

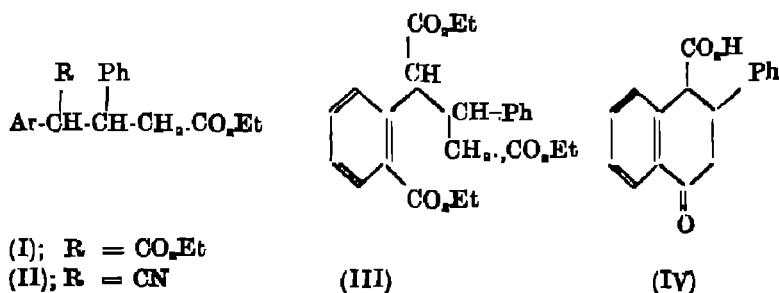
## NOTES

### Polycyclic Compounds. Part IV\*. Michael Reaction of Cinnamic Ester with a few Active Methylene Compounds

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Ethyl cinnamate is known<sup>1</sup> to undergo Michael reaction with ethyl phenylacetate to give ethyl  $\alpha,\beta$ -diphenylglutarate. In the present communication, we like to report a few more condensation reactions of ethyl cinnamate with arylacetic esters and arylacetoneitriles furnishing respectively polycarboxylic esters and nitrile-esters which could be further utilised for the synthesis of polycyclic compounds.

Ethyl 1-naphthylacetate and ethyl homophthalate for this purpose have been condensed



with ethyl cinnamate in presence of ethanolic sodium ethoxide to yield the polyesters (I; Ar = 1-naphthyl) and (III) in good yield. The latter on Dieckmann condensation<sup>2</sup> followed by hydrolysis afforded 1,2,3,4-tetrahydro-2-phenyl-4-oxo-1-naphthoic acid (IV). The structure of the compound was confirmed by its conversion into 2-phenyl-1,2,3,4-tetrahydro-1-naphthoic acid and then into ethyl 2-phenylnaphthalene-1-carboxylate.

Naphthylacetonitriles ( $\alpha$  and  $\beta$ ) were likewise converted into nitrile-esters (II; Ar = 1-naphthyl and 2-naphthyl). Uses of these compounds for syntheses of polycyclic compounds are in progress.

\*Part III. This *Journal*, 1965, 470.

1. Borsche, *Ber.*, 42, 4497.

2. Bachmann, Cole and Wilds, *J. Amer. Chem. Soc.*, 1940, 62, 835.

## EXPERIMENTAL

*Ethyl β-phenyl-α-(1-naphthyl)glutarate* (I; Ar=1-naphthyl).—Ethyl 1-naphthylacetate (16.1 g.) prepared from 1-chloromethylnaphthalene<sup>3</sup> by the usual method<sup>4</sup> was added to a solution of sodium (1.7 g.) in anhydrous ethanol (37 ml.) Ethyl cinnamate (13.3 g.) was added to it in the cold and the mixture refluxed on a water bath for 6 hr. The product was cooled and then acidified with HCl (conc., 8.25 ml). *Ethyl β-phenyl-α-(1-naphthyl)glutarate* (I; Ar=1-naphthyl) (17.1 g.) formed colourless prism from alcohol, m.p. 110°. (Found: C, 76.91; H, 6.58. C<sub>23</sub>H<sub>26</sub>O<sub>4</sub> requires C, 76.92; H, 6.6%). *β-Phenyl-α-1-naphthylglutaric acid* melted at 205°. (Found: C, 75.21; H, 5.44. Equiv. 167. C<sub>21</sub>H<sub>18</sub>O<sub>4</sub> requires C, 75.44; H, 5.38%. Equiv. 167).

*Ethyl β-phenyl-γ-cyano-γ-(1-naphthyl)butyrate* (II; Ar=1-naphthyl)<sup>5</sup>.—A mixture of 1-cyanomethylnaphthalene (16.7 g.) and ethyl cinnamate (17.6 g.) was added slowly to a stirred ice-cold mixture of dry ether (200 ml.) and sodium ethylate prepared from sodium (2.3 g.) in anhydrous ethanol (40 ml.) according to the known procedure due to Avery<sup>5</sup>. After addition, stirring and cooling were continued for about 5 hr. The product was cooled, acidified with hydrochloric acid and the ether layer was separated. The aqueous phase was extracted once with ether and the combined ether extract was washed with water and the solvent evaporated. The residue of the viscous oil crystallised from ethanol, m.p. 108°, yield 8.1 g. (Found: C, 80.45; H, 8.10. C<sub>23</sub>H<sub>21</sub>O<sub>2</sub>N requires C, 80.46; H, 6.12%).

A list of other compounds prepared under similar experimental condition is given below:

Compounds	m.p.	Formula	Found	Reqd.
Ethyl β-phenyl-α-(2-ethoxy-carbonylphenyl) glutarate (III).	100° (b.p. 210-215°/4mm).	C <sub>24</sub> H <sub>28</sub> O <sub>6</sub>	C:69.68% H:7.3	69.9% 6.79
β-Phenyl-α-(2-carboxyphenyl) glutaric acid.	159°	C <sub>15</sub> H <sub>16</sub> O <sub>6</sub>	C:65.76 H:5.0	65.85 4.87
Ethyl β-phenyl-γ-cyano-γ-(2-naphthyl) butyrate (II; Ar=2-naphthyl).	107°	C <sub>23</sub> H <sub>21</sub> O <sub>2</sub> N	C:80.39 H:6.0	80.46 6.12

*2-Phenyl-4-oxo-1,2,3,4-tetrahydro-1-naphthoic Acid* (IV).—The triester (III) (8.26 g.), sodium dust (0.552 g.) and dry benzene (16.4 ml.) were refluxed on a steam bath till sodium went completely into solution. The crude residue (10 g.) worked up in the usual manner was hydrolysed with HCl (conc., 20 ml.) and glacial acetic acid (40 ml.) and worked up as usual. The oily residue was dissolved in ethyl acetate and then diluted with petroleum (60-80°) to give crystalline *oxo-acid*, m.p. 115° (d). (Found: C, 76.68; H, 5.36. C<sub>17</sub>H<sub>14</sub>O<sub>3</sub> requires C, 76.68; H, 5.26%).

It afforded *2-phenyl-1,2,3,4-tetrahydro-1-naphthoic acid*, m.p. 140-141°. (Found: C,

3. *Org. Syntheses*, Vol. 24, p.30.

4. cf. *Rec. Trav. Chim.*, 1937, 56, 853; *Ber.*, 55, 1841.

5. cf. Helm Kamp *et al.*, *J. Amer. Chem. Soc.*, 1940, 3217; Cook, *J. Chem. Soc.*, 1945, 439.

6. cf. *J. Amer. Chem. Soc.*, 1928, 50, 2516.

80.87; H, 6.29.  $C_{17}H_{16}O_2$  requires C, 80.95; H, 6.3% on Clemmensen reduction<sup>7</sup>. The *ethyl ester* boiled constantly at 160°/4 mm. (Found: C, 81.47; H, 7.2.  $C_{19}H_{20}O_2$  requires C, 81.42; H, 7.14%). Sulphur dehydrogenation of the latter gave *ethyl 2-phenyl-naphthalene-1-carboxylate*, b.p. 185°/4 mm. (Found: C, 82.50; H, 5.76.  $C_{19}H_{16}O_2$  requires C, 82.6; H, 5.79%).

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