

TABLE II  
 5-SUBSTITUTED 5-(2-NAPHTHYL)HYDANTOINS

R	Mp, °C <sup>a</sup>	Yield, %	Calcd, %			Found, % <sup>b</sup>		
			C	H	N	C	H	N
CH <sub>3</sub>	247-248	83	69.99	5.04	11.66	70.31	4.97	11.53
<i>n</i> -C <sub>3</sub> H <sub>7</sub>	239-240	78	71.62	6.01	10.44	71.84	6.09	10.30
<i>i</i> -C <sub>3</sub> H <sub>7</sub>	261-263	72	...	...	10.44	...	...	10.29
<i>n</i> -C <sub>4</sub> H <sub>9</sub>	201-202	76	72.32	6.43	9.92	72.21	6.49	9.85
<i>i</i> -C <sub>4</sub> H <sub>9</sub>	217-218	69	...	...	9.92	...	...	10.04
<i>sec</i> -C <sub>4</sub> H <sub>9</sub>	256-257	57	...	...	9.92	...	...	10.01
<i>t</i> -C <sub>4</sub> H <sub>9</sub>	292-293 dec	43	72.32	6.43	9.92	71.99	6.42	10.09
<i>n</i> -C <sub>5</sub> H <sub>11</sub>	188-189	72	72.95	6.80	9.45	72.96	6.66	9.39
<i>i</i> -C <sub>5</sub> H <sub>11</sub>	244-245	87	...	...	9.45	...	...	9.32
<i>n</i> -C <sub>6</sub> H <sub>13</sub>	177-178	85	...	...	9.03	...	...	9.01
<i>n</i> -C <sub>7</sub> H <sub>15</sub>	167-168	95	...	...	8.63	...	...	8.74
<i>n</i> -C <sub>8</sub> H <sub>17</sub>	169-170	64	...	...	8.27	...	...	8.24
<i>n</i> -C <sub>10</sub> H <sub>21</sub>	167-168	73	...	...	7.65	...	...	7.62
<i>n</i> -C <sub>12</sub> H <sub>25</sub>	169-170	70	...	...	7.10	...	...	7.09
C <sub>6</sub> H <sub>5</sub>	281-282	63	...	...	9.27	...	...	9.32
1-C <sub>10</sub> H <sub>7</sub>	323-324	68	78.39	4.58	7.95	77.82	4.29	8.33
2-C <sub>10</sub> H <sub>7</sub>	313-314	64	78.39	4.58	7.95	78.39	4.51	7.92

<sup>a</sup> All melting points were determined by the capillary method and are corrected. <sup>b</sup> Carbon, hydrogen, and nitrogen microanalyses were performed by the Huffman Laboratories, Inc., Wheatridge, Colo.

## Heterocycles. II.<sup>1</sup> Synthesis of 3-Carbomethoxy-3-methyl-7,8-benzothiochromanone

TOSIO MORIWAKE

Department of Industrial Chemistry, School of Engineering,  
Okayama University, Okayama, Japan

Received December 4, 1965

During an attempted syntheses of 11-thia steroid homologs, the title compound was synthesized as an intermediate. The method of synthesis is analogous to the route used by Bachmann, *et al.*,<sup>2</sup> for the preparation of equilenin.

### Experimental Section

**Methyl 7,8-Benzothiochromanone-3-glyoxalate.**—To a suspension of 3.2 g of sodium methoxide in 40 ml of benzene was added 7.1 g of dimethyl oxalate, and the mixture was refluxed for 10 min. To the ice-cooled solution was added a solution of 6.4 g of 7,8-benzothiochromanone<sup>3</sup> in 70 ml of benzene over a 10-min period, and the mixture was stirred at room temperature for 4 hr. Within a few minutes a light red solution resulted, which soon deposited a light yellow precipitate. The mixture was hydrolyzed with 100 ml of water. The benzene solution which separated was extracted twice with 60 ml of 2% NaOH solution and the combined aqueous solution was acidified with dilute HCl. The light yellow crystals were filtered off and dried. Recrystallization from ethanol gave 7.3 g (81%) of glyoxalate as pale yellow clusters which melted at 107-109°. Further recrystallizations from alcohol gave a pure sample of mp 108.5-109.5°.

*Anal.* Calcd for C<sub>16</sub>H<sub>12</sub>O<sub>4</sub>S: C, 64.00; H, 4.03. Found: C, 64.08; H, 4.10.

**3-Carbomethoxy-7,8-benzothiochromanone.**—A mixture of 7.0 g of the above-mentioned glyoxalate and 3.5 g of powdered soft glass was heated at 180-200° for 1 hr with occasional stirring. After cooling, the dark brown product was dissolved in a mixture of benzene and acetone (1:1), and the solution was decanted from the glass. The solution was evaporated, and the residue was digested with methanol, whereupon crystallization took place. Recrystallization from ethyl acetate gave 5.1 g of product, mp 114-116°, as yellow needles.

*Anal.* Calcd for C<sub>15</sub>H<sub>12</sub>O<sub>3</sub>S: C, 66.17; H, 4.44. Found: C, 66.21; H, 4.52.

**3-Carbomethoxy-3-methyl-7,8-benzothiochromanone.**—A warm solution of 3.6 g of 3-carbomethoxy-7,8-benzothiochromanone in 30 ml of benzene was added to a solution of sodium methoxide prepared from 1.6 g of sodium and 30 ml of methanol. The mixture was refluxed for 2 hr, cooled, and treated with 4 ml of methyl iodide. After 1 hr at room temperature, an additional 4 ml of methyl iodide was added. The resulting mixture was stirred at room temperature for 30 min, then refluxed for 2 hr, cooled, neutralized with acetic acid, and evaporated nearly to dryness. The residue was treated with benzene and water, and the organic solution after separating was washed with 5% NaOH solution with water, dried, and evaporated. Recrystallization of the residue from ethanol gave 3.5 g (92%) of the product, mp 112-113°, as tan needles.

*Anal.* Calcd for C<sub>16</sub>H<sub>14</sub>O<sub>3</sub>S: C, 67.12; H, 4.93. Found: C, 67.30; H, 5.14.

## Synthesis of Some 3-Arylacetyl- and 1,3-Di(arylacetyl)indoles<sup>1</sup>

THOMAS E. YOUNG AND MICHAEL F. MIZIANTY<sup>2</sup>

William H. Chandler Chemistry Laboratory, Lehigh University,  
Bethlehem, Pennsylvania

Received December 13, 1965

The recent demonstration of anticonvulsant activity<sup>3</sup> of certain 3-acylindoles has prompted us to report eighteen new 3-arylacetylindoles, of which compounds Ia-n (Table I) were all prepared by acylation of the corresponding indolylmagnesium bromides with the appropriate arylacetyl chloride, a method first described by Oddo<sup>4</sup> and briefly elaborated by others.<sup>5</sup> The 1,3-diacetyl derivatives (II) were also produced as coproducts, and could be obtained pure in several cases (Table II). The 3-arylacetyl-2-methylindoles (Io-r, Table I) were prepared by reac-

(1) This investigation was supported by research Grant C-4425 from the National Cancer Institute, National Institutes of Health, U. S. Public Health Service.

(2) Abstracted in part from the Ph.D. Dissertation of M. F. M., Lehigh University, 1963.

(3) H. H. Keasling, R. E. Willette, and J. Szmuzkovicz, *J. Med. Chem.*, **7**, 94 (1964).

(4) B. Oddo and L. Sessa, *Gazz. Chim. Ital.*, **41**, 234 (1911).

(5) N. P. Buu-Hoi, E. Bisagni, and C. Routier, *J. Chem. Soc.*, 625 (1957); S. Takagi, A. Sugii, and K. Machida, *Pharm. Bull. (Tokyo)*, **5**, 617 (1957); T. E. Young, *J. Org. Chem.*, **27**, 509 (1962); T. E. Young and M. F. Mizianty, *ibid.*, **29**, 2030 (1964).

(1) T. Moriwake, *J. Med. Chem.*, **9**, 163 (1966).

(2) W. E. Bachmann, W. Cole, and A. L. Wilds, *J. Am. Chem. Soc.*, **61**, 974 (1939); **62**, 824 (1940); see also ref. 1.

(3) F. Krollpfeiffer and H. Schultze, *Ber.*, **56**, 1821 (1923).