

agent, the resulting mixture was stirred for several minutes longer, heated under reflux for two hours and then allowed to cool and stand overnight at room temperature. The reaction flask was cooled in an ice-water-bath and the pale yellow product decomposed with dilute hydrochloric acid. A 300-ml. quantity of diethyl ether was added to dilute the benzene solution of 2-(2'-methoxybenzoyl)-benzoic acid and 2,2-di-*o*-anisylphthalide. The ether-benzene layer was extracted with potassium carbonate solution, and the carbonate extract then run dropwise into vigorously stirred, ice-cold hydrochloric acid. In this way the keto acid first separated as a pasty mass which soon became solid. This pale fawn-colored, granular solid was filtered off and washed with cold diethyl ether. Upon drying, 34.5 g. (54%) of crude acid was obtained. After four recrystallizations from glacial acetic acid, 2-(2'-methoxybenzoyl)-benzoic acid was secured in the form of short, colorless, small prisms; m. p. 143–143.5°; yield 30 g. (47%, based on phthalic anhydride). Sieglitz<sup>2</sup> reported 144–145° as the m. p. of this acid.

*Anal.* Neutralization equivalent, calcd. for C<sub>15</sub>H<sub>12</sub>O<sub>4</sub>: 256.2. Found: 257.9.

From the above-mentioned benzene-ether layer, after the potassium carbonate extraction and concentration of the solution to a small volume, a colorless powder was obtained. This material, 2,2-di-*o*-anisylphthalide, when recrystallized three times from glacial acetic acid, formed small, colorless crystals, m. p. 148–149°; yield 10.5 g. (18%, based on *o*-bromoanisole). The phthalide was found to be soluble in concentrated sulfuric acid with a very deep violet color which gradually changed to ruby-red and finally to orange.

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### The Dissociation of Hexaarylethanes. XIV.<sup>1</sup> Ethanes Derived from Mixtures of Triaryl Halides

By C. S. MARVEL AND CHESTER M. HIMEL

Since symmetry seemed to be important in connection with the influence of substituents on the degree of dissociation of triarylmethyls, it occurred to us that treating an equimolecular mixture of two triarylmethyl halides with silver might produce a truly unsymmetrical ethane rather than a mixture of ethanes. Table I contains a list of some mixtures of chlorides which were thus treated and the degree of dissociation calculated from magnetic susceptibility measurements on the assumption that a mixed ethane was formed. In one case (the first in the table) the mixed ethane was prepared by mixing preformed solutions of the individual ethanes rather than by action of silver on the mixed halides.

(1) For the thirteenth communication see *J. Org. Chem.*, **7**, July (1942).

TABLE I  
DISSOCIATION OF SOME MIXED HEXAARYLETHANES

| Chlorides used                                   | $-\chi \times 10^6$ | $\alpha$ at 0.1 M, % | $\alpha$ of corresponding ethane at 0.1 M, % | Ref. |
|--|---------------------|----------------------|--|------|
| <i>o</i> -Chlorophenyldiphenylmethyl             | 0.6620              | 14 = 2               | 12 = 1                                       | 1    |
| <i>o</i> -Bromophenyldiphenylmethyl              |                     |                      | 17 = 1                                       | 1    |
| <i>o</i> -Tolyldiphenylmethyl                    | .6320               | 26 = 2               | 25 = 1                                       | 2    |
| $\alpha$ -Naphthyldiphenylmethyl                 |                     |                      | 27 = 2                                       | 3    |
| <i>o</i> -Tolyldiphenylmethyl                    | .6282               | 27 = 2               | 25 = 1                                       | 2    |
| Di- <i>o</i> -tolylphenylmethyl                  |                     |                      | 82 = 2                                       | 4    |
| Tri- <i>p</i> -diphenylmethyl                    | .6765               | 25 = 3 <sup>a</sup>  | 25 = 5                                       | 3    |
| Tri- $\beta$ -naphthylmethyl                     |                     |                      | 25 = 5                                       | 3    |
| <i>p</i> - <i>t</i> -Amylphenyldiphenylmethyl    | .6985               | 3.5 = 1              | 8.0 = 1                                      | 4    |
| Phenyl-di- <i>p</i> - <i>t</i> -amylphenylmethyl |                     |                      | 9.0 = 1                                      | 4    |
| Tri- <i>p</i> - <i>t</i> -butylphenylmethyl      | .6997               | 3.5 = 1              | 20 = 4                                       | 4    |
| <i>p</i> - <i>t</i> -Butylphenyldiphenylmethyl   |                     |                      | 7.5 = 1                                      | 4    |

<sup>a</sup> The actual measurement was made at 0.025 M and the dissociation at this concentration was 43 = 4%. The value in the table was calculated by means of the mass law.

The method and apparatus have been described in previous papers in this series. It can be seen readily that the observed degrees of dissociation are not the average of the two simple ethanes, but in general are lower than this value. There are several equilibria possible in this complicated system and a complete appraisal of the significance of these experimental results will not be possible until these equilibria are more thoroughly studied. Since it seems unlikely that this can be done soon, these results are recorded.

(2) Marvel, Mueller, Himel and Kaplan, *THIS JOURNAL*, **61**, 2771 (1939).

(3) Marvel, Shackleton, Himel and Whitson, *ibid.*, **64**, 1824 (1942).

(4) Marvel, Kaplan and Himel, *ibid.*, **63**, 1892 (1941).

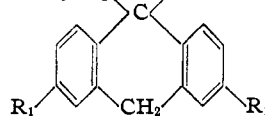
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### The Reaction of Furoic Acid with Aromatic Compounds. III

By CHARLES C. PRICE, E. C. CHAPIN AND MARTIN RIEGER

The isolation of methyl 9-ethyl-9,10-dihydro-9-anthroate (I) from the aluminum chloride catalyzed reaction of methyl furoate with benzene<sup>1</sup> suggests the presence of the corresponding acid (II) in the mixture obtained from a similar re-



I (R = CH<sub>3</sub>, R<sub>1</sub> = H)  
II (R = R<sub>1</sub> = H)  
III (R = H, R<sub>1</sub> = CH<sub>3</sub>)

(1) Price and Huber, *THIS JOURNAL*, **64**, 2136 (1942).