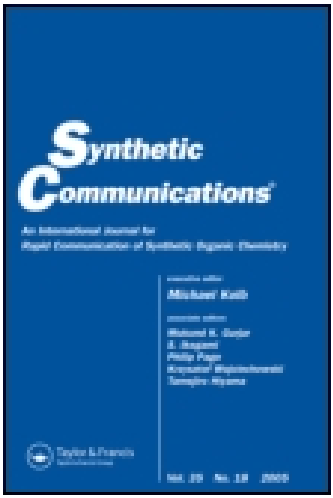


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# Triton-B-Catalyzed, Efficient, One-Pot Synthesis of Carbazates Through Alcoholic Tosylates

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**Abstract:** A quick, efficient, one-pot synthesis of carbazates was accomplished in high yields by the reaction of various tosylates of primary, secondary, and *tert* alcohols, with a variety of substituted hydrazines using the benzyltrimethylammonium hydroxide (Triton-B)/$CO_2$ system. The reaction conditions are mild with simpler workup procedures than the reported methods.

**Keywords:** Alcoholic tosylates, benzyltrimethylammonium hydroxide, carbazates, carbon dioxide, substituted hydrazines

## INTRODUCTION

Organic carbazates have unique applications in the fields of pharmaceuticals[1] and agrochemicals,[2] as intermediates in organic synthesis,[3] for protection of amino groups in peptide synthesis,[4] as linkers in solid-phase organic synthesis,[5] and as donor ligands in complexation reactions with transition metals.[6] To satisfy demand, their synthesis has been changed from the use of costly and toxic chemicals such as phosgene[7] and its derivatives,[8] directly or indirectly, to the abundantly available cheap and safe reagents such as $CO_2$. However, their formation using

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$CO_2$ employed harsh reaction conditions, such as use of strong bases, higher reaction temperatures, and longer reaction times.[9] Thus, we were prompted to embark on improved procedures. Our group[10] has been engaged during the past several years with the development of new methodologies for the preparation of carbamates, dithiocarbamates, and related compounds using cheap, abundantly available, and safe reagents such as $CO_2$ and $CS_2$. Recently,[11] we found that benzyltrimethyl ammonium hydroxide (Triton-B) is the best catalyst for the synthesis of carbamates, dithiocarbamates, and dithiocarbonates (xanthates). We report here an efficient, one-pot synthesis of carbazates from a variety of alcoholic tosylates of primary, secondary, and tertiary alcohols and substituted hydrazines using the Triton-B/$CO_2$ system. The alcoholic tosylates of various alcohols were prepared from the corresponding alcohols using *p*-toluene sulfonyl chloride following the standard procedure.[12]

## RESULTS AND DISCUSSION

Substituted hydrazine was taken in dry dimethylsulfoxide (DMSO) and purified $CO_2$ gas was bubbled in it at 60 °C for 30 min with constant stirring. Triton-B was slowly added with constant stirring. The reaction continued for another 30 min, and then corresponding alcoholic tosylate was added. The reaction was further continued until complete as checked by Thin-Layer Chromatography (TLC; see Table 1). It is proposed that the $O^-$ of the carbazate ion produced will attack the electrophilic carbon of the respective alcoholic tosylates to afford carbazates in high yields (80–98%) at room temperature in 2–4 h, as mentioned in Table 1. The reaction proved to be successful, and the desired products were isolated and their structures confirmed by various spectroscopic and analytical techniques. Thus, various substituted hydrazines were reacted with a variety of alcoholic tosylates of primary, secondary, and tertiary alcohols using the Triton-B/$CO_2$ system to afford the corresponding carbazates in good to excellent yields (Table 1). The reaction conditions are shown in Scheme 1.

We have screened several solvents such as *n*-heptane, *n*-hexane, acetonitrile, benzene, toluene, methanol, dichloromethane, chloroform, DMSO, dimethylformamide, and hexamethylphosphoric triamide, of which dry DMSO proved to be the most suitable at room temperature.

In conclusion, we developed a convenient and efficient protocol for the one-pot, three-component coupling of various substituted hydrazines with a variety of alcoholic tosylates of primary, secondary, and tertiary alcohols via a $CO_2$ bridge using Triton-B. This method generates the

**Table 1.** Conversion of alcoholic toylates into carbazates of formula 1–16

| Entry | $R^1$ | $R^2$ | $R^3$ | R | Time (h) |
|---|---|---|---|---|---|
| 1 | $n$-$C_3H_7$ | H | H | 4-MeO-Ph | 3 |
| 2 | $PhCH_2CH_2$ | H | H | Ph | 3 |
| 3 | $PhCH_2$ | H | H | Ph | 4 |
| 4 | Ph | H | H | Bn | 4.5 |
| 5 | $C_2H_5$ | Me | H | Bn | 4.5 |
| 6 | Ph-4-MeO | H | H | Ph-3-$NO_2$ | 4.5 |
| 7 | $C_3H_7$ | H | H | Ph-4-$NO_2$ | 4.5 |
| 8 | $C_3H_7$ | H | H | Ph-2,4-$NO_2$ | 5 |
| 9 | $C_3H_7$ | H | H | Naphthyl | 4.5 |
| 10 | $C_4H_9$ | $C_4H_9$ | H | Ph | 4.5 |
| 11 | $C_4H_9$ | $C_4H_9$ | $C_4H_9$ | Ph | 4.5 |
| 12 | $C_5H_{11}$ | H | H | $n$-$C_4H_9$ | 3 |
| 13 | $C_7H_{15}$ | H | H | Ph | 3 |
| 14 | $C_9H_{19}$ | H | H | $n$-$C_4H_9$ | 3 |
| 15 | $C_3H_7$ | $C_3H_7$ | H | Ph | 4.5 |
| 16 | Ph | $CH_3$ | H | Ph | 5 |

[a]All the products were characterized by IR, NMR, and mass spectroscopic data.

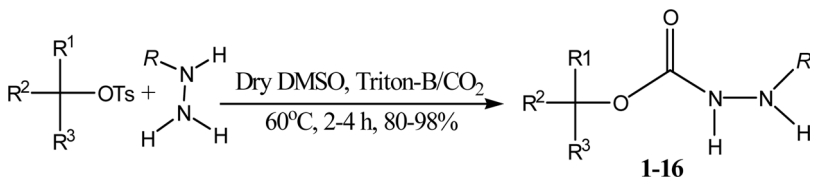
[b]Isolated yields.



corresponding carbazates in good to excellent yields. Furthermore, this method exhibits substrate versatility, mild reaction conditions, and experimental convenience. This synthetic protocol is believed to offer a more general method for the formation of the carbon–oxygen bonds essential to numerous organic syntheses.

## EXPERIMENTAL

Chemicals were procured from Merck, Aldrich, and Fluka chemical companies. Reactions were carried out under an atmosphere of argon. IR spectra (4000–200 $cm^{-1}$) were recorded on a Bomem MB-104 Fourier




***Scheme 1.***

transform infrared (FTIR) spectrophotometer using the neat technique, whereas NMRs were scanned on an AC-300F NMR (300-MHz), instrument using $CDCl_3$ and some other deutrated solvents and TMS as internal standard. Elemental analyses were conducted by means of a Carlo-Erba EA 1110-CNNO-S analyzer and agreed favorably with calculated values.



## Typical Experimental Procedure

To a stirred solution (under Ar) of 3 mmol of substituted hydrazine in 5 $cm^3$ of anhyd. DMSO, 2 $cm^3$ Triton-B were slowly added and carbon dioxide was continuously bubbled in it at 60 °C. Then the mixture was stirred for 0.5 h, at which point 3 $cm^3$ of the required alkyl halide was added over a period of 5 min. The stirring was further continued until the completion of the reaction (cf. Table 1). The reaction mixture was poured into 20 $cm^3$ of water, and the organic layer was extracted with $3 \times 10$ $cm^3$ EtOAc. The organic layer was washed with 20 $cm^3$ of 0.1 *N* HCl, 25 $cm^3$ of saturated solution of $NaHCO_3$, and 30 $cm^3$ of brine, and then dried ($Na_2SO_4$) and concentrated to get the desired compound.

## Data

*N*′-(4-Methoxyphenyl) Hydrazine Carboxylic Acid Butyl Ester (**1**, $C_{12}H_{18}N_2O_3$)

Yield: 94%; mp: oil; IR (neat) $\ddot{\upsilon} = 1680\ cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta = 0.96$ (t, 3H, $J = 7.3$ Hz), 1.34 (m, 2H), 1.86 (m, 2H), 3.73 (s, 3H), 4.12 (t, 2H), 4.85 (m, NH), 6.74–7.66 (m, 4H), 8.0 (br, NH); $^{13}C$ NMR ($CDCl_3$) $\delta = 13.7$, 19.5, 32.5, 63.5, 112.5, 114.9, 134.5, 152.4, 160.6 (C=O) ppm; MS (EI): $m/z = 238$.

*N*′-Phenyl Hydrazine Carboxylic Acid 3-Phenyl Propyl Ester (**2**, $C_{16}H_{18}N_2O_2$)

Yield: 96%; mp: oil; IR (neat) $\ddot{\upsilon} = 1685\ cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta = 1.92$ (m, 2H), 2.56 (t, 2H, $J = 7.2$ Hz, Ph.$CH_2$), 4.10 (t, 2H), 4.67 (br, N*H*Ph), 6.66–7.12 (m, 10H, Ar-H), 8.0 (br, NH); $^{13}C$ NMR ($CDCl_3$), $\delta = 32.4$, 34.4, 63.5, 112.6, 119.4, 125.7, 128.8, 129.6, 138.7, 161 (C=O) ppm; MS: $m/z = 270$.

*N′*-Phenyl Hydrazine Carboxylic Acid Phenethyl Ester (**3**, $C_{15}H_{16}N_2O_2$)

Yield: 87%; mp: oil; IR (neat) $\ddot{\upsilon} = 1681\,cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta = 2.83$ (2H, t, $J = 6.7$ Hz, Ph.$CH_2CH_2O$), 4.42 (t, 2H, $J = 7.2$ Hz, Ph$CH_2O$), 4.77 (br, H, Ph*NH*), 6.69–7.15 (m, 10H, Ar-H), 8.05 (br, NH); $^{13}C$ NMR ($CDCl_3$), $\delta = 35.5$, 65.9, 112.3, 118.6, 128.5, 129.5, 140.3, 142.5, 165.4 (C=O) ppm; MS: $m/z = 256$.

*N′*-Butyl Hydrazine Carboxylic Acid Benzyl Ester (**4**, $C_{12}H_{18}N_2O_2$)

Yield: 92%; mp: oil; IR (neat) $\ddot{\upsilon} = 1680\,cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta = 0.99$ (t, 3H, $CH_3$), 1.34 (m, 2H, $CH_2CH_3$), 1.56 (m, 2H, $CH_2 \cdot CH_2CH_3$), 2.15 (br, NH), 2.66 (m, 2H, NH$CH_2$), 5.13 (s, 2H, Ph$CH_2$), 7.10–7.19 (m, 5H, Ar-H), 8.0 (br, NH); $^{13}C$ NMR ($CDCl_3$), $\delta = 13.8$, 20.3, 31.6, 51.2, 69.3, 126.8, 127.6, 128.5, 141.8, 158 (C=O) ppm; MS: $m/z = 222$.

*N′*-Butyl Hydrazine Carboxylic Acid Sec-butyl Ester (**5**, $C_9H_{20}N_2O_2$)

Yield: 90%; mp: oil; IR (neat) $\ddot{\upsilon} = 1681\,cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta = 0.98$ (t, 3H, $CH_3$), 1.15 (t, 3H, $CH_3$), 1.38 (m, 2H, $CH_2 \cdot CH_3$), 1.42 (d, 3H, $CHCH_3$), 1.56 (m, 2H, $CH_3CH_2CH_2$), 2.0 (br, H, NH), 2.66 (m, 2H, NH$CH_2$), 4.20 (m, C*H*$CH_3$), 8.0 (br, NH); $^{13}C$ NMR ($CDCl_3$) $\delta = 8.2$, 13.8, 19.2, 20.5, 29.3, 71.4, 156.9 (C=O) ppm; MS: $m/z = 188$.

*N′*-(3-Nitrophenyl)-hydrazine Carboxylic Acid 4-Methoxy Benzyl Ester (**6**, $C_{15}H_{15}N_3O_5$)

Yield: 85%; mp: oil; IR (neat) $\ddot{\upsilon} = 1682\,cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta = 3.73$ (s, 3H, O$CH_3$), 4.05 (br, H, *NHPh* · O*Me*), 5.34 (s, 2H), 6.66–7.69 (m, 8H, Ar-H), 8.1 (br, NH); $^{13}C$ NMR ($CDCl_3$) $\delta = 69.3$, 107.6, 114.8, 118.8, 128.5, 129.9, 133.6, 143.6, 148.7, 160.6 (C=O) ppm; MS: $m/z = 317$.

*N′*-(4-Nitrophenyl)-hydrazine Carboxylic Acid Butyl Ester (**7**, $C_{11}H_{15}N_3O_4$)

Yield: 85%; mp: oil; IR (neat) $\ddot{\upsilon} = 1682\,cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta = 0.99$ (t, 3H, $CH_3$), 1.36 (m, 2H, $CH_2CH_3$), 1.57 (m, 2H, $OCH_2 \cdot CH_2$), 4.04 (br, N, *NH*Ar$NO_2$), 6.92–8.15 (m, 4H, Ar-H), 8.0 (br, NH); $^{13}C$ NMR ($CDCl_3$) $\delta = 13.8$, 21.7, 32.3, 63.7, 113.5, 124.6, 138.8, 143.3, 159 (C=O) ppm; MS: $m/z = 253$.

*N′*-(2,4-Dinitro-phenyl)hydrazinecarboxylic Acid Butyl Ester (**8**, $C_{11}H_{14}N_4O_6$)

Yield: 80%; mp: oil; IR (neat) $\ddot{\upsilon} = 1681\,cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta = 0.98$ (t, 3H, $CH_3$), 1.36 (m, 2H, $CH_2CH_3$), 1.59 (m, 2H, $SCH_2 \cdot CH_2$), 4.08 (br, N, $NHArNO_2$), 7.19–9.50 (m, 3H, Ar-H), 8.10 (br, NH); $^{13}C$ NMR ($CDCl_3$) $\delta = 13.8$, 19.3, 31.8, 63.8, 113.6, 119.2, 130.2, 132.8, 139.7, 143.3, 160 (C=O) ppm; MS: $m/z = 298$.

*N′*-Naphthalen-2-yl Hydrazine Carboxylic Acid Butyl Ester (**9**, $C_{15}H_{18}N_2O_2$)

Yield: 83%, mp: oil; IR (neat) $\ddot{\upsilon} = 1681\,cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta = 0.96$ (t, 3H, $CH_3$), 1.36 (m, 2H, $CH_2CH_3$), 1.57 (m, 2H, $OCH_2 \cdot CH_2$), 4.05 (br, H, Ar-N*H*), 4.12 (t, 2H), 6.76–7.55 (m, 7H, Ar-H), 8.02 (br, NH); $^{13}C$ NMR ($CDCl_3$) $\delta = 13.9$, 22.1, 32.5, 33.9, 107.4, 117.2, 121.3, 124.5, 126.6, 127.2, 133.5, 142.6, 161 (C=O) ppm; MS: $m/z = 258$.

*N′*-Phenyl-hydrazine Carboxylic Acid 1-Butyl Pentyl Ester (**10**, $C_{16}H_{26}N_2O_2$)

Yield: 89%; mp: oil; IR (neat) $\ddot{\upsilon} = 1682\,cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta = 0.99$ (t, 6H, $CH_3$), 1.33 (m, 4H, $CH_2CH_2CH$), 1.38 (m, 4H, $CH_2CH_3$), 1.54 (m, 4H, $CHCH_2$), 3.95 (t, H, O*CH*), 4.15 (br, H, *NH*Ar), 6.66–7.18 (m, 5H, Ar-H), 8.0 (br, NH); $^{13}C$ NMR ($CDCl_3$) $\delta = 14.3$, 23.1, 28.5, 36.2, 72.7, 112.2, 119.3, 129.0, 142.4, 158 (C=O) ppm; MS: $m/z = 278$.

*N′*-Phenyl-hydrazine Carboxylic Acid 1,1-Dibutyl Pentyl Ester (**11**, $C_{20}H_{34}N_2O_2$)

Yield: 88%; mp: oil; IR (neat) $\ddot{\upsilon} = 1684\,cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta = 0.96$ (t, 9H, $CH_3$), 1.29 (m, 4H, $CH_2CH_2C$), 1.33 (m, 4H, $CH_2CH_3$), 1.50 (m, 4H, $CHCH_2$), 4.0 (br, H, *NH*-Ar), 6.67–7.19 (m, 5H, Ar-H), 8.0 (br, NH); $^{13}C$ NMR ($CDCl_3$) $\delta = 14.3$, 23.5, 26.8, 39.8, 72.4, 112.5, 119.3, 129.6, 142.2, 162 (C=O) ppm; MS: $m/z = 334$.

*N′*-Butyl-hydrazine Carboxylic Acid Hexyl Ester (**12**, $C_{11}H_{24}N_2O_2$)

Yield: 96%; mp: oil; IR (neat) $\ddot{\upsilon} = 1684\,cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta = 0.98$ (t, 6H, $CH_3$), 1.30 (m, 4H, $CH_2CH_2CH_2CH_3$), 1.36 (t, 2H, $CH_2CH_3$),

1.58 (m, 2H, $NHCH_2CH_2$), 1.63 (t, 2H, $CH_2N$), 2.0 (br, 2H, NH), 2.66 (t, 2H, $NHCH_2$), 4.10 (t, 2H, $OCH_2$), 8.0 (br, NH); $^{13}C$ NMR ($CDCl_3$) $\delta = 13.7$, 14.1, 20.2, 23.1, 28.6, 31.5, 32.6, 69.5, 164.5 (C=O) ppm; MS: $m/z = 216$.

*N′*-Phenyl-hydrazine Carboxylic Acid n-Octyl Ester (**13**, $C_{15}H_{24}N_2O_2$)

Yield: 96%; mp: oil; IR (neat) $\ddot{\upsilon} = 1685\,cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta = 0.96$ (t, 3H, $CH_3$), 1.30 (m, 8H, $CH_2$), 1.35 (m, 2H, $CH_2CH_3$), 1.63 (m, 2H, $OCH_2CH_2$), 4.0 (br, H, Ph.*NH*), 4.12 (t, 2H, $OCH_2$), 6.66–7.25 (m, 5H, Ar-H), 8.0 (br, NH); $^{13}C$ NMR ($CDCl_3$) $\delta = 14.5$, 23.10, 27.5, 30.5, 32.5, 63.6, 112.2, 129.6, 118.9, 142.2, 163 (C=O) ppm; MS: $m/z = 264$.

*N′*-Butyl Hydrazine Carboxylic Acid Decyl Ester (**14**, $C_{15}H_{32}N_2O_2$)

Yield: 98%; mp: oil; IR (neat) $\ddot{\upsilon} = 1683\,cm^{-1}$; $^1H$ NMR ($CDCl_3$), $\delta = 0.97$ (s, 3H, $CH_3$), 0.99 (s, 3H, $CH_3$), 1.29 (m, 12H, $CH_2$), 1.34 (m, 4H, $CH_2CH_3$), 1.57 (m, 2H, $CH_2CH_2CH_3$), 2.0 (br, NH), 2.65 (m, 2H, $NHCH_2$), 4.12 (t, 2H, $OCH_2$), 8.0 (br, NH); $^{13}C$ NMR ($CDCl_3$) $\delta = 13.7$, 14.5, 20.3, 23.1, 28.9, 30.6, 30.9, 31.5, 32.5, 63.5, 160 (C=O) ppm; MS: $m/z = 272$.

*N′*-Phenyl Hydrazine Carboxylic Acid 1-Propyl Butyl Ester (**15**, $C_{14}H_{22}N_2O_2$)

Yield: 86%; mp: oil; IR (neat) $\ddot{\upsilon} = 1680\,cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta = 0.97$ (s, 3H, $CH_3$), 1.33 (m, 4H, $CH_2CH_3$), 1.54 (m, 4H, $CHCH_2$), 3.95 (m, H, C*H*-O), 4.1 (br, H, NH-Ar), 6.66–7.22 (m, 5H, Ar-H), 8.0 (br, NH); $^{13}C$ NMR ($CDCl_3$) $\delta = 14.5$, 20.1, 38.4, 72.8, 112.5, 118.3, 129.6, 143.3, 160 (C=O) ppm; MS: $m/z = 250$.

*N′*-Phenyl Hydrazine Carboxylic Acid 1-Phenyl Ethyl Ester (**16**, $C_{15}H_{16}N_2O_2$)

Yield: 82%; mp: oil; IR (neat) $\ddot{\upsilon} = 1682\,cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta = 1.69$ (d, 3H, $CH_3$), 4.2 (br, H, NH-Ph), 5.42 (m, H, *CH*-O), 6.66–7.22 (m, 10H, Ar-H), 8.0 (br, NH); $^{13}C$ NMR ($CDCl_3$) $\delta = 23.4$, 74.2, 112.5, 118.9, 126.5, 128.5, 129.7, 141.3, 142.5, 163.5 (C=O) ppm; MS: $m/z = 256$.

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