] B.p. 112–114° (0.02 mm.); $d_D^{27°}$ 1.1538.[3] [e] Reported,[b] b.p. 162° (6 mm.); n_D^{20} 1.4390; d_D^{20} 1.0744. [f] Crude residue product. [g] Distilled on Spinning Plate Molecular Still. [h] B.p. 223° (5 mm.); n_D^{30} 1.4540; d^{24} 0.9502.[1]

TABLE II

UNSATURATED ESTERS OF 1,2,4-BUTANETRICARBOXYLIC ACID

R	R′	R″	Formula	Yield, %	B.P.[a]/Mm.	n_D^{30}	d_{20}^{20}	Purity,[b] %	Calcd., %		Found, %	
									C	H	C	H
$—CH_2CH=CH_2$	H	$—CH_2CH=CH_2$	$C_{13}H_{18}O_6$	100	—[d]	—	—	100	—	—	—	—
$—CH_2CH=CH_2$	$—CH_2CH=CH_2$	$—CH_2CH=CH_2$	$C_{16}H_{22}O_6$	97	151/0.5[c]	1.4633	1.0846	98	61.92	7.15	61.85	7.09
$—CH_2—\underset{CH_3}{\underset{\mid}{C}}=CH_2$	$—CH_2—\underset{CH_3}{\underset{\mid}{C}}=CH_2$	$—CH_2—\underset{CH_3}{\underset{\mid}{C}}=CH_2$	$C_{19}H_{28}O_6$	83	—[d]	1..4612	1.0500	99.5	64.75	8.01	63.31	8.01
$—CH_2CH=CH_2$	$—CH_2\underset{C_2H_5}{\underset{\mid}{CH}}—C_4H_9$	$—CH_2CH=CH_2$	$C_{21}H_{34}O_6$	88	—[d]	1.4559	1.0183	100	65.94	8.55	66.72	8.58
$—CH_2CH=CH_2$	$—CH_2\underset{C_2H_5}{\underset{\mid}{CH}}—C_4H_9$	$—CH_2\underset{C_2H_5}{\underset{\mid}{CH}}—C_4H_9$	$C_{26}H_{46}O_6$	94	—[d]	1.4520	0.9757	99	68.68	10.20	68.68	9.88
$—CH_2\underset{C_2H_5}{\underset{\mid}{C}}=CH—C_3H_7$	$—CH_2\underset{C_2H_5}{\underset{\mid}{C}}=CH—C_3H_7$	$—CH_2\underset{C_2H_5}{\underset{\mid}{C}}=CH—C_3H_7$	$C_{31}H_{52}O_6$	92	—[d]	1.4676	0.9726	100	71.50	10.07	71.51	10.00
$\begin{matrix}CH_3(CH_2)_7CH\\ \|\\ —(CH_2)_8CH\end{matrix}$	$\begin{matrix}CH_3(CH_2)_7CH\\ \|\\ —(CH_2)_8CH\end{matrix}$	$\begin{matrix}CH_3(CH_2)_7CH\\ \|\\ —(CH_2)_8CH\end{matrix}$	$C_{61}H_{112}O_6$	97	—[d]	1.4670	0.9079	93	77.81	11.99	77.92	11.96
	H		$C_{21}H_{30}O_6$	98	—[d]	1.4972	1.1207	100	66.64	7.99	66.37	7.92
	$—CH=CH_3$		$C_{23}H_{32}O_6$	80	—[d]	1.4958	1.1058	88.6	68.29	7.97	68.80	7.76
	$—CH_2—\underset{C_2H_5}{\underset{\mid}{CH}}—C_4H_9$		$C_{29}H_{46}O_6$	82	—[d]	1.4798	—	99	70.98	9.45	70.80	9.43
			$C_{28}H_{40}O_6$	95	—[d]	1.4990	1.0954	99	71.16	8.53	71.77	8.82
$—CH_2—\underset{C_2H_5}{\underset{\mid}{CH}}—C_4H_9$		$—CH_2\underset{C_2H_5}{\underset{\mid}{CH}}—C_4H_9$	$C_{30}H_{52}O_6$	86.7	—[d]	1.4642	0.9877	96.5	—	—	—	—
-"oxo" $C_{10}H_{21}$		-"oxo" $C_{10}H_{21}$	$C_{34}H_{60}O_6$	84.6	—[d]	1.9650	0.9750	98.7	—	—	—	—

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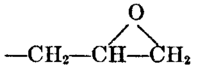
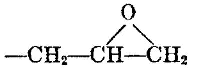
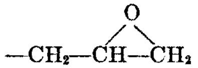
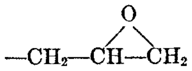
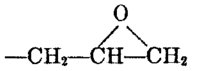
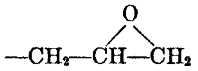
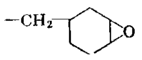
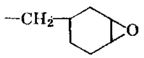
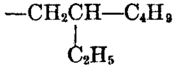
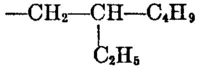
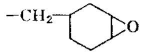
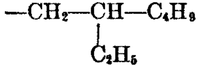
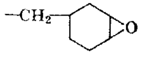
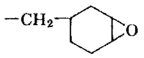
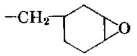
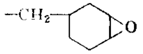
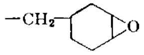
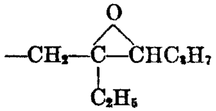
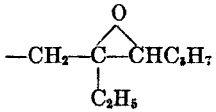
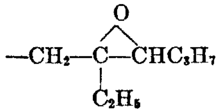
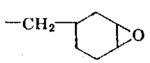
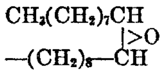
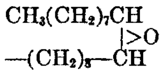
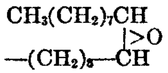
[a] All temperatures are uncorrected. [b] By saponification. [c] Rutoyskii and Leznon [*Zhur. Priklad. Khim.*, **22**, 887 (1949)] give b.p. 163–164° (1 mm.); n_D^{20} 1.4672; d_4^{20} 1.0820. [d] Crude residue product.

TABLE III

EPOXYALKYL ESTERS OF 1,2,4-BUTANETRICARBOXYLIC ACID

R	R′	R″	Formula	Yield, %	B.P.[a]/Mm.	Oxirane,[b] %	Purity, %	Calcd., % C	Calcd., % H	Found, % C	Found, % H
$-CH_2-CH{=}CH_2$	$-CH_2CH{=}CH_2$		$C_{16}H_{22}O_7$	31.6	168/0.06		66	—	—	—	—
$-CH_2-CH{=}CH_2$			$C_{16}H_{22}O_8$	9.2	186/0.14		65	—	—	—	—
			$C_{16}H_{22}O_9$	57.3	—[c]	7.6	56.6	—	—	—	—
	$-CH{=}CH_2$		$C_{23}H_{32}O_8$	66	—[c]	6.2	85	—	—	—	—
 $-CH_2CH(C_2H_5)-C_4H_9$	 $-CH_2-CH(C_2H_5)-C_4H_9$		$C_{30}H_{52}O_7$	90	—[c]	2.75	91	68.67	9.99	68.64	9.99
 $-CH_2-CH(C_2H_5)-C_4H_9$			$C_{29}H_{46}O_8$	81.3	—[c]	5.17	84	66.64	8.87	66.81	8.99
			$C_{28}H_{40}O_9$	81	—[c]	6.98	75.7	—	—	—	—
			$C_{31}H_{52}O_9$	—	—[c]	6.75	80	—	—	—	—
-“oxo” $C_{10}H_{21}$	-“oxo” $C_{10}H_{12}$		$C_{34}H_{60}O_7$	98	—[c]	2.37	90	70.31	10.41	69.92	10.42
			$C_{61}H_{112}O_9$	76	—[c]	3.89	77	74.04	11.41	73.79	11.57

[a] All temperatures are uncorrected. [b] Determination by reaction with pyridine hydrochloride. [c] Crude residue product.

merized to yield polymers of varying properties depending on the functionality of the epoxide monomer.

The triamide of 1,2,4-butanetricarboxylic acid was prepared by ammonolysis of the triethyl ester.

EXPERIMENTAL

General procedure for esterification of 1,2,4-butanetricarboxylic acid. A mixture of 3 moles of alcohol (or less if only mono- or diester is desired), 190 g. (1 mole) of 1,2,4-butanetricarboxylic acid, 500 g. of toluene, and 5 g. of *p*-toluenesulfonic acid was heated to reflux and water removed azeotropically. The reaction mixture was washed with one-third volume of 5% sodium bicarbonate solution and then with one-third volume of water. Solvent was removed by distillation at reduced pressure and the ester obtained by distillation or as a stripped residue product.

Vinylation method. Bis(3-cyclohexenylmethyl) vinyl 1,2,4-butanetricarboxylate. A mixture of 300 g. (0.795 mole) of bis(3-cyclohexenylmethyl) hydrogen 1,2,4-butanetricarboxylate, 500 ml. of toluene, 5 g. of zinc oxide, and 8 g. of anhydrous zinc acetate was charged to a 3-l. "316" stainless steel bomb in a rocking furnace. The system was purged five times with nitrogen to 75 p.s.i.g. at room temperature. Nitrogen was added to 25 p.s.i.g. and the system was heated to 160° with rocking. Nitrogen was added to 130 p.s.i.g., then acetylene was added to 300 p.s.i.g. Reaction occurred at 178° and the pressure was maintained at 280–300 p.s.i.g. by addition of acetylene. After 6 hr. at 178–191°, the gas uptake ceased. After cooling the system the reaction mixture was discharged, the catalyst removed by filtration, and the filtrate washed twice with 10% sodium carbonate solution to remove unreacted acid and then washed with water. Hydroquinone (0.5 g.) was added and the toluene was removed by distillation. The product was taken as a residual dark colored oil in 82% yield (n_D^{30} 1.4958, d_{20}^{20} 1.1058).

Anal. Calcd. for $C_{23}H_{32}O_6$: C, 68.3; H, 7.97. Found: C, 68.8; H, 7.76.

General procedure for epoxidation of unsaturated esters. Tri-(2,3-epoxy-2-ethylhexyl) 1,2,4-butanetricarboxylate. A solution (915 g.) of 21.5% peracetic acid in ethyl acetate was added dropwise to tri(2-ethyl-2-hexenyl) 1,2,4-butanetricarboxylate (309 g.) at 50° over a period of 90 min. After an additional 3 hr. at 50°, analysis of the mixture for peracetic acid indicated that the reaction was complete. The reaction mixture was then fed dropwise to an equal volume of ethylbenzene which was refluxing in a 24 inch × 1 inch glass helix-packed column at 25–30 mm. As the reaction mixture was fed to the kettle, ethyl acetate, acetic acid, and excess peracetic acid together with ethylbenzene were taken off overhead. After the volatiles had been removed, the kettle contents were filtered to remove a small amount of suspended matter, and tri(2,3-epoxy-2-ethylhexyl) 1,2,4-butanetricarboxylate was obtained as a residue product having the following properties: 6.75% oxirane oxygen (80% purity), 0.31% acid (calculated as acetic acid), iodine value (Wyj's method) 1.09, color less than 1 Gardner 1933.

Preparation of 1,2,4-butanetricarboxamide. Anhydrous ammonia was bubbled through a stirred mixture of 409 g. of triethyl 1,2,4-butanetricarboxylate (1.5 moles) in 500 g. of ethylene glycol at 59–60° over 38 hr., at which point 96 g. of ammonia (5.65 moles) had been adsorbed. The crude product mixture was freed of excess ammonia, by-product ethanol, and glycol by evaporation under reduced pressure at ambient temperature. The residual crystalline mass was taken up in hot dimethylformamide, treated with decolorizing carbon, filtered, and cooled to crystallize. After filtration, the semidry product was washed with ethyl ether and dried to give 143 g., a 51.6% yield, of fine white crystals (m.p. 195–196°) of 1,2,4-butanetricarboxamide.

Anal. Calcd. for $C_7H_{13}N_3O_3$: C, 44.91; H, 7.00; H, 22.45. Found: C, 45.18; H, 7.09; N, 22.50.

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SOUTH CHARLESTON, W. VA.

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(3) K. Freudenberg and J. Geiger, *Ann.*, **575**, 145 (1952).

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, CORNELL UNIVERSITY]

Enamines of 2-Indanones[1a,b]

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Enamines 2-(*N*-pyrrolidyl) (IIa), 2-(*N*-piperidyl) (IIb), 2-(*N*-hexamethyleneimino) (IIc), and 2-(*N*-morpholinyl)indene (IId) were prepared from 2-indanone and the appropriate *sec*-amines; similarly 3-phenyl-2-(*N*-hexamethyleneimino)indene was obtained from 1-phenyl-2-indanone (VIII). C-Alkylation of IIa–IId with bromomethyl benzyl ether was highest with IIc. 1-Methyl (V), 1-benzyloxymethyl- (VI), and 1-benzyl-2-indanone (VII) were prepared by C-alkylation of IIc with the appropriate alkyl halide. A shift of 108–131 cm.$^{-1}$ in the double bond stretching maximum was observed in going from IIa–IId to their respective ternary iminium salts. Selenium dioxide oxidation of VIII and 3-phenyl-1-indanone gave, respectively, 1-hydroxy-1-phenyl-2-indanone and 3-hydroxy-3-phenyl-1,2-indandione, and 3-phenylindone. Nitrosation of V and VI under alkaline conditions gave no α-oximinoindanones but unexpectedly yielded ring expanded products tentatively identified as alkylated 3-hydroxyisoquinoline *N*-oxides.

Enamine C-alkylation and acylation is now firmly established as a signal addition to the methods available for the preparation of α-substituted ketones from carbonyl compounds.[3] In this paper we report on the preparation, alkylation, and absorption spectra of enamines of 2-indanones

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