lactone (m.p. 179–180°) in 80% yield by the method of Beschke.[8b] Upon treatment of the monolactone with alcoholic sodium hydroxide as described by Beschke[8b] for its ethyl ester, disodium $\beta,\beta'$-diphenylmuconate was obtained.

*1,4-Dibenzoyl-2,3-diphenyl-1,3-butadiene* (III). Phenyllithium was prepared from 1.7 g. (0.011 mole) of bromobenzene and 0.16 g. (0.023 mole) of lithium in 30 ml. of dry ether by the procedure of Evans and Allen.[14] To this solution was added 1 g. (0.003 mole) of disodium $\beta,\beta'$-diphenylmuconate (VI) and the resulting mixture was refluxed, with stirring, for 5 hours, after which time it was poured onto ice. The resulting mixture was extracted with ether, and the ether extract was dried and evaporated to 10 ml. The precipitated product weighed 0.2 g. (16% yield) and melted at 190–191°. It showed no melting point depression in admixture with the dibenzoyldiphenylbutadiene of Wislicenus and Lehmann.[3]

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(14) Evans and Allen in Blatt, *Org. Syntheses*, Coll. Vol. 2, 517 (1943).

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## Derivatives of 3,4,5-Trimethoxybenzamide[1]

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In the course of an investigation, involving the evaluation of qualitative and quantitative effects of certain radicals and functional groups upon the pharmacological action of mescaline, we have prepared several arylalkylamides of 3,4,5-trimethoxybenzoic acid. The pharmacological evaluation of these compounds is in progress.

### EXPERIMENTAL

(All melting points are uncorrected. Microanalyses by Drs. G. Weiler and F. B. Strauss, Oxford, England. The compounds were obtained in their highest state of purity in 30 to 40% yields.)

*dl-[N-(α-Methylbenzyl)-3,4,5-trimethoxybenzamide]* (I). The 3,4,5-trimethoxybenzoyl chloride was prepared by using a modification of the Marsh and Stephen procedure.[2] To 12 g. (0.0565 mole) of 3,4,5-trimethoxybenzoic acid, dispersed in 200 ml. of anhydrous benzene, 67 g. (0.565 mole) of thionyl chloride was added. The resulting mixture was heated on the water-bath (50°) until a clear solution was obtained and the latter was refluxed for 30 minutes to 1 hour on the steam-bath. The excess thionyl chloride and the benzene were removed under reduced pressure (max. pot temp. 40°). The residual thionyl chloride was removed by azeotropic distillation under reduced pressure with two 100-ml. portions of anhydrous benzene. The residue was taken up in 400 ml. of anhydrous benzene and 35 g. (0.289 mole) of *dl*-(α-methylbenzylamine), in 100 ml. of anhydrous benzene, was added gradually. The contents of the reaction vessel were refluxed for 8 hours on the steam-bath. The reaction mixture was cooled to 20° and treated with cold 40% aqueous potassium hydroxide. The benzene layer was drawn off, dried over magnesium sulfate, filtered, and the benzene was removed under reduced pressure. The white crystalline residue was recrystallized thrice from benzene. The crystals melted at 177.5–178.0°.

*Anal.* Calc'd for $C_{18}H_{21}NO_4$: C, 68.54; H, 6.71; N, 4.44. Found: C, 68.74; H, 6.71; N, 4.40.

*N-Phenethyl-3,4,5-trimethoxybenzamide* (II). The acid chloride of 3,4,5-trimethoxybenzoic acid (12 g., 0.0565 mole) was prepared as above. To the acid chloride, dissolved in 400 ml. of anhydrous benzene, 35 g. (0.289 mole) of phenethylamine in 100 ml. of anhydrous benzene, was added gradually. The resulting mixture was heated for 3 hours on the water-bath (50°). The reaction mixture was cooled to 20° and the phenethylamine hydrochloride formed during the reaction was filtered off. The benzene solution was washed with cold 40% aqueous potassium hydroxide, dried over magnesium sulfate, filtered, and the benzene was removed under reduced pressure. The crystalline residue was three times recrystallized from benzene. The white crystals melted at 122.0–123.0°.

*Anal.* Calc'd for $C_{18}H_{21}NO_4$: C, 68.54; H, 6.71; N, 4.44. Found: C, 68.46; H, 6.71; N, 4.42.

*N-(3,4-Dimethoxyphenethyl)-3,4,5-trimethoxybenzamide* (III). The acid chloride of 3,4,5-trimethoxybenzoic acid (12 g., 0.0565 mole) was prepared as above. To the acid chloride, 12 g. (0.0665 mole) of 3,4-dimethoxyphenethylamine, dispersed in 200 ml. of 15% aqueous sodium hydroxide, was added. The reaction mixture was heated for 2 hours on the water-bath (50°), while applying vigorous stirring. The precipitate formed during the reaction was filtered off, washed with water, and the residual moisture was removed by azeotropic distillation under reduced pressure with two 300-ml. portions of anhydrous benzene. The product was three times recrystallized from benzene. The white crystals melted at 133.0–133.4°.

*Anal.* Calc'd for $C_{20}H_{25}NO_6$: C, 63.98; H, 6.71; N, 3.73. Found: C, 64.03; H, 6.74; N, 3.84.

*N-(3,4,5-Trimethoxyphenethyl)-3,4,5-trimethoxybenzamide* (IV). The acid chloride of 3,4,5-trimethoxybenzoic acid (4.5 g., 0.0212 mole) was prepared as above. To the acid chloride, 4.5 g. (0.0182 mole) of 3,4,5-trimethoxyphenethylamine hydrochloride, dispersed in 100 ml. of 15% aqueous sodium hydroxide, was added. The reaction mixture was heated for 4 hours on the water-bath (50°), while applying vigorous stirring. The precipitate formed during the reaction was filtered off and washed with water. The air-dried product was recrystallized thrice from a benzene-ethanol solvent system. The white crystals melted at 180.5–181.0°.

*Anal.* Calc'd for $C_{21}H_{27}NO_7$: C, 62.21; H, 6.71; N, 3.46. Found: C, 62.43; H, 6.76; N, 3.44.

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(2) J. T. Marsh and H. Stephen, *J. Chem. Soc.*, 127, 1635 (1925).

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## N-Benzylidenebenzylamine from Benzylamine and Butyl Nitrite

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When carefully dried benzylamine and butyl nitrite were mixed there was no apparent reaction, but in the presence of even a trace of water, a slow

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