($C_7H_6NS_2^+$, 41%), 164 ($C_9H_{10}NS^+$, 25%), 143 ($C_5H_5NS_2^+$, 50%), 110 ($C_5H_4NS^+$, 77%). Due to the limited quantity of sample, further spectral studies were not conducted.

The mother liquor from fraction A was concentrated to afford the 2:1 compound **25:** mp 56–58 °C; NMR δ 1.77–1.95 (m, β-CH_2, 4 H), 3.12–3.30 (m, α-CH_2, 4 H), 6.91 (dd, 3- or 5-pyr H, J = 7.9, 1.0 Hz, 2 H), 7.01 (dd, 5- or 3-pyr H, J = 7.9, 1.0 Hz, 2 H), 7.34 (t, 4-pyr H, J = 7.9 Hz, 2 H); IR (KBr) 2990, 1540, 1245, 1130 cm^{-1}.

Fraction B was recrystallized from diethyl ether and hexane to afford the 3:2 open-chain compound **26:** mp 55–56 °C; NMR ($CDCl_3$) δ 1.77–1.96 (m, all β-CH_2, 8 H), 3.10–3.29 (m, all α-CH_2, 8 H), 6.80 (d, 3- or 5-pyr H, J = 8.2 Hz, 2 H), 6.92 (dd, 3′- or 5′-pyr H, J = 7.7, 0.9 Hz, 2 H), 7.01 (dd, 5′- or 3′-pyr H, J = 7.7, 0.9 Hz, 2 H), 7.2 (t, 4-pyr H, J = 8.2 Hz, 1 H), 7.35 (t, 4′-pyr H, J = 7.7 Hz, 2 H); IR (KBr) 2915, 1535, 1395, 1125 cm^{-1}.

Fraction C was recrystallized from hexane and acetone to afford the 4:3 compound **27:** mp 80.5–82 °C; NMR ($CDCl_3$) δ 1.77–1.94 (m, all β-CH_2, 12 H), 3.10–3.27 (m, all α-CH_2, 12 H), 6.79 (d, 3- or 5-pyr H, J = 8.2 Hz, 2 H), 6.80 (d, 5- or 3-pyr H, J = 8.0 Hz, 2 H), 6.92 (dd, 3′- or 5′-pyr H, J = 7.8, 0.9 Hz, 2 H), 7.01 (dd, 3′- or 5′-pyr H, J = 7.8, 0.9 Hz, 2 H), 7.20 (dd, 4-pyr H, J = 8.2, 8.0 Hz, 2 H), 7.34 (t, 4′-pyr H, J = 7.8 Hz, 2 H); IR (KBr) 2920, 1560, 1380, 1130 cm^{-1}.

Acknowledgment. The authors gratefully acknowledge partial support of this work by a Public Health Service grant from the National Institutes of Health, National Science Foundation, and the Merck Sharp and Dohme Company.

Registry No.—**4a,** 109-09-1; **4b,** 109-04-6; **5,** 66119-95-7; **6,** 66119-96-8; **7a,** 2402-78-0; **7b,** 626-05-1; **8,** 54945-37-8; **9a,** 66119-97-9; **9b,** 66119-98-0; **10,** 66119-99-1; **11a,** 66120-00-1; **11b,** 66120-01-2; **12a,** 66120-02-03; **12b,** 66120-03-4; **13,** 66120-04-5; **14a,** 66120-05-6; **15a,** 66120-06-7; **15b,** 66119-82-2; **16,** 66119-83-3; **17,** 66119-84-4; **18,** 66119-85-5; **20,** 66119-86-6; **21,** 4262-06-0; **22,** 66119-87-7; **23a,** 66119-88-8; **23b,** 66119-89-9; **23c,** 66119-90-2; **24,** 66119-91-3; **25,** 66119-92-4; **26,** 66119-93-5; **27,** 66119-94-6; bis(2-mercaptoethyl) ether, 2150-02-9; bis(2-mercaptoethyl) sulfide, 3570-55-6; 1,2-ethanedithiol, 540-63-6; 1,4-butanedithiol, 1191-08-8.

Supplementary Material Available: All analytical data for the new compounds in Table II (2 pages). Ordering information is given on any current masthead page.

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A Convenient Synthesis of Tertiary Alkyl *N*-Phenylcarbamates from Tertiary Alcohols and Phenyl Isocyanate with a Lithium Alkoxide Catalyst[1]

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Received November 23, 1977

Although the direct addition of tertiary alcohols to isocyanates usually gives no reaction at low temperatures and produces olefins on being heated, the use of catalysts, such as lithium alkoxides and dibutyltin diacetate, makes possible the synthesis of tertiary alkyl *N*-phenylcarbamates in good yields. Thus the addition of *tert*-amyl alcohol to phenyl isocyanate in the presence of lithium *tert*-amyloxide gave *tert*-amyl *N*-phenylcarbamate in an 81% yield. For comparison the same reaction in the presence of dibutyltin diacetate gave a 60% yield of the carbamate and the uncatalyzed reaction gave a 15% yield. The addition of *tert*-butyl alcohol to phenyl isocyanate in the presence of lithium *tert*-butoxide gave an 82% yield of *tert*-butyl *N*-phenylcarbamate. By the same technique the *N*-phenylcarbamates of 1,1-diphenylethanol, 2-phenyl-2-propanol and 3-ethyl-3-pentanol were prepared in 74, 77, and 39% yields, respectively.

In a program to evaluate various tertiary alkyl oxycarbonyl groups as blocking groups for amines a convenient synthesis of tertiary alkyl *N*-phenylcarbamates was desired. However, a search of the literature indicated that there were no good general methods described for the synthesis of tertiary alkyl derivatives. Since a number of very active catalysts have been

0022-3263/78/1943-2690$01.00/0

Table I. Reaction of Tertiary Alcohols with Phenyl Isocyanate[a]

Tertiary alcohol	Registry no.	Catalyst	Time, min	Temp, °C	Yield of carbamate, %
tert-Amyl	75-84-3	LiOR	25	37	81
		$Bu_2Sn(OAc)_2$	145	25–40	60
		None	145	25–27	15
tert-Butyl	75-65-0	LiOR	10	37	82
		$Bu_2Sn(OAc)_2$	14	32–60	42
		None	1440	25	27
2-Phenyl-2-propanol	617-94-7	LiOR	55	25–36	77
1,1-Diphenylethanol	1883-32-5	LiOR	30	37	74
3-Ethyl-3-pentanol	597-49-9	LiOR	30	25–60	39[8]

[a] Registry no. 103-71-9.

reported for use in the synthesis of polyurethanes, it was hoped that one of these catalysts would increase the rate of addition of tertiary alcohols to isocyanates sufficiently to make possible a convenient synthetic procedure.

Davis and Farnum[3] reported that the relative rates of the uncatalyzed reactions of primary, secondary, and tertiary alcohols with phenyl isocyanate had the relative ratios of 100:33:1, respectively. The primary alcohols react fairly rapidly at room temperature with phenyl isocyanate, while the secondary alcohols usually must be warmed before they will react rapidly. On the other hand, most of the tertiary alcohols react slowly, even at 100 °C, and the main product is not the carbamate but the corresponding olefin. For example, Neuberg and Kansky[4] reported the reaction of *tert*-butyl and *tert*-amyl alcohols with 1-naphthyl isocyanate produced the *tert*-butyl *N*-1-naphthylcarbamate in a 35% yield and the *tert*-amyl derivative in a 3.4% yield. There are scattered reports in the literature of attempts to use isocyanates with tertiary alcohols, but in no case were any yields of products given.

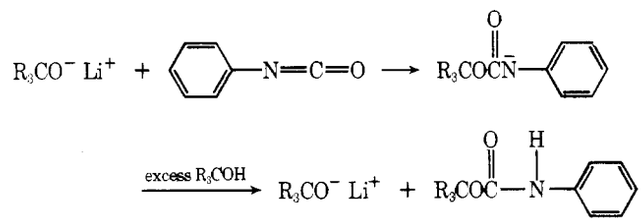
Although there have been many reports concerning the use of basic catalysts for the isocyanate–alcohol reaction, the disclosure by Cox and Hostettler[5] that organo–tin compounds are very effective as catalysts for the primary alcohol–isocyanate reaction led to the speculation that it might also increase the rate of addition with tertiary alcohols. Thus, they had shown that dibutyltin diacetate had a catalytic effect on the phenyl isocyanate–methanol reaction which was 2400 times as great as the activity of triethylamine. Since the organo–tin compounds are amphoteric, they apparently combine the action of the acid catalysts with that of the medium strength base catalyst to produce a synergistic effect.

tert-Butyl alcohol appears to be the exception to the general case of tertiary alcohols, in that its N-aryl carbamates can be formed readily and in fair yields without catalysts.[4] For this reason the effect of dibutyltin diacetate on the reaction of *tert*-butyl alcohol with phenyl isocyanate was determined for the addition reaction in both dibutyl ether and without a solvent. The qualitative effects of the catalyst were determined by treatment of one of two similar portions of *tert*-butyl alcohol and isocyanate with the catalyst and by the use of a spectrophotometer to follow the disappearance of the isocyanate absorption band at 4.5 μm. On a preparative scale the reaction of *tert*-butyl alcohol and phenyl isocyanate in the presence of dibutyltin diacetate gave a 42% yield of the *tert*-butyl *N*-phenylcarbamate after the reaction mixture was allowed to stand for 15 min, while the uncatalyzed reaction gave a 27% yield after being allowed to stand for 18 h.

Unlike *tert*-butyl alcohol, *tert*-amyl alcohol does not react with phenyl isocyanate at a satisfactory rate in the absence of a catalyst. Although the dibutyltin diacetate catalyst did not greatly accelerate the reactions of *tert*-amyl alcohol, the catalyzed reaction is still much more rapid then the uncatalyzed reaction. When the reaction was carried out on a preparative scale with dried *tert*-amyl alcohol in the presence of the organotin catalyst, a 60% yield of the *tert*-amyl *N*-phenylcarbamate was obtained.

Since Tarbell[6] had shown that the catalytic power of the basic catalysts was related to the relative basicities as determined by Hall,[7] we thought it would be of interest to study much stronger bases as catalysts for this reaction. Although the early investigators showed that alkoxides are very effective catalysts for the trimerization, dimerization, and polymerization of phenylisocyanate, these easily prepared polar bases have been apparently neglected as catalysts for other isocyanate reactions. For these reasons we decided to investigate the use of alkoxides as catalysts for the tertiary alcohol addition to phenyl isocyanate.

One could visualize the reaction of the tertiary alkoxide with an isocyanate in the presence of a large excess of tertiary alcohol to be as follows:



The key to the successful addition appeared to be the use of a small amount of lithium alkoxide in the presence of an excess of the tertiary alcohol. It was expected that the tertiary derivatives would be stable under the basic conditions and prevent the elimination reaction which would produce the olefin. In the choice of the particular alkali metal salt, there were several considerations that appeared to favor lithium over sodium and potassium. Generally, the lithium salts are more soluble in organic solvents and therefore greater concentrations of the alkoxides can be achieved by the use of lithium alkoxides of high molecular weight alcohols.

Lithium *tert*-amyl oxide was found to be an extremely active catalyst for the trimerization of phenyl isocyanate. However, if the isocyanate is added dropwise to an excess of *tert*-amyl alcohol in ethyl ether solution, which also contains a small quantity of the alkoxide, then the ether boils vigorously on each dropwise addition. Thus, the carbamate formation is optimized by the use of an excess of tertiary alcohol to avoid the isocyanate trimerization and the use of ethyl ether as a solvent to keep the reaction mixture cool and thus avoid alcohol dehydration. By the use of this technique an 81% yield of *tert*-amyl *N*-phenylcarbamate was obtained. It was found that sodium and potassium alkoxides are almost as effective as lithium alkoxide in the promotion of a rapid reaction of phenyl isocyanate with a tertiary alcohol; however, lithium hydroxide, which can form if water is present, does not interfere with this reaction.

Since only low yields have been reported for the addition of *tert*-butyl alcohol to phenyl isocyanate, this reaction was

reinvestigated with lithium *tert*-butoxide as a catalyst. To ensure a smooth reaction ether was added to the solution of the lithium alkoxide in excess alcohol and a seed crystal was added after one-third of the isocyanate had been added. By this technique an 82% yield of *tert*-butyl *N*-phenylcarbamate was obtained in less than 10 min.

In order to demonstrate the generality of this synthetic method, the *N*-phenylcarbamates of three other tertiary alcohols, several of which were quite sensitive to acid-catalyzed dehydration, were investigated and the results are listed in Table I. Thus, we have demonstrated that lithium alkoxides can be used as effective catalysts for the synthesis on a preparative scale of *tert*-alkyl *N*-phenylcarbamates by the direct reaction of isocyanates with tertiary alcohols. A qualitative procedure for the preparation of solid derivatives from a wide variety of tertiary alcohols with isocyanates has been reported separately.[1]

Experimental Section[9]

***tert*-Amyl *N*-Phenylcarbamate. A. Addition Reaction Catalyzed by Lithium Alkoxide.** After 0.2 g of lithium metal had been reacted with 60.0 g (0.68 mol) of *tert*-amyl alcohol (dried over calcium hydride), 100 mL of ethyl ether (dried over calcium hydride) was added. From the addition funnel 40.0 g (0.34 mol) of phenyl isocyanate was added dropwise with vigorous stirring just rapidly enough to keep the solution boiling gently. Upon completion of the isocyanate addition the resulting solution was extracted with five 100-mL portions of water. After the ether was removed from the dried solution by evaporation, the residue was dissolved in 50 mL of petroleum ether (bp 30–36 °C); no insoluble residue remained. When the solution was stored at −20 °C overnight, the precipitated carbamate was removed by filtration. Two-thirds of the petroleum ether was removed from the filtrate by evaporation and the concentrated solution was stored at −20 °C for an additional 24 h to give a second crop. The combined precipitates were recrystallized from petroleum ether to yield 56.0 g (81%) of pure *tert*-amyl *N*-phenylcarbamate, mp 42 °C (lit.[10] mp 42 °C).

B. Addition Reaction Catalyzed by Dibutyltin Diacetate. To a homogeneous mixture of 2.75 g (0.025 mol) of phenyl isocyanate and 2.0 g (0.0225 mol) of dried *tert*-amyl alcohol was added 0.05 g (0.0002 mol) of dibutyltin diacetate. The temperature of the mixture slowly rose from 25 to 40 °C over a period of 25 min. After the mixture was allowed to stand for 2 h, it had cooled to room temperature to give a viscous liquid. After the mixture had been allowed to stand overnight, no odor of phenyl isocyanate was noted. When 10 mL of petroleum ether (bp 30–60 °C) was added, an insoluble residue of 0.1 g remained undissolved and was removed by filtration. After the filtrate was stored at −20 °C for 24 h and the resulting solid was collected by filtration and washed with 5 mL of cold petroleum ether, 2.8 g (60%) of *tert*-amyl *N*-phenylcarbamate, mp 43–44 °C, was obtained.

C. Uncatalyzed Addition Reaction. When an experiment nearly identical with that described in B above but omitting the dibutyltin diacetate was performed, a temperature rise of only 2 °C was noted and 0.7 g (15%) of the carbamate was obtained.

***tert*-Butyl *N*-Phenylcarbamate. A. Addition Reaction Catalyzed by Lithium *tert*-Butoxide.** A freshly cut 0.06-g piece of lithium metal was reacted with 14.8 g (0.20 mol) of *tert*-butyl alcohol (dried over calcium sulfate and then calcium hydride). After 35 mL of dry ether was added to form a slightly cloudy solution, a solution of 29.8 g (0.25 mol) of phenyl isocyanate in 35 mL of ether was added dropwise at such a rate as to maintain gentle reflux. After about one-third of the isocyanate solution had been added, the addition was interrupted and a seed crystal of *tert*-butyl *N*-phenylcarbamate was introduced to prevent excessive supersaturation and the resulting uncontrollable reaction. After the mixture had been allowed to stand for an additional 5 min, filtration gave 39.9 g of crude product, mp 100–115 °C. Recrystallization from boiling ligroin gave 31.5 g (82%) of pure *tert*-butyl *N*-phenylcarbamate, mp 135–6 °C (lit.[10] mp 136 °C).

B. Addition Reaction Catalyzed by Dibutyltin Diacetate. To a homogeneous mixture of 2.7 g (0.025 mol) of phenyl isocyanate and 2.0 g (0.025 mol) of thoroughly dried *tert*-butyl alcohol was added 0.05 g (0.0002 mol) of dibutyltin diacetate. When the mixture was shaken rapidly, within 12 min its temperature rose quickly from 32 to 60 °C. At 14 min its temperature rose very sharply, and with much vigor the reacting mass suddenly solidified. The reaction mixture was dissolved completely in about 20 mL of boiling petroleum ether (bp 100–120 °C). When the solution was cooled, 3.9 g (42%) of white crystalline needles of the *tert*-butyl *N*-phenylcarbamate, mp 134–136 °C, separated.

2-Phenyl-2-propyl *N*-Phenylcarbamate. To 13.6 g (0.10 mol) of dry 2-phenyl-2-propanol in 50 mL of dry ether was added 0.03 g of freshly cut lithium metal, which partially dissolved over a 30-min heating period on a steam bath. Over a 10-min period 12.0 g (0.10 mol) of phenyl isocyanate in 25 mL of dry ether was added with vigorous stirring. After the addition was complete, the mixture was heated under reflux for an additional 45 min. When the mixture was cooled to room temperature and allowed to stand for 2 h, the mass crystallized. The ether was removed by evaporation on a steam bath and the residue (27.2 g) was recrystallized from 100 mL of boiling ligroin to give 19.7 g (77%) of 2-phenyl-2-propyl *N*-phenylcarbamate, mp 110–111 °C (lit.[11] mp 113 °C).

1,1-Diphenylethyl *N*-Phenylcarbamate. To 0.5 g of 1,1-diphenylethanol melted in a test tube was added about 0.05 g of freshly cut lithium metal. The resulting solution was added to 9.9 g of a 10.4-g sample (0.053 mol) of 1,1-diphenylethanol in 50 mL of dry ether and the mixture was stirred until homogeneous. With stirring a solution of 7.26 g (0.061 mol) of phenyl isocyanate in 40 mL of dry ether was added rapidly to maintain gentle reflux. After the ether had been removed by evaporation, the crude solid was recrystallized from methanol to yield 12.5 g (74%) of 1,1-diphenylethyl *N*-phenylcarbamate, mp 119–120 °C. Anal. Calcd for $C_{21}H_{19}NO_2$: C, 79.47; H, 6.03. Found: C, 79.70; H, 6.30.

3-Ethyl-3-pentyl *N*-Phenylcarbamate. After 0.05 g of lithium metal had been dissolved in 5.0 g (0.043 mol) of 3-ethyl-3-pentanol, 5.0 mL (5.5 g, 0.05 mol) of phenyl isocyanate was added dropwise with stirring over a period of 15 min. An oily product began to separate when approximately one-half of the isocyanate had been added. Upon complete addition of the isocyanate, 50 mL of petroleum ether (bp 30–60 °C) was added and the mixture was heated to boiling. After 0.9 g of an insoluble residue was removed by filtration, the filtrate was cooled overnight at −20 °C. Filtration gave 6.4 g of crude carbamate which was recrystallized from 20 mL of petroleum ether by cooling at −20 °C to yield 3.9 g (39%) of pure 3-ethyl-3-pentyl *N*-phenylcarbamate, mp 61–62 °C (lit.[6] mp 61 °C).

Registry No.—*tert*-Amyl *N*-phenylcarbamate, 37534-82-0; *tert*-butyl *N*-phenylcarbamate, 3422-01-3; 2-phenyl-2-propyl *N*-phenylcarbamate, 5037-72-9; 1,1-diphenylethyl *N*-phenylcarbamate, 5037-73-0; 3-ethyl-3-pentyl *N*-phenylcarbamate, 66303-76-2.

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