

point of the *p*-nitrophenylhydrazone was depressed by the addition of pure *p*-nitrophenylhydrazine, m. p. 157°.

*Anal.* Calcd. for the *p*-nitrophenylhydrazone, C<sub>12</sub>H<sub>11</sub>N<sub>3</sub>O<sub>2</sub>: C, 61.78; H, 6.49. Found: C, 61.76; H, 6.48. Calcd. for the 2,4-dinitrophenylhydrazone, C<sub>12</sub>H<sub>11</sub>N<sub>4</sub>O<sub>4</sub>: C, 51.79; H, 5.07. Found: C, 51.82; H, 4.81.

Attempts to prepare the *p*-methoxy- and *p*-bromophenylhydrazones by similar methods were not successful.

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### Condensation of Chloromaleic Anhydride with Substituted Propenylbenzenes

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The condensation of maleic anhydride with iso-

#### DERIVATIVES OF 3-METHYL-3,4-DIHYDRONAPHTHALENE-1,2-DICARBOXYLIC ACID ANHYDRIDE

Derivative	Yield, g.	Solvent	M. p., °C.	Formula	Analyses, %			
					Carbon Calcd.	Carbon Found	Hydrogen Calcd.	Hydrogen Found
6,7-Methylenedioxy	10	Benzene	176-177	C <sub>14</sub> H <sub>10</sub> O <sub>5</sub>	65.1	65.0	3.91	3.95
6-Methoxy-7-hydroxy	9	Xylene	225-226	C <sub>14</sub> H <sub>12</sub> O <sub>5</sub>	64.7	65.0	4.65	4.69
6-Ethoxy-7-hydroxy	6	Toluene	192-196	C <sub>15</sub> H <sub>14</sub> O <sub>5</sub>	65.7	65.6	5.14	5.07

safrole to form 6,7-methylene-dioxy-3-methyl-1,2,3,4-tetrahydronaphthalene-1,2-dicarboxylic acid anhydride has been reported by Hudson

and Robinson.<sup>1</sup> They similarly condensed isosafrole with ethyl acetylenedicarboxylate, obtaining the corresponding 3,4-dihydronaphthalene derivative as the acid anhydride, m. p. 178°.

The latter compound has now been shown to form when chloromaleic anhydride is heated in xylene with isosafrole. Analogous compounds formed when chloromaleic anhydride was condensed with isoeugenol and 2-ethoxy-4-propenylphenol.

**Experimental.**—The procedures were similar for the three cases. Thirteen grains of chloromaleic anhydride, 15 g. of the propenyl compound, and 50 ml. of xylene were refluxed for six hours. The color turned to a bright red and hydrogen chloride was eliminated during the reaction. On cooling, the products crystallized from the xylene. The yields and properties are tabulated.

(1) Hudson and Robinson, *J. Chem. Soc.*, 715 (1941).

(2) Analyses by Elisabeth Heber-Smith.

BOYCE THOMPSON INSTITUTE FOR PLANT RESEARCH, INC.  
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## COMMUNICATIONS TO THE EDITOR

### TRIFLUOROACETIC ACID AS A CONDENSING AGENT

Sir:

I wish to report the use of trifluoroacetic acid as an agent for the condensation of acetic anhydride with anisole to produce *p*-methoxyacetophenone. The reaction proceeds well (63%, or 91% if allowance is made for recovered anisole) at moderate temperatures (60-70°). Trifluoroacetic acid has several advantages over other reagents commonly employed for such condensations: there is no demethylation of anisole or the product; there is little if any heat of reaction so that all reagents may be mixed at one time; no stirring is required; no corrosive gases are used or formed; the trifluoroacetic acid may be recovered.

**Procedure.**—On mixing 15 g. of anisole, 14.8 g. of anhydrous trifluoroacetic acid, and 27 g. of acetic anhydride, heat (of mixing) was evolved and a pink color was produced. On warming to 60-70° the color deepened into cherry red. After six hours at 60-70° the mixture was poured into

water. The acids were removed by shaking with water and the organic layer was extracted with alkali, but no phenol or *p*-hydroxyacetophenone was found. On vacuum distillation there were isolated 4.7 g. (31%) of anisole and 13.1 g. (63%) of *p*-methoxyacetophenone, b. p. 134-137 at 15-16 mm. This material crystallized and melted over the range 30-36°. The crude semicarbazone formed in 76% yield melted at 193-195°. On recrystallization it melted at 195.6-197.0° cor. These facts indicate that almost pure para derivative was produced.<sup>1</sup> The mixed melting point with purified semicarbazone from commercial *p*-methoxyacetophenone (Eastman Kodak Co.) was not depressed. In a similar experiment, except that the reaction was carried out at 110-125°, a much smaller yield of ketone was obtained as tars were formed.

I have also used trichloroacetic acid<sup>2</sup> in a similar manner, but it is less desirable than trifluoro-

(1) Wahl and Silberzweig, *Bull. soc. chim.*, [4] 11, 69 (1912), give 36°, 197°, of 138-139° as melting points of ketone and semicarbazone and boiling point of ketone at 15 mm.

(2) Unger, *Ann.*, 504, 269 (1933).