

THE PHENACYL, *p*-PHENYLPHENACYL AND *p*-NITROBENZYL ESTERS OF CERTAIN UNEVEN NUMBERED FATTY ACIDS

The general method of preparation consisted in dissolving 150 mg. of the fatty acid in 4 cc. of 95% ethyl alcohol, titrating to phenolphthalein with 0.5 *N* sodium hydroxide and just reacidifying by the addition of a crystal of the fatty acid so that the solution becomes colorless; 150 mg. of the phenacyl bromide was then added and the mixture refluxed for one and one-half hours. Two recrystallizations from 95% ethyl alcohol were sufficient to purify the derivatives of the acids from tridecyl upward, while two to three crystallizations from approximately 65% ethanol were required for the lower derivatives.

The melting points were taken with a Fisher melting point apparatus<sup>1</sup> using a thermometer calibrated against a set of Anschutz thermometers corrected by the Bureau of Standards, the temperature being raised at the rate of one degree in about two minutes.

Acid <sup>a</sup>	Ester	M. p. (cor.), C.	Empirical formula	Analysis, %			
				Calcd. C	Calcd. H	Found C	Found H
Pelargonic	Phenacyl	oil					
	<i>p</i> -Phenylphenacyl	70.8–71.3	C <sub>22</sub> H <sub>28</sub> O <sub>3</sub>	78.36	8.01	78.29	8.34
	<i>p</i> -Nitrobenzyl	oil					
Undecylic	Phenacyl	oil					
	<i>p</i> -Phenylphenacyl	79.5–80	C <sub>26</sub> H <sub>32</sub> O <sub>3</sub>	78.87	8.48	78.85	8.62
	<i>p</i> -Nitrobenzyl	oil					
Tridecyl	Phenacyl	45.0–45.5	C <sub>21</sub> H <sub>27</sub> O <sub>3</sub>	75.84	9.71	75.97	9.81
	<i>p</i> -Phenylphenacyl	86.5–87	C <sub>27</sub> H <sub>36</sub> O <sub>3</sub>	79.36	8.89	79.42	8.73
	<i>p</i> -Nitrobenzyl	oil					
Pentadecylic	Phenacyl <sup>b</sup>	53.6	C <sub>23</sub> H <sub>30</sub> O <sub>3</sub>	76.61	10.07	76.34	10.25
	<i>p</i> -Phenylphenacyl	91.3–91.8	C <sub>29</sub> H <sub>40</sub> O <sub>3</sub>	79.76	9.24	79.55	9.50
	<i>p</i> -Nitrobenzyl	39.5–40	C <sub>22</sub> H <sub>25</sub> O <sub>4</sub> N	69.97	9.35	70.16	9.71
Margaric	Phenacyl	60.0–60.5	C <sub>25</sub> H <sub>40</sub> O <sub>3</sub>	77.26	10.38	77.16	10.50
	<i>p</i> -Phenylphenacyl	95.3–95.8	C <sub>31</sub> H <sub>44</sub> O <sub>3</sub>	80.11	9.55	79.76	9.67
	<i>p</i> -Nitrobenzyl <sup>c</sup>	48.5–49.0	C <sub>24</sub> H <sub>30</sub> O <sub>4</sub> N	71.05	9.70	70.84	10.06

<sup>a</sup> Heptico *p*-nitrobenzyl ester was obtained only as an oil. <sup>b</sup> This ester melts at 53.6° only if heated rapidly; if heated in the normal way it melts several degrees lower. <sup>c</sup> Lyons and Reid [THIS JOURNAL, 39, 1737 (1917)] reported an attempt to prepare this ester, but, as they could not get consistent melting points, abandoned the experiment.

(1) H. L. Fisher, "Laboratory Manual of Organic Chemistry," 3rd ed., John Wiley and Sons, Inc., New York, N. Y., 1934, p. 28.

ORGANIC RESEARCH LABORATORY DONALD PRICE  
NATIONAL OIL PRODUCTS CO. RICHARD GRIFFITH  
HARRISON, N. J.

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NITRO DERIVATIVE OF 2-BROMO-4-AMINO-1,3-DIMETHYLBENZENE

2-Bromo-4-acetylamino-6-nitro-*m*-xylene.—Two grams of 2-bromo-4-acetylamino-*m*-xylene<sup>1</sup> was dissolved in 20 g. of concentrated sulfuric acid and treated with 0.75 g. of concentrated nitric acid below 15°. After ten minutes, the solution was poured into ice water. The precipitate was collected, washed with water, and recrystallized from dilute alcohol. It was obtained as small white needles, m. p. 171–172°. Yields on 30-g. quantities of starting material were consistently above 90%.

(1) Noelting, Braun, and Thesmar, *Ber.*, 34, 2242 (1901).

*Anal.* Calcd. for C<sub>10</sub>H<sub>11</sub>O<sub>2</sub>N<sub>2</sub>Br: N, 9.76. Found: N, 9.83.

2-Bromo-4-amino-6-nitro-*m*-xylene.—Thirty grams of 2-bromo-4-acetylamino-6-nitro-*m*-xylene was refluxed with 50% sulfuric acid until all had dissolved. The solution was poured into cold water and neutralized with ammonia water. The precipitate was collected, washed with water, and recrystallized from alcohol after decolorization with Norit. It formed yellow needles, m. p. 129–130°, with extensive sublimation.

*Anal.* Calcd. for C<sub>8</sub>H<sub>9</sub>O<sub>2</sub>N<sub>2</sub>Br: N, 11.44. Found: N, 11.67.

The structure of both compounds was proved by conversion of the latter into 2-bromo-4-nitro-*m*-xylene, by reduction of the diazonium sulfate with alcohol. The product, isolated by steam distillation, was obtained in colorless, waxy needles of plastic consistency, m. p. 56.5–58°, after several decolorizations and recrystallizations from alcohol. No depression was observed when a mixed

melting point was taken with 2-bromo-4-nitro-*m*-xylene obtained from 2-amino-4-nitro-*m*-xylene by the method of Noelting, Braun, and Thesmar.<sup>1</sup> The color of the compound obtained by their method is light yellow.

GEORGE HERBERT JONES LABORATORY  
UNIVERSITY OF CHICAGO WILLIAM CARL SPITZER  
CHICAGO, ILL.

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γ,γ'-DI-*p*-TOLYL-γ,γ'-SUBERODILACTONE

The reduction of 50 g. of β-*p*-toluylpropionic acid with 75 g. of zinc dust in 200 cc. of boiling 80% acetic acid for six hours yielded, in addition to the main product, γ-*p*-tolyl-γ-butyrolactone (m. p. 67–68°),<sup>1</sup> 2.5 g. of glistening white plates, m. p. 275–276°, which separated from the reaction mixture on cooling. This material has been identified as γ,γ'-di-*p*-tolyl-γ,γ'-suberodilactone, result-

(1) Gergel and Wagner (*Ann.*, 482, 74 (1930)) obtained the same product by reduction with sodium amalgam, m. p. 69°.