

melting point now found for the Schmidt reaction sample (110.5° cor.) as well as that obtained from 3,4-dimethylacetanilide differs from the 111–111.5° previously reported, which was determined using a Fisher-Johns apparatus. The present melting points were obtained in capillary tubes using a Hershberg type apparatus.

*Anal.* Calcd. for C<sub>10</sub>H<sub>12</sub>N<sub>4</sub>: C, 63.80; H, 6.43; N, 29.77. Found: C, 63.6; H, 6.2; N, 29.8.

The methiodides were best prepared by heating the tetrazole in a sealed tube with an excess of methyl iodide at 80–90° for 1.5 hours. The precipitate which formed was collected on a filter and recrystallized twice from ethanol. Both methiodides melted at 206° cor. The melting point of their mixture showed no depression.

*Anal.* Calcd. for C<sub>11</sub>H<sub>15</sub>N<sub>4</sub>I: C, 40.01; H, 4.58; N, 16.97. Found (A) methiodide from tetrazole prepared from 3,4-dimethylacetanilide: C, 39.8; H, 4.6; N, 17.2. (B) methiodide from tetrazole from Schmidt reaction: C, 39.5; H, 4.9; N, 16.8.

The absorption spectra were determined with a Model DU Beckman quartz spectrophotometer. Absolute ethanol was used as solvent; the following concentrations of tetrazole were employed for the wave lengths in question: 7.969 × 10<sup>-5</sup> M for 215–260 mμ; 1.594 × 10<sup>-4</sup> M for 260–270 mμ; 1.328 × 10<sup>-3</sup> M for 270–280 mμ; 1.198 × 10<sup>-2</sup> M for 285 mμ.

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### Some Aryloxyaliphatic Acids

By L. F. BERHENKE, L. E. BEGIN, B. M. WILLIAMS AND F. L. BEMAN

Several aryloxyaliphatic acids, not previously reported, have been made and are reported in Table I. The aryloxyacetic acids have been proposed as

described, we have found that crystallization of the acid from chlorobenzene or of the sodium salts from water at pH 10–13 are also effective methods for separating the acids from unreacted phenols.

The α- and β-substituted propionic acids and the α-substituted butyric acids were similarly prepared from α- and β-chloropropionic acid and α-bromobutyric acid, respectively.

The γ-substituted butyric acid was prepared by a modification of the method previously reported.<sup>3</sup> Two hundred thirty-two grams of *p*-phenylphenol was neutralized with 55 g. of sodium hydroxide in 1.5 l. of water and 148 g. of γ-bromobutyronitrile added over one hour, then the mixture was refluxed for two hours. Sixty-eight grams of sodium hydroxide was added as 10 N solution and the nitrile hydrolyzed by refluxing overnight. The reaction mixture (pH about 11) was cooled, filtered, washed with water and the moist cake resuspended in 10 l. of water, acidified with concentrated hydrochloric acid, digested on the steam-bath for several hours, cooled and filtered. The crystals were dried and recrystallized from 2 l. of chlorobenzene; yield 190 g., 74%, m.p. 151–155°. Further recrystallization gives material m.p. 158.5–160°.

(3) Lohman, *Ber.*, **24**, 2631 (1891).

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### The Conversion of Δ<sup>4</sup>-Cholestone-3-one to Cholesterol<sup>1</sup>

By B. BELLEAU AND T. F. GALLAGHER

Because of our need for effecting the transformation of cholestone to cholesterol in the maximum yield for partial synthesis of the isotopically labelled sterol we have investigated the action of sodium borohydride on the enol acetate of cholestone and have obtained cholesterol in 70 to 85% yield. Dauben and Eastham<sup>2</sup> with lithium aluminum

TABLE I

Compound	Formula	M.p., °C.	Carbon, %		Hydrogen, %		Chlorine, %		Neut. equiv.	
			Calcd.	Found	Calcd.	Found	Calcd.	Found	Calcd.	Found
<b>Acetic acid</b>										
<i>p</i> -Acetylphenoxy-	C <sub>10</sub> H <sub>10</sub> O <sub>4</sub>	172.5–174.5	61.84	61.77	5.19	5.18			194.2	195.5
4- <i>s</i> -Butyl-2,6-dichlorophenoxy-	C <sub>12</sub> H <sub>14</sub> Cl <sub>2</sub> O <sub>2</sub>	78.4–80					25.62	25.60	277.1	276.0
3-Chloro-4-biphenyloxy-	C <sub>14</sub> H <sub>11</sub> ClO <sub>2</sub>	158–159					13.50	13.53	262.7	263.8
5-Chloro-2-biphenyloxy-	C <sub>14</sub> H <sub>11</sub> ClO <sub>2</sub>	123–125					13.50	13.58	262.7	265.2
4-Chloro- <i>o</i> -cumyloxy-	C <sub>11</sub> H <sub>13</sub> ClO <sub>2</sub>	170–171					15.50	15.37	228.7	231.7
2,6-Dichlorophenoxy-	C <sub>8</sub> H <sub>6</sub> Cl <sub>2</sub> O <sub>2</sub>	134.7–135					32.10	32.27	221.0	221.0
3,5-Dichlorophenoxy-	C <sub>8</sub> H <sub>6</sub> Cl <sub>2</sub> O <sub>2</sub>	116–116.5					32.10	32.18	221.0	221.0
2,3,6-Trichlorophenoxy-	C <sub>8</sub> H <sub>5</sub> Cl <sub>3</sub> O <sub>2</sub>	147–148					41.60	41.59	255.5	261.8
<b>Butyric acid</b>										
γ-(4-Biphenyloxy)-	C <sub>16</sub> H <sub>16</sub> O <sub>3</sub>	158.5–160	74.97	74.90	6.29	6.31			256.3	265.2
α-( <i>p</i> - <i>t</i> -Butylphenoxy)-	C <sub>14</sub> H <sub>20</sub> O <sub>2</sub>	89–90.5	71.15	71.06	8.53	8.49			236.3	238.7
α-( <i>o</i> -Chlorophenoxy)-	C <sub>10</sub> H <sub>11</sub> ClO <sub>2</sub>	80–80.5					16.53	16.43	214.6	212.3
α-( <i>p</i> -Chlorophenoxy)-	C <sub>10</sub> H <sub>11</sub> ClO <sub>2</sub>	77.5–78							214.6	213.7
α-(2,4,5-Trichlorophenoxy)-	C <sub>10</sub> H <sub>5</sub> Cl <sub>3</sub> O <sub>2</sub>	140–141					37.52	37.36	283.5	282.3
<b>Propionic acid</b>										
α-( <i>p</i> -Butylphenoxy)- <sup>a</sup>	C <sub>13</sub> H <sub>18</sub> O <sub>2</sub>	89–90.5	70.26	70.08	8.16	8.20			222.2	218.6
β-(2,4,5-Trichlorophenoxy)-	C <sub>9</sub> H <sub>7</sub> Cl <sub>3</sub> O <sub>2</sub>	143–144							269.5	269.1

<sup>a</sup> Preparation reported by Salminen and Weissberger, U. S. Patent 2,423,730, but no constants are given.

identifying derivatives for phenols<sup>1</sup> and can be made by the method there given or by modifications thereof.<sup>2</sup> In addition to the purification schemes

hydride reduced cholestone enol acetate to

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(2) W. G. Dauben and J. F. Eastham, *THIS JOURNAL*, **72**, 2305 (1950).

(1) Koelsch, *THIS JOURNAL*, **53**, 304 (1931).

(2) Hayes and Branch, *ibid.*, **65**, 1555 (1943).