Triton-B catalyzed, efficient one-pot synthesis of carbazates

Devdutt Chaturvedi¹, Amit K. Chaturvedi², Nisha Mishra², Virendra Mishra²

¹ Bio-Organic Chemistry Division, Indian Institute of Integrative Medicine, Jammu-Tawi, J&K, India

² Synthetic Research Laboratory, Department of Chemistry, B.S.A.P.G. College, Mathura, U.P., India

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Abstract A quick and efficient, one-pot synthesis of carbazates was accomplished in high yields by the reaction of various primary, secondary, and tertiary alkyl halides with a variety of substituted hydrazines using the benzyltrimethylammonium hydroxide (Triton-B)/CO₂ system. The reaction conditions are mild with simpler work-up procedures than the reported methods.

Keywords Alkyl halides; Benzyltrimethylammonium hydroxide; Carbon dioxide; Substituted hydrazines; Carbazates.

Introduction

Organic carbazates have attracted much attention due to their numerous remarkable medicinal, industrial, and synthesis applications [1, 2]. They have extensively been used as pharmaceuticals [3], agrochemicals [4], intermediates in organic synthesis [5], for protection of amino groups in peptide synthesis [6], as linkers in solid phase organic synthesis [7], and as donor ligands in complexation reactions with transition metals [8]. To satisfy their demand, their synthesis has been changed from the use of costly and toxic chemicals like phosgene [9] and its derivatives [10] directly or indirectly, to the abundantly available cheap and safe reagents like CO_2 . However, their formation using CO_2 employed harsh reaction conditions, such as use of strong bases, high reaction temperatures, and long reaction times [11]. Thus, we were prompted to embark on the improved procedures. Our group [12] has been engaged during the past several years in the development of new methodologies for the preparation of carbamates, dithiocarbamates, and related compounds using cheap, abundantly available, and safe reagents like CO₂ and CS₂. Recently [13], we have found that benzyltrimethylammonium 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 primary, secondary, and tertiary alkyl halides and substituted hydrazines using the Triton- B/CO_2 system.

Results and discussion

Substituted hydrazine was taken in dry *DMSO* and purified CO₂ gas was bubbled in it at 60°C for 30 min with constant stirring. Now, Triton-B was slowly added with constant stirring. The reaction was continued for another 30 min, and then the corresponding alkyl halide was added. The reaction was further continued until completion as checked by TLC (see Table 1). It is proposed that the O⁻ of the carbazate ion produced will attack the electrophilic carbon of the respective alkyl halide to afford carbazates in high yields (80–98%) at room temperature in 3–5 h, as mentioned in Table 1. The reaction proved to be successful and the desired products

Correspondence: Devdutt Chaturvedi, Bio-Organic Chemistry Division, Indian Institute of Integrative Medicine, Canal Road, Jammu-Tawi 18000, J&K, India. E-mail: dchaturvedi002@yahoo.co.in

Product	R^1	R^2	R^3	Х	R	Time/h	Yields/% ^b
1	<i>n</i> -C ₃ H ₇	Н	Н	Br	4-MeO-Ph	3	94
2	$PhCH_2CH_2$	Н	Н	Br	Ph	3	96
3	$PhCH_2$	Н	Н	Cl	Ph	4	87
4	Ph	Н	Н	Cl	Bn	4.5	92
5	C_2H_5	Me	Н	Br	Bn	4.5	90
6	Ph-4-MeO	Н	Н	Cl	Ph-3-NO ₂	4.5	85
7	C_3H_7	Н	Н	Br	$Ph-4-NO_2$	4.5	85
8	C_3H_7	Н	Н	Br	<i>Ph</i> -2,4-NO ₂	5	80
9	C_3H_7	Н	Н	Br	Naphthyl	4.5	83
10	C_4H_9	C_4H_9	Н	Br	Ph	4.5	89
11	C_4H_9	C_4H_9	C_4H_9	Br	Ph	4.5	88
12	$C_{5}H_{11}$	Н	Н	Cl	$n-C_4H_9$	3	96
13	$C_{7}H_{15}$	Н	Н	Cl	Ph	3	96
14	$C_{9}H_{19}$	Н	Н	Cl	$n-C_4H_9$	3	98
15	C_3H_7	C_3H_7	Н	Br	Ph	4.5	86
16	Ph	CH_3	Н	Br	Ph	5	82

Table 1 Conversion of alkyl halides into carbazates 1-16^a

^a All the products were characterized by IR, NMR, and mass spectroscopic data

^b Isolated yields





were isolated and their structures confirmed by various spectroscopic and analytical techniques. Thus, various substituted hydrazines were reacted with a variety of primary, secondary, and tertiary alkyl halides using the Triton-B/CO₂ system to afford the corresponding carbazates in good to excellent yields (Table 1) with the reaction conditions as shown in Scheme 1.

We tried several solvents like *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 one 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 primary, secondary, and tertiary alkyl halides *via* a CO_2 bridge using Triton-B. This method generates the corresponding carbazates in good to excellent yields. Furthermore, this method exhibits substrate versatility, mild reaction conditions, and

experimental convenience. This synthesis protocol developed is believed to offer a more general method for the formation of carbon–oxygen bonds essential to numerous organic syntheses.

Experimental

Chemicals were procured from Merck, Aldrich, and Fluka chemical companies. Reactions were carried out under Argon. IR spectra ($4000-200 \text{ cm}^{-1}$) were recorded on a Bomem MB-104-FTIR spectrophotometer using neat technique, whereas NMRs were scanned on an AC-300F NMR (300 MHz) instrument using CDCl₃ and *TMS* as internal standard. Elemental analysis 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 substituted hydrazine in 5 cm³ anhyd. *DMSO* carbon dioxide was continuously bubbled and 2 cm³ Triton-B were added at 60°C. Then the mixture was stirred for 0.5 h at which point 3 cm³ of the required alkyl halide were added over a period of 5 min. The stirring was further continued until the completion of reaction (*cf.* Table 1). The reaction mixture was poured into 20 cm^3 water and the organic layer was extracted with $3 \times 10 \text{ cm}^3 \text{ EtOAc}$. The organic layer was washed with 20 cm^3 0.1 *N* HCl, 25 cm³ saturated NaHCO₃ solution, 30 cm^3 brine, and then dried (Na₂SO₄) and concentrated to get the desired compound.

N'-(4-Methoxyphenyl)hydrazine carboxylic acid butyl ester (1, C₁₂H₁₈N₂O₃)

Oil; IR (neat): $\bar{\nu} = 1680 \text{ cm}^{-1}$; ¹H NMR (CDCl₃): $\delta = 0.96$ (t, 3H, J = 7.3 Hz), 1.34 (m, 2H), 1.86 (m, 2H), 3.73 (s, 3H), 4.12 (t, J = 6.5 Hz, 2H), 4.85 (m, NH), 6.74–7.66 (m, 4H), 8.0 (br, NH) ppm; ¹³C NMR (CDCl₃): $\delta = 13.7$, 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'-Phenylhydrazine carboxylic acid 3-phenyl propyl ester (**2**, C₁₆H₁₈N₂O₂)

Oil; IR (neat): $\bar{\nu} = 1685 \text{ cm}^{-1}$; ¹H NMR (CDCl₃): $\delta = 1.92$ (m, 2H), 2.56 (t, 2H, J = 7.2 Hz, $-PhCH_2$), 4.10 (t, J = 6.5 Hz, 2H), 4.67 (br, NHPh), 6.66–7.12 (m, 10H, Ar–H), 8.0 (br, NH) ppm; ¹³C NMR (CDCl₃): $\delta = 32.4$, 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'-Phenylhydrazine carboxylic acid phenethyl ester (3, $C_{15}H_{16}N_2O_2$)

Oil; IR (neat): $\bar{\nu} = 1681 \text{ cm}^{-1}$; ¹H NMR (CDCl₃): $\delta = 2.83$ (2H, t, J = 6.7 Hz, PhCH₂CH₂O), 4.42 (t, 2H, J = 7.2 Hz, PhCH₂O), 4.77 (br, H, PhNH), 6.69–7.15 (m, 10H, Ar–H), 8.05 (br, NH) ppm; ¹³C NMR (CDCl₃): $\delta = 35.5$, 65.9, 112.3, 118.6, 128.5, 129.5, 140.3, 142.5, 165.4 (C=O) ppm; MS: m/z = 256.

N'-Butylhydrazine carboxylic acid benzyl ester (4, C₁₂H₁₈N₂O₂)

Oil; IR (neat): $\bar{\nu} = 1680 \text{ cm}^{-1}$; ¹H NMR (CDCl₃): $\delta = 0.99$ (t, J = 7.2 Hz, 3H, CH₃), 1.34 (m, 2H, CH_2 CH₃), 1.56 (m, 2H, CH_2 CH₂CH₃), 2.15 (br, NH), 2.66 (m, 2H, NHCH₂), 5.13 (s, 2H, $PhCH_2$), 7.10–7.19 (m, 5H, Ar–H), 8.0 (br, NH) ppm; ¹³C NMR (CDCl₃): $\delta = 13.8$, 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'-Butylhydrazine carboxylic acid sec-butyl ester (5, C₉H₂₀N₂O₂)

Oil; IR (neat): $\bar{\nu} = 1681 \text{ cm}^{-1}$; ¹H NMR (CDCl₃): $\delta = 0.98$ (t, J = 7.2 Hz, 3H, CH₃), 1.15 (t, J = 7.0 Hz, 3H, CH₃), 1.38 (m, 2H, CH₂CH₃), 1.42 (d, J = 6.5 Hz, 3H, CHCH₃), 1.56 (m, 2H, CH₃CH₂CH₂), 2.0 (br, NH), 2.66 (m, 2H, NHCH₂), 4.20 (m, CHCH₃), 8.0 (br, NH) ppm; ¹³C NMR (CDCl₃): $\delta = 8.2$, 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-methoxybenzyl ester (**6**, C₁₅H₁₅N₃O₅)

Oil; IR (neat): $\bar{\nu} = 1682 \text{ cm}^{-1}$; ¹H NMR (CDCl₃): $\delta = 3.73$ (s, 3H, OCH₃), 4.05 (br, H, NHPhOMe), 5.34 (s, 2H), 6.66–7.69 (m, 8H, Ar–H), 8.1 (br, NH) ppm; ¹³C NMR (CDCl₃):

 $\delta = 69.3$, 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, C₁₁H₁₅N₃O₄)

Oil; IR (neat): $\bar{\nu} = 1682 \text{ cm}^{-1}$; ¹H NMR (CDCl₃): $\delta = 0.99$ (t, J = 7.2 Hz, 3H, CH₃), 1.36 (m, 2H, *CH*₂CH₃), 1.57 (m, 2H, OCH₂*CH*₂), 4.04 (br, *NH*ArNO₂), 6.92–8.15 (m, 4H, *Ar*–H), 8.0 (br, NH) ppm; ¹³C NMR (CDCl₃): $\delta = 13.8$, 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-Dinitrophenyl)hydrazine carboxylic acid butyl ester (8, C₁₁H₁₄N₄O₆)

Oil; IR (neat): $\bar{\nu} = 1681 \text{ cm}^{-1}$; ¹H NMR (CDCl₃): $\delta = 0.98$ (t, J = 7.2 Hz, 3H, CH₃), 1.36 (m, 2H, CH_2 CH₃), 1.59 (m, 2H, SCH₂CH₂), 4.08 (br, *NH*ArNO₂), 7.19–9.50 (m, 3H, *Ar*–H), 8.10 (br, NH) ppm; ¹³C NMR (CDCl₃): $\delta = 13.8$, 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'-Naphth-2-ylhydrazine carboxylic acid butyl ester (**9**, C₁₅H₁₈N₂O₂)

Oil; IR (neat): $\bar{\nu} = 1681 \text{ cm}^{-1}$; ¹H NMR (CDCl₃): $\delta = 0.96$ (t, J = 7.2 Hz, 3H, CH₃), 1.36 (m, 2H, *CH*₂CH₃), 1.57 (m, 2H, OCH₂*CH*₂), 4.05 (br, H, Ar–N*H*), 4.12 (t, J = 7.0 Hz, 2H), 6.76–7.55 (m, 7H, *Ar*–H), 8.02 (br, NH) ppm; ¹³C NMR (CDCl₃): $\delta = 13.9, 22.1, 32.5, 33.9, 107.4, 117.2, 121.3, 124.5, 126.6, 127.2, 133.5, 142.6, 161 (C=O) ppm; MS: <math>m/z = 258$.

N'-Phenylhydrazine carboxylic acid 1-butylpentyl ester (10, $C_{16}H_{26}N_2O_2$)

Oil; IR (neat): $\bar{\nu} = 1682 \text{ cm}^{-1}$; ¹H NMR (CDCl₃): $\delta = 0.99$ (t, J = 7.2 Hz, 6H, CH₃), 1.33 (m, 4H, *CH*₂CH₂CH₂CH), 1.38 (m, 4H, *CH*₂CH₃), 1.54 (m, 4H, CH*CH*₂), 3.95 (t, O*CH*), 4.15 (br, *NH*Ar), 6.66–7.18 (m, 5H, *Ar*–H), 8.0 (br, NH) ppm; ¹³C NMR (CDCl₃): $\delta = 14.3$, 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'-Phenylhydrazine carboxylic acid 1,1-dibutylpentyl ester (11, C₂₀H₃₄N₂O₂)

Oil; IR (neat): $\bar{\nu} = 1684 \text{ cm}^{-1}$; ¹H NMR (CDCl₃): $\delta = 0.96$ (t, J = 7.2 Hz, 9H, CH₃), 1.29 (m, 4H, *CH*₂CH₂C), 1.33 (m, 4H, *CH*₂CH₃), 1.50 (m, 4H, CH*CH*₂), 4.0 (br, H, *NH*–Ar), 6.67–7.19 (m, 5H, *Ar*–H), 8.0 (br, NH) ppm; ¹³C NMR (CDCl₃): $\delta = 14.3$, 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'-Butylhydrazine carboxylic acid hexyl ester

$(12, C_{11}H_{24}N_2O_2)$

Oil; IR (neat): $\bar{\nu} = 1684 \text{ cm}^{-1}$; ¹H NMR (CDCl₃): $\delta = 0.98$ (t, J = 7.0 Hz, 6H, CH₃), 1.30 (m, 4H, $CH_2CH_2CH_2CH_3$), 1.36 (t, J = 7.0 Hz, 2H, CH_2CH_3), 1.58 (m, 2H, NHCH_2CH_2), 1.63 (t, J = 6.5 Hz, 2H, CH_2N), 2.0 (br, 2H, NH), 2.66 (t, 2H, NHCH_2), 4.10 (t, 2H, J = 7.2 Hz, OCH_2), 8.0 (br, NH) ppm; ¹³C NMR (CDCl₃): $\delta = 13.7$, 14.1, 20.2, 23.1, 28.6, 31.5, 32.6, 69.5, 164.5 (C=O) ppm; MS: m/z = 216.

N'-Phenylhydrazine carboxylic acid n-octyl ester (13, C₁₅H₂₄N₂O₂)

Oil; IR (neat): $\bar{\nu} = 1685 \text{ cm}^{-1}$; ¹H NMR (CDCl₃): $\delta = 0.96$ (t, J = 7.2 Hz, 3H, CH₃), 1.30 (m, 8H, CH₂), 1.35 (m, 2H, CH₂CH₃), 1.63 (m, 2H, OCH₂CH₂), 4.0 (br, Ph*NH*), 4.12 (t, 2H, J = 7.0 Hz, OCH₂), 6.66–7.25 (m, 5H, A*r*–H), 8.0 (br, NH) ppm; ¹³C NMR (CDCl₃): $\delta = 14.5$, 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'-Butylhydrazine carboxylic acid decyl ester

 $(14, C_{15}H_{32}N_2O_2)$

Oil; IR (neat): $\bar{\nu} = 1683 \text{ cm}^{-1}$; ¹H NMR (CDCl₃): $\delta = 0.97$ (s, 3H, CH₃), 0.99 (s, 3H, CH₃), 1.29 (m, 12H, CH₂), 1.34 (m, 4H, *CH*₂CH₃), 1.57 (m, 2H, *CH*₂CH₂CH₃), 2.0 (br, NH), 2.65 (m, 2H, NH*CH*₂), 4.12 (t, *J* = 7.0 Hz, 2H, O*CH*₂), 8.0 (br, NH) ppm; ¹³C NMR (CDCl₃): $\delta = 13.7$, 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'-Phenylhydrazine carboxylic acid 1-propylbutyl ester (**15**, C₁₄H₂₂N₂O₂)

Oil; IR (neat): $\bar{\nu} = 1680 \text{ cm}^{-1}$; ¹H NMR (CDCl₃): $\delta = 0.97$ (s, 3H, CH₃), 1.33 (m, 4H, *CH*₂CH₃), 1.54 (m, 4H, CH*CH*₂), 3.95 (m, H, *CH*–O), 4.1 (br, H, NH–*Ar*), 6.66–7.22 (m, 5H, *Ar*–H), 8.0 (br, NH) ppm; ¹³C NMR (CDCl₃): $\delta = 14.5$, 20.1, 38.4, 72.8, 112.5, 118.3, 129.6, 143.3, 160 (C=O) ppm; MS: m/z = 250.

N'-Phenylhydrazine carboxylic acid 1-phenyl ethyl ester (16, $C_{15}H_{16}N_2O_2$)

Oil; IR (neat): $\bar{\nu} = 1682 \text{ cm}^{-1}$; ¹H NMR (CDCl₃): $\delta = 1.69$ (d, J = 7.2 Hz, 3H, CH₃), 4.2 (br, NH–Ph), 5.42 (m, *CH*–O), 6.66–7.22 (m, 10*Ar*–H), 8.0 (br, NH) ppm; ¹³C NMR (CDCl₃): $\delta = 23.4$, 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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