2-Phenyl-3-*n*-butyl-4-thiazolidone-1-dioxide.—The following procedure is typical of the method used for the preparation of the 1-dioxides listed in Table II. To a mixture of 59 g. (0.25 mole) of 2-phenyl-3-*n*-butyl-4-thiazolidone, 500 ml. of glacial acetic acid and 125 ml. of acetic anhydride was added in one portion 125 ml. of 30% hydrogen peroxide. The temperature of the mixture slowly increased to 55°. At this point the flask was intermittently immersed in cold water so as to keep the temperature below 60°. When the evolution of heat had apparently ceased, the solution was allowed to stand for several hours at room temperature. It was then concentrated *in vacuo* on a water-bath at 65° until practically all of the solvent had been removed. The residue was mixed with 200 ml. of methanol and 300 ml. of water, heated for a short time on a steam-bath and cooled. The solid product was filtered off and recrystallized from 275 ml. of methanol.

Summary

A series of 2,3-disubstituted-4-thiazolidones has been prepared for testing as possible anticonvulsants. Several of the compounds have been oxidized to the corresponding 2,3-disubstituted-4-thiazolidone-1-dioxides.

Preliminary results indicate that certain of the derivatives are effective in giving protection against electrically induced convulsions while other members of the series inhibit metrazol induced convulsions. Oxidation of the 4-thiazolidones leads to less active derivatives.

DETROIT, MICHIGAN RECEIVED APRIL 7, 1948

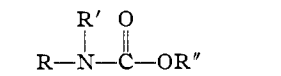
[CONTRIBUTION FROM THE ABBOTT RESEARCH LABORATORIES]

Carbamate Antimalarials

BY MARLIN T. LEFFLER AND EDWARD J. MATSON

In the broad antimalarial screening program sponsored by the Committee on Medical Research, there were, among the samples submitted by Abbott Laboratories, a number of compounds with the carbamate linkage. These carbamates were tested at Johns Hopkins University under the direction of E. K. Marshall, Jr., and it was observed that one of them in particular, *p*-carbobutoxyphenyl carbanilate (SN-1285), showed definite antimalarial effect against *P. lophurae* in ducks. The activity appeared to be in the neighborhood of one-tenth that of quinine. While this order of activity was not striking, the lead did seem unique in that these compounds bore little resemblance to previously known antimalarials, and for this reason a more extensive investigation of the series was desirable.

In this study the substituents on the carbamate moiety were varied as follows



where, as seen in Table I, R and R′ are hydrogen, alkyl, aryl or heterocyclic groups, while R″ is usually a substituted aryl group. In general, two methods of synthesis were used: the carbamates were formed either from the chloroformates and amines (method A), or through the isocyanates with or without a solvent (methods C and B, respectively).

In method A, it developed that pyridine was an ideal solvent for the condensation with aromatic amines and could easily be removed from the product at the end of the reaction by dilution with cold water. However, when more basic amines such as dimethylamine were employed, pyridine was unsatisfactory and two moles of the amine in an inert solvent were necessary.

When isocyanates were used, a trace of triethylamine catalyst[1] greatly increased the rate of the addition reaction. Many of the carbamates described in Table I were made in this manner using a reaction temperature of about 200° in the absence of a solvent (method B); however, it is worth special mention that, in certain instances, this simple procedure gave only low yields of the desired carbamates and produced instead rather significant amounts of the corresponding ureas. In fact, in some cases (SN-4178, SN-3231, Table I) urea formation predominated, and in the case of 2,4-dinitrophenol, only diphenylurea resulted. While the mechanism by which ureas are formed is not clearly understood, it was found that the amount of urea produced could be influenced to a considerable degree by varying the reaction temperature. It was for this reason that the low temperature procedure with a solvent (method C) was employed for the preparation of a number of the carbamates herein reported. By using a mixed solvent of dioxane and toluene, it was possible to effect a complete solution of most of the isocyanate–phenol combinations that were used and, when the temperature was controlled at about 10–15°, urea formation in most cases was diminished or completely avoided. In this instance also, triethylamine was added as a catalyst.

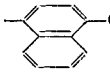
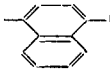
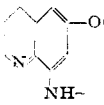
Antimalarial Activity.—The details of the tests and activities of these compounds will be found in the monograph, "A Survey of Antimalarial Drugs 1941–1945."[2] The results may be summarized by saying that the introduction of carbobutoxy or sulfamyl groups on the phenyl in R″ (Table I) and the *p*-methoxy substitution in the phenyl R′ when R was hydrogen increased the activity against *P. lophurae*. Other groups were not so effective. However, highly active

(1) Tarbell, Mallatt and Wilson, THIS JOURNAL, **64**, 2229 (1942).

(2) F. Y. Wiselogle, "A Survey of Antimalarial Drugs 1941–1945," J. W. Edwards, Ann Arbor, Michigan, 1947.

TABLE I

CARBAMATES $R'R''N-\overset{O}{\overset{\|}{C}}-OR'''$

SN[a]	R'R''N-	-R'''	M. p., °C.	Method	Yield[j]	Formula	N Analyses, % Calcd.	Found
3,211	H_2N-	*p*-$C_6H_4CO_2C_4H_9$-*n*	97–97.5	A[a]	..	(3) $C_{12}H_{15}NO_4$	5.90	6.03
2,194	*i*-C_4H_9NH-	*p*-$C_6H_4CO_2CH_3$	104–106	B	16	(1) $C_{13}H_{17}NO_4$	5.58	5.58
2,193	$(CH_3)_2N$-	*p*-$C_6H_4CO_2C_4H_9$-*n*	B. p. 206–207 (8 mm.)	A[b]	75	$C_{14}H_{19}NO_4$	5.28	5.33
3,227	C_6H_5NH-	*m*-$C_6H_4OCH_3$	123–124	B[c]	..	(3) $C_{14}H_{13}NO_3$	5.76	6.02
3,795	C_6H_5NH-	*m*-$C_6H_4CF_3$	138–140	C	69	(1) $C_{14}H_{10}F_3NO_2$	4.98	4.98
3,229	C_6H_5NH-	*o*-$C_6H_4CO_2CH_3$	119–120[d]	B[c]	89	(4) $C_{15}H_{13}NO_4$[d]		
1,282	C_6H_5NH	*p*-$C_6H_4CO_2CH_3$	141–142[e]	B	65	(7) $C_{15}H_{13}NO_4$[b]		
1,285	C_6H_5NH-	*p*-$C_6H_4CO_2C_4H_9$-*n*	87–88	B	53	(4) $C_{18}H_{19}NO_4$	4.47	4.79
4,730	C_6H_5NH-	*p*-$C_6H_4CO_2C_4H_9$-*iso*	89.5–90.5	B	64	(4) $C_{18}H_{19}NO_4$	4.47	4.61
4,377	C_6H_5NH-	*p*-$C_6H_4CONH_2$	187–188	C[f]	95	(1) $C_{14}H_{12}N_2O_3$	10.92	11.06
2,159	C_6H_5NH-	*p*-$C_6H_4SO_2NH_2$	177–178	C	58	(8) $C_{13}H_{12}N_2O_4S$	9.59	9.47
3,230	C_6H_5NH-	*p*-$C_6H_4SO_2NHC_4H_4N_2$[g]	207–209[h]	C	89	(5) $C_{17}H_{14}N_4O_4S$	15.12	15.13
3,231	C_6H_5NH-		123–125	C	42	(4) $C_{18}H_{13}NO_2$	4.81	5.01
3,232	C_6H_5NH-		159–161	C	66	(1) $C_{19}H_{15}NO_4$	4.36	4.47
7,085	C_6H_5NH-	*p*-C_6H_4CN	154–155	C	81	(5) $C_{14}H_{10}N_2O_2$	11.76	11.60
10,848	C_6H_5NH-	*p*-$C_6H_4C(OC_2H_5)=NH$	196–198 Hydrochloride	..	84	(2) $C_{16}H_{17}ClN_2O_3$	8.73	8.82
10,830	C_6H_5NH-	*p*-$C_6H_4C(SC_2H_5)=NH$	203[h] Hydrochloride	..	97	$C_{16}H_{17}ClN_2O_2S$	8.51	7.97[k]
10,938	C_6H_5NH-	*p*-$C_6H_4-C(=S)-NH_2$	161–162	...	..	(6) $C_{14}H_{12}N_2O_2S$	10.29 S: 11.77	10.31 11.44
10,937	C_6H_5NH-	*p*-$C_6H_4-C(=S)-SC_2H_5$	133–134	C	50	(1) $C_{16}H_{15}NO_2S_2$	4.41	4.68
....	*p*-$CH_3OC_6H_4NH$-	*p*-$C_6H_4-C(=S)-SC_2H_5$	142–143	C	34	(1) $C_{17}H_{17}NO_3S_2$	4.03	4.25
4,177	C_6H_5NH-		161–162	B	..	(3) $C_{15}H_{12}N_2O_2$	10.59	10.54
3,226	*p*-$CH_3OC_6H_4NH$-	C_6H_5	145–146	A	..	(3) $C_{14}H_{13}NO_3$	5.76	5.86
3,218	*p*-(*n*)-$C_4H_9O_2CC_6H_4NH$-	C_6H_5	125–126	A	..	(4) $C_{18}H_{19}NO_4$	4.47	4.70
2,195	$C_6H_5(C_2H_5)N$-	*p*-$C_6H_4CO_2C_4H_9$-*n*	B. p. 225–230 (2 mm.)	A	88	$C_{20}H_{23}NO_4$	4.10	3.98
2,609	$(C_2H_5)_2N-(CH_2)_3-N(C_6H_5)-$	*p*-$C_6H_4CO_2C_4H_9$-*n*	B. p. 250 (3 mm.)[l]	A[b]	71	$C_{26}H_{34}N_2O_4$	6.57	6.59[l]
1,286	α-$C_{10}H_7NH$-	*p*-$C_6H_4CO_2C_4H_9$-*n*	105–107	B	69	(4) $C_{22}H_{21}NO_4$	3.86	4.12
2,160	*p*-ClC_6H_4NH-	*p*-$C_6H_4CO_2C_4H_9$-*n*	118–119	A	95	(1) $C_{18}H_{16}ClNO_4$	4.03	4.04
2,610	*p*-Cl-*o*-$CH_3OC_6H_3NH$-	*p*-$C_6H_4CO_2C_4H_9$-*n*	86–87	A	..	(1) $C_{19}H_{20}ClNO_5$	3.71	3.93
3,233	*o*-$CH_3OC_6H_4NH$-	*p*-$C_6H_4CO_2C_4H_9$-*n*	72.5–73	A	71	(4) $C_{19}H_{21}NO_5$	4.08	4.16
1,048	*p*-$CH_3OC_6H_4NH$-	*p*-$C_6H_4CO_2C_4H_9$-*n*	100–101	A	98	(3) $C_{19}H_{21}NO_5$	4.08	4.16
2,196	*p*-$C_4H_9O_2C-C_6H_4-NH$-	*p*-$C_6H_4CO_2C_4H_9$-*n*	107–108	A[b]	72	(4) $C_{22}H_{27}NO_6$	3.39	3.64
....	*p*-$O_2NC_6H_4NH$-	*p*-$C_6H_4CO_2C_4H_9$-*n*	120–121	A	96	(1) $C_{18}H_{18}N_2O_5$	7.82	7.91
4,178	*p*-$CH_3OC_6H_4NH$-	*p*-$C_6H_4SO_2NH_2$	173–174[h]	C	50	(1) $C_{14}H_{14}N_2O_5S$	8.69	8.69
2,192	$C_8H_{10}N$-	*p*-$C_6H_4CO_2C_4H_9$-*n*	62–64	A	39	(1) $C_{17}H_{23}NO_4$	4.59	4.24
2,197	C_4H_8ON-	*p*-$C_6H_4CO_2C_4H_9$-*n*	B. p. 210–211 (3 mm.)	A[b]	80	$C_{16}H_{21}NO_5$	4.55	4.25
2,606	$CH_2(OCH_2-O-CH_2-C(CH_3)_2-NH-)_2$	*p*-$C_6H_4CO_2C_4H_9$-*n*	77–78		86	(1) $C_{17}H_{23}NO_5$	4.15	4.15
2,161		*p*-$C_6H_4CO_2C_4H_9$-*n*	116–118	A[b]		(1) $C_{22}H_{22}N_2O_5$	7.10	7.25

[a] Reaction run in excess 15% ammonium hydroxide at 0–10°; no pyridine used. [b] Dry benzene was substituted for pyridine as reaction medium using 2 moles of amine. [c] Reaction carried out at 65°. Higher temperature promoted formation of N,N'-diphenylurea. [d] Michael and Cobb, *Ann.*, **363**, 89 (1908), report m. p. 115–116°. [e] Michael and Cobb, *loc. cit.*, have reported m. p. 134–135°. [f] Dioxane–pyridine (50:50) used for reaction solvent and product precipitated with cold water. [g] Made from sulfadiazine. [h] Melted with decomposition. [i] For synthesis see description under Experimental. [j] Figures in parentheses refer to the following solvents used for recrystallization: (1) alcohol, (2) absolute alcohol–dry ether, (3) benzene (4) benzene–Skelly B (1:8), (5) dioxane, (6) ether, (7) ethyl acetate, (8) ethyl acetate–Skelly B (1:2). [k] Attempts to purify this unstable compound resulted in decomposition. [l] Refractive index. $n^{22}D$ 1.5310.

compounds were not found in this series. An activity 0.6 that of quinine was reached with *p*-carbobutoxyphenyl *p'*-methoxycarbanilate (SN-1048), and *p*-sulfamylphenyl *p'*-methoxycarbanilate (SN-4178) gave an effect equal to about one-third that of quinine. In both cases, this activity fell to less than Q 0.1 when tests were made against *P. knowlesi* (monkey).[3]

Experimental[4]

Starting Materials.—5-Amino-5-methyl-1,3-dioxane was furnished by the Commercial Solvents Corporation.

The methods for preparing the substituted phenols listed herewith are given in the following references: ethyl *p*-hydroxydithiobenzoate[5]; *p*-hydroxybenzamide[6]; *p*-hydroxybenzenesulfonamide[7]; 4-hydroxy-1-naphthaldehyde[8]; *p*-hydroxybenzonitrile[9]; methyl 4-hydroxy-1-naphthoate.[10]

Phenyl chloroformate was prepared as described by Raiford and Inman.[11]

***p*-Carbobutoxyphenyl Chloroformate.**—Essentially the same procedure, described by Raiford and Inman[11] for phenyl chloroformate, gave a 91% yield of *p*-carbobutoxyphenyl chloroformate boiling at 130–131° (1 mm.).

Anal. Calcd. for $C_{12}H_{13}ClO_4$: Cl, 13.85. Found: Cl, 14.28.

N-(γ-Diethylaminopropyl)-formanilide.—By using a procedure similar to that developed by Walker[12] for the corresponding ethylene homolog, a 48.3% yield of product was obtained, b. p. 150–160° (2 mm.); $n^{26}D$ 1.5200.

Anal. Calcd. for $C_{14}H_{22}N_2O$: N, 11.96. Found: N, 11.95.

N-(γ-Diethylaminopropyl)-aniline.—The hydrolysis of the above N-(γ-diethylaminopropyl)-formanilide was carried out according to Walker,[12] giving a 77% yield of product, b. p. 128–130° (1 mm.); $n^{20}D$ 1.5231.

Anal. Calcd. for $C_{13}H_{22}N_2$: C, 75.67; H, 10.74; N, 13.58. Found: C, 75.52; H, 10.61; N, 13.51.

General Procedures for Carbamates, Method A.—In a 250-ml. three-necked flask, fitted with a mechanical stirrer, thermometer and dropping funnel, was placed 0.11 mole of the desired amine in 75 ml. of dry pyridine. To this solution was then added, dropwise with stirring, 0.10 mole of the chloroformate. The temperature of the reaction mixture was maintained at 25–35°. After the addition was complete the reaction mixture was stirred at room temperature for several hours. It was then poured into an excess of ice water which precipitated the product. The solid was separated by filtration (oily products were extracted with benzene), washed well with cold 5% hydrochloric acid, and then with water. After thorough drying, the product was recrystallized from the solvent indicated in Table I.

Method B.—In a round-bottomed flask equipped with a thermometer and a reflux condenser protected by a calcium chloride drying tube were placed 1 mole of the phenolic compound, 1 mole of the isocyanate and a few drops of triethylamine as a catalyst.[1] The internal temperature was raised gradually, by heating the flask in a suitable bath until the reaction was initiated. Generally, the reaction started at about 150° and became sufficiently vigorous to raise the temperature spontaneously to approximately 200°. During the initial heating period the reactants were mixed thoroughly by gentle swirling until a clear melt was obtained. Then, if necessary, the heating was continued to bring the temperature to 200° where it was maintained for approximately one-half hour.

At the end of this time, the reaction mixture was allowed to cool and was poured into four volumes of petroleum ether (b. p. approx. 70°). The solid which separated was purified by recrystallization from the solvent indicated in Table I.

Method C.—In a 1-liter three-necked flask equipped with an agitator, thermometer and dropping funnel was placed a solution of 0.2 mole of the substituted phenol in 300 ml. of dry dioxane–dry toluene mixture (1:1). The solution was cooled to 10–15°, two or three drops of triethylamine were added as a catalyst[1] and this was followed by the dropwise addition, with stirring, of 0.2 mole of the desired isocyanate in 50 ml. of dry dioxane. The temperature of the reaction mixture was held at 10–15° during the addition of the isocyanate and then for several hours longer. Finally the solution was allowed to stand overnight at room temperature. The carbamate was isolated by precipitation with an excess of ether or petroleum ether. Purification was accomplished by recrystallizing the product from the solvent indicated in Table I.

***p*-Carbimidoethoxyphenyl Carbanilate Hydrochloride (SN-10,848).**—*p*-Cyanophenyl carbanilate (SN-7085), 5.0 g. (0.02 mole), was dissolved in a mixture of 100 ml. of absolute ethanol and 30 ml. of dry dioxane, cooled to 0°, and treated with anhydrous hydrogen chloride. The solution was allowed to stand at 0–5° for four days with resaturation by hydrogen chloride at the end of the first two days. Since no solid had separated after this time, the solution was concentrated *in vacuo* at 50° to about 10 ml. Anhydrous ether was added, the colorless solid was separated by filtration and recrystallized from an anhydrous alcohol–ether mixture, m. p. 196–198° (see Table I).

***p*-Carbobutoxyphenyl N-(5-Methyl-1,3-dioxan-5-yl)-carbamate (SN-2606).**—To 25 g. (0.21 mole) of 5-amino-5-methyl-1,3-dioxane in 100 ml. of water was added, dropwise and with good stirring, 25.7 g. (0.1 mole) of 4-carbobutoxyphenyl chloroformate. The temperature rose but was controlled at 30–32°. A cloudiness developed in the reaction mixture and two layers gradually formed. Stirring was continued for two hours longer at room temperature. The reaction mixture was then extracted completely with ether, the organic layer was washed with water and dried over anhydrous sodium sulfate. Removal of the solvent left a colorless viscous residue. When triturated with alcohol the entire amount solidified. Crystallization from alcohol gave 29 g. (86%) of colorless prisms melting at 77–78°.

***p*-Thiocarbamidophenyl Carbanilate (SN-10,938).**—A solution containing 10.0 g. (0.4 mole) of *p*-cyanophenyl carbanilate (SN-7085) and 10.4 g. (0.168 mole) of ethyl mercaptan in 100 ml. of dry dioxane was cooled in an ice-bath and then saturated with dry hydrogen chloride gas. The reaction mixture was allowed to stand at room temperature for several days; it was resaturated with hydrogen chloride once during this period. Removal of most of the solvent *in vacuo* gave a colorless solid which was washed well with anhydrous ether. This material amounted to 13.2 g. (97%), m. p. 203° (dec.). Attempted recrystallization gave decomposition. It is assumed that this substance is *p*-carbimidothioethoxyphenyl carbanilate hydrochloride (SN-10,830).

In an attempt to convert the above hydrochloride into *p*-dithiocarboethoxyphenyl carbanilate by the following method, *p*-thiocarbamidophenyl carbanilate was the only product isolated. *p*-Carbimidothioethoxyphenyl carbanilate hydrochloride, 21.0 g. (0.06 mole), was treated under 50 ml. of ether with 100 ml. of cold 5% aqueous sodium hydroxide solution. The mixture was shaken for a few minutes, the organic layer was separated, dried over anhydrous magnesium sulfate and then gassed with hydrogen sulfide for eighteen hours. The solid product obtained by the evaporation of the solvent was recrystallized several times from an ether–pentane mixture. It melted at 161–162° and the analysis indicated it to be *p*-thiocarbamidophenyl carbanilate.

(3) Arthur P. Richardson, University of Tennessee, O. S. R. D. Contract OEMcmr 481.

(4) Melting points are uncorrected.

(5) Jorg, *Ber.*, **60B**, 1466 (1927).

(6) Hartmann, *J. prakt. Chem.*, **16**, 50 (1877).

(7) Schreinemakers, *Rec. trav. chim.*, **16**, 424 (1897).

(8) Adams and Levine, THIS JOURNAL, **45**, 2377 (1923).

(9) Hartmann, *J. prakt. Chem.*, **16**, 50 (1877).

(10) Montmollin and Spieler, U. S. Patent 1,474,928 (1928).

(11) Raiford and Inman, THIS JOURNAL, **56**, 1586 (1934).

(12) Walker, *J. Chem. Soc.*, **691** (1940).

Anal. Calcd. for $C_{14}H_{12}N_2O_2S$: N, 10.29; S, 11.77. Found: N, 10.31; S, 11.44.

Acknowledgments.—The authors wish to thank Mr. E. F. Shelberg of the Micro-analytical Department for the micro-analyses herein reported. The assistance of Messrs. Robert J. Hathaway and Charles J. Strickler in the preparation of some of the products is gratefully acknowledged.

Summary

The preparation and properties of thirty-seven carbamates of varying complexity are described. Moderate antimalarial activity was observed in a number of these, two of the more active ones being *p*-carbobutoxyphenyl *p'*-methoxycarbanilate (SN 1048) and *p*-sulfamylphenyl *p'*-methoxycarbanilate (SN-4178).

RECEIVED JUNE 11, 1948

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, THE UNIVERSITY OF TEXAS]

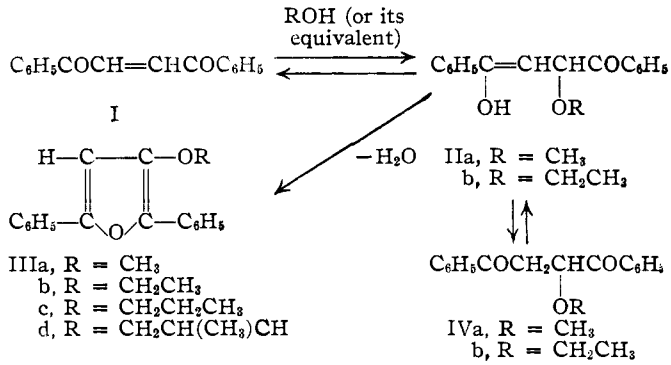
The Reaction of Alcohols with 1,2-Dibenzoylethylene. II.[1,2,3,4]

BY PHILIP S. BAILEY AND JOE T. KELLY[5]

The reaction of alcohols with 1,2-dibenzoylethylene (I) in the presence of an amine hydrochloride and a trace of hydrogen chloride to give 3-alkoxy-2,5-diphenylfurans (III) has been given further study. Previously, the reaction had been carried out with methanol, ethanol and isopropyl alcohol.[4] It has now been found that *n*-propanol and isobutyl alcohol will give the reaction,[6] but the other three isomeric butyl alcohols and *n*-amyl alcohol will not react with dibenzoylethylene to give alkoxyfurans to any appreciable extent. Phenol also failed to give the reaction, an intractable oil being obtained. Apparently the length of the chain as well as the complexity of the alcohol is important in determining whether or not reaction will occur.

In the earlier work on this reaction, the ratio by weight of triethylamine hydrochloride to dibenzoylethylene employed was 1:1.[4] Later work, with the ethanol reaction, has shown that with a 1:10 ratio no decrease in yield of alkoxyfuran is obtained, but with a 1:100 ratio the yield of alkoxyfuran is decreased by about 95%.

One would assume that the alkoxyfurans (III) are obtained from dibenzoylethylene (I) by the equilibrium reactions shown involving structures I, II, III and IV, in which the reaction is probably initiated by the attack of some cation of the reaction mixture upon the oxygen of the diketone. Evidence in favor of this equilibrium was obtained



when IVa was placed in a methanolic reaction mixture in place of dibenzoylethylene (I) under identical conditions, and the mixture was refluxed for the usual length of time. The products isolated were dibenzoylethylene (I), the methoxyfuran (IIIa) (in approximately the same yield as obtained from dibenzoylethylene) and an oily material. Where the high melting[4,7] and oily by-products fit into this picture is uncertain, since their structures are as yet undetermined. Alkoxy diketones (IV) have never been isolated from the dibenzoylethylene–alcohol reaction mixtures refluxed for one, five, ten, twenty-four or sixty hours. However, this is not surprising, since the only one of these compounds known, IVa, is extremely hard to crystallize.

Kohler[8] has found that ethanol will add to

(1) Constructed from the thesis submitted by Mr. Joe T. Kelly to the Graduate Faculty of The University of Texas in partial fulfillment of the requirements for the degree of Master of Arts, January, 1948.

(2) Presented at the Southwest Regional Meeting of the American Chemical Society, Houston, Texas, December 13, 1947.

(3) This work was supported in part by grants from The University Research Institute, Project No. 70.

(4) Paper I, Bailey and Lutz, THIS JOURNAL, **69**, 498 (1947).

(5) Present address: Pan-American Refining Co., Texas City, Texas.

(6) Attempts to prove the structures of the products, 2,5-diphenyl-3-*n*-propoxyfuran and 3-*i*-butoxy-2,5-diphenylfuran by the same general method used in proving the structure of the ethoxyfuran (IIIb) (ref. 4, *i. e.*, treatment of 1,2-dibenzoyl-1,2-dibromoethane with the desired sodium alkoxide, followed by reductive furanization of the resulting 1-alkoxy-1,2-dibenzoylethylene) failed in the first step, from which a resinous material was obtained. Likewise, 2,5-diphenyl-3-*i*-propoxyfuran could not be made by this method (ref. 4). There can be little doubt of the structures of these compounds, however, since they were obtained from reactions identical with those which yielded the corresponding methoxy and ethoxyfurans. Also, the melting points of the alkoxyfurans (Me, 114–115°; Et, 94–95°; Pr, 86–87°, *i*-Pr, 87–88°; *i*-Bu, 71–72°) are in harmony with the proposed structure.

(7) The fact that the high melting by-products (ref. 4) were not isolated in the last experiment described is probably not significant in view of the small amount of methoxydiketone (IVa) employed and the small amounts of dibenzoylethylene (I) and the methoxyfuran (IIIa) produced.

(8) Kohler, *Am. Chem. J.*, **42**, 375 (1909).