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

Devdutt Chaturvedi[1], Nisha Mishra[2], Virendra Mishra[2]

[1] Medicinal and Process Chemistry Division, Central Drug Research Institute, Lucknow, U.P., India
[2] Synthetic Research Laboratory, Department of Chemistry, B.S.A. College, Mathura, U.P., India

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Abstract A quick and efficient, one-pot synthesis of dithiocarbazates was accomplished in high yields by the reaction of various primary, secondary, and *tert.* alkyl halides with a variety of substituted hydrazines using the benzyl-trimethylammonium hydroxide (Triton-B)/CS_2 system. The reaction conditions are mild with simpler work-up procedures than the reported methods.

Keywords Alkyl halides; Benzyltrimethylammonium hydroxide; Carbon disulfide; Substituted hydrazines; Dithiocarbazates.

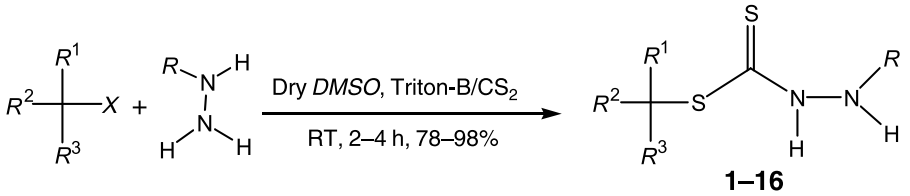
Introduction

Organic dithiocarbazates have received much attention due to their numerous remarkable medicinal, industrial, and synthetic applications [1, 2]. They have extensively been used as pharmaceuticals [3], agrochemicals [4], intermediates in organic synthesis [5], protection of amino groups in peptide synthesis [6], 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 thiophosgene [9] and its derivatives [10] directly or indirectly, to the abundantly available cheap and safe reagents like CS_2. However, their formation using CS_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 for the development of new methodologies for the preparation of carbamates, dithiocarbamates, and related compounds using cheap, abundantly available, and safe reagents like CO_2, and CS_2. Recently [13], 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 dithiocarbazates from a variety of primary, secondary, and tertiary alkyl halides and substituted hydrazines using the Triton-B/CS_2 system.

Results and discussion

A mixture of substituted hydrazine and CS_2 were taken in dry *DMSO* and Triton-B was added. The reaction was stirred for 30 min at room temperature, and then corresponding alkyl halide was added. The reaction was further continued until completion as checked by TLC (see Table 1). It is proposed that the S^- of the dithiocarbazate ion produced will attack to the electrophilic carbon of the respective alkyl halide to afford dithiocarbazates in high yields (78–98%) at room temperature in 2–4 h, as mentioned in Table 1. The reaction proved to be

Correspondence: Devdutt Chaturvedi, Medicinal and Process Chemistry Division, Central Drug Research Institute, Lucknow-226001, U.P., India. E-mail: ddchaturvedi002@yahoo.co.in



Scheme 1

Table 1 Conversion of alkyl halides into dithiocarbazates of formula **1–16**

Entry	R^1	R^2	R^3	X	R	Time/h
1	n-C_3H_7	H	H	Br	4-*MeO–Ph*	2
2	*Ph*CH_2CH_2	H	H	Br	*Ph*	2
3	*Ph*CH_2	H	H	Cl	*Ph*	2.5
4	*Ph*	H	H	Cl	*Bn*	3
5	C_2H_5	*Me*	H	Br	*Bn*	3
6	*Ph*-4-*Me*O	H	H	Cl	*Ph*-3-NO_2	3
7	C_3H_7	H	H	Br	*Ph-4*NO_2	3
8	C_3H_7	H	H	Br	*Ph-2,4-*NO_2	4
9	C_3H_7	H	H	Br	naphthyl	3
10	C_4H_9	C_4H_9	H	Br	*Ph*	3
11	C_4H_9	C_4H_9	C_4H_9	Br	*Ph*	3
12	C_5H_{11}	H	H	Cl	n-C_4H_9	2.5
13	C_7H_{15}	H	H	Cl	*Ph*	2.5
14	C_9H_{19}	H	H	Cl	n-C_4H_9	2
15	C_3H_7	C_3H_7	H	Br	*Ph*	3
16	*Ph*	CH_3	H	Br	*Ph*	3.5

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

successful and the desired products were isolated and their structures confirmed by various spectroscopic and analytical techniques. The whole reaction conditions are shown in Scheme 1.

We tried several solvents like *n*-heptane, *n*-hexane, acetonitrile, benzene, toluene, methanol, dichloromethane, chloroform, *DMSO*, dimethylformamide, hexamethylphosphoric triamide of which dry *DMSO* proved to be most suitable at room temperature.

In conclusion, we developed a convenient and efficient protocol for the one-pot, three components coupling of various amines with a variety of primary, secondary, and tertiary alkyl halides *via* CS_2 bridge using Triton-B. This method generates the corresponding dithiocarbazates 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–sulfur 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 Bomem MB-104-FTIR spectrophotometer using neat technique, where as NMRs were scanned on AC-300F, NMR (300 MHz), instrument using $CDCl_3$ and some other deutrated solvents 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 cm^3 anhyd. *DMSO* was slowly added, 8 cm^3 carbon disulfide, and 2 cm^3 Triton-B at room temperature. 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 till the completion of reaction (*cf* Table 1). The reaction mixture was poured into 20 cm^3 water and organic layer was extracted with 3×10 cm^3 *Et*O*Ac*. The organic layer was washed with 20 cm^3 0.1 *N* HCl, 25 cm^3 saturated solution of $NaHCO_3$, 30 cm^3 brine, and then dried (Na_2SO_4) and concentrated to get the desired compound.

Butyl 2-(4-methoxyphenyl)hydrazinecarbodithioate

(**1**, $C_{12}H_{18}N_2OS_2$)

Yield 93%; mp yellow oil; IR (neat): $\bar{\nu} = 675$, 1210 cm^{-1}; ^{1}H NMR ($CDCl_3$): $\delta = 0.85$ (t, 3H, $J = 7.3$ Hz), 1.33 (m, 2H), 1.85 (m, 2H), 2.0 (s, NH), 2.95 (t, 2H, $J = 6.3$ Hz), 3.73 (s, 3H), 4.05 (m, NH), 6.75–7.60 (m, 4H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 13.5$, 21.8, 32.4, 33.9, 43.7, 55.6, 112.5, 114.9, 134.5, 152.4, 222.5 (C=S) ppm; MS (EI): $m/z = 270$.

3-Phenylpropyl 2-phenylhydrazinecarbodithioate

(**2**, $C_{16}H_{18}N_2S_2$)

Yield 96%; mp yellow oil; IR (neat): $\bar{\nu} = 676$, 1205 cm^{-1}; ^{1}H NMR ($CDCl_3$): $\delta = 2.05$ (s, H, NH), 2.30 (m, 2H, *Ph*·CH_2·*CH_2*·CH_2–S), 2.56 (t, 2H, $J = 7.2$ Hz, *Ph*·*CH_2*), 2.87 (t, 2H, *Ph*·CH_2·CH_2·*CH_2*·S), 4.03 (m, H, *Ph*·*NH*), 6.66–7.12 (m, 10H, *Ar*–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta =$ 32.2, 33.6, 34.4, 112.5, 119.2, 125.8, 128.6, 129.5, 138.6, 221.6 (C=S) ppm; MS: $m/z = 302$.

2-Phenylethyl 2-phenylhydrazinecarbodithioate
(**3**, $C_{15}H_{16}N_2S_2$)
Yield 86%; mp yellow oil; IR (neat): $\bar{\nu} = 673$, 1203 cm^{-1}; ^1H NMR ($CDCl_3$): $\delta = 2.10$ (s, H, NH), 3.20 (2H, t, $J = 6.5$ Hz, $Ph \cdot CH_2CH_2S$), 3.24 (m, 2H, $J = 7.2$ Hz, $PhCH_2$), 4.52 (m, H, *PhNH*), 6.69–7.15 (m, 10H, *Ar*–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 34.5$, 37.3, 47.2, 49.9, 118.6, 192.7, 223.3 (C=S) ppm; MS: $m/z = 288$.

Benzyl 2-butylhydrazinecarbodithioate (**4**, $C_{12}H_{18}N_2S_2$)
Yield 91%; mp yellow oil; IR (neat): $\bar{\nu} = 676$, 1207 cm^{-1}; ^1H NMR ($CDCl_3$): $\delta = 1.05$ (t, 3H, CH_3), 1.33 (m, 2H, CH_2CH_3), 1.56 (m, 2H, $CH_2 \cdot CH_2CH_3$), 2.05 (br, NH), 2.65 (m, 2H, $NHCH_2$), 4.13 (s, 2H, $PhCH_2$), 7.06–7.15 (m, 5H, *Ar*–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 13.7$, 20.2, 31.5, 38.5, 50.9, 126.8, 127.6, 128.5, 141.8, 223.5 ppm; MS: $m/z = 254$.

sec-Butyl 2-butylhydrazinecarbodithioate (**5**, $C_9H_{20}N_2S_2$)
Yield 89%; mp IR (neat): $\bar{\nu} = 682$, 1214 cm^{-1}; ^1H NMR ($CDCl_3$): $\delta = 0.99$ (t, 3H, CH_3), 1.05 (t, 3H, CH_3), 1.35 (m, 2H, $CH_2 \cdot CH_3$), 1.41 (d, 3H, $CHCH_3$), 1.55 (m, 2H, $CH_3CH_2CH_2$), 1.96 (m, 2H, $CHCH_2$), 2.0 (br, H, NH), 2.65 (m, 2H, $NHCH_2$), 2.70 (m, H, *CH*–S) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 10.2$, 13.7, 20.2, 21.5, 31.2, 32.3, 40.1, 49.9, 223.4 ppm; MS: $m/z = 220$.

4-Methoxybenzyl 2-(3-Nitrophenyl)hydrazinecarbodithioate
(**6**, $C_{15}H_{15}N_3O_3S_2$)
Yield 84%; mp yellow oil; IR (neat): $\bar{\nu} = 678$, 1211 cm^{-1}; ^1H NMR ($CDCl_3$): $\delta = 2.05$ (br, H, $NHPh \cdot OMe$), 3.73 (s, 3H, OCH_3), 4.06 (br, H, $NHPh \cdot NO_2$), 6.65–7.66 (m, 8H, *Ar*–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 38.3$, 56.7, 107.5, 114.6, 118.4, 128.5, 129.9, 133.6, 143.6, 148.7, 160.6, 223.2 ppm; MS: $m/z = 349$.

Butyl 2-(4-nitrophenyl)hydrazinecarbodithioate
(**7**, $C_{11}H_{15}N_3O_2S_2$)
Yield 84%; mp yellow oil; IR (neat): $\bar{\nu} = 666$, 1203 cm^{-1}; ^1H NMR ($CDCl_3$): $\delta = 0.96$ (t, 3H, CH_3), 1.33 (m, 2H, CH_2CH_3), 1.96 (m, 2H, $SCH_2 \cdot CH_2$), 2.05 (br, H, N*H*), 2.87 (t, 2H, SCH_2), 4.04 (br, N, $NHArNO_2$), 6.92–8.15 (m, 4H, *Ar*–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 13.7$, 21.6, 32.2, 33.7, 113.5, 124.6, 138.8, 143.3, 223.5 ppm; MS: $m/z = 285$.

Butyl 2-(2,4-dinitrophenyl)hydrazinecarbodithioate
(**8**, $C_{11}H_{14}N_4O_4S_2$)
Yield 78%; mp yellow oil; IR (neat): $\bar{\nu} = 670$, 1212 cm^{-1}; ^1H NMR ($CDCl_3$): $\delta = 0.94$ (t, 3H, CH_3), 1.32 (m, 2H, CH_2CH_3), 1.95 (m, 2H, $SCH_2 \cdot CH_2$), 2.02 (br, H, N*H*), 2.83 (t, 2H, SCH_2), 4.04 (br, N, $NHArNO_2$), 7.19–9.50 (m, 3H, *Ar*–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 13.8$, 21.9, 32.3, 33.8, 113.6, 119.2, 130.2, 132.8, 139.7, 143.3, 222.5 ppm; MS: $m/z = 330$.

Butyl 2-(naphth-2-yl)hydrazinecarbodithioate
(**9**, $C_{15}H_{18}N_2S_2$)
Yield 82%; mp yellow oil; IR (neat): $\bar{\nu} = 677$, 1209 cm^{-1}; ^1H NMR ($CDCl_3$): $\delta = 0.95$ (t, 3H, CH_3), 1.33 (m, 2H, CH_2CH_3), 1.97 (m, 2H, $SCH_2 \cdot CH_2$), 2.05 (br, H, N*H*), 2.84 (t, 2H, SCH_2), 4.05 (br, N, $NHArNO_2$), 6.76–7.55 (m, 7H, *Ar*–H) ppm; ^{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, 224.1 ppm; MS: $m/z = 290$.

1-Butylpentyl 2-phenylhydrazinecarbodithioate
(**10**, $C_{16}H_{26}N_2S_2$)
Yield 88%; mp yellow oil; IR (neat): $\bar{\nu} = 677$, 1212 cm^{-1}; ^1H NMR ($CDCl_3$): $\delta = 0.96$ (t, 6H, CH_3), 1.29 (m, 4H, CH_2CH_2CH), 1.33 (m, 4H, CH_2CH_3), 1.92 (m, 4H, $CHCH_2$), 2.05 (br, H, N*H*), 2.52 (t, H, S*CH*), 4.05 (br, H, *NHAr*), 6.66–7.18 (m, 5H, *Ar*–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 14.2$, 23.1, 28.5, 36.2, 41.4, 112.2, 119.3, 129.0, 142.4, 223.3 ppm; MS: $m/z = 310$.

1,1-Dibutylpentyl 2-phenylhydrazinecarbodithioate
(**11**, $C_{20}H_{34}N_2S_2$)
Yield 86%; mp yellow oil; IR (neat): $\bar{\nu} = 669$, 1210 cm^{-1}; ^1H NMR ($CDCl_3$): $\delta = 0.96$ (t, 6H, CH_3), 1.29 (m, 4H, CH_2-CH_2C), 1.33 (m, 4H, CH_2CH_3), 1.88 (m, 4H, $CHCH_2$), 2.04 (br, H, N*H*), 4.0 (br, H, *NH–Ar*), 6.67–7.19 (m, 5H, *Ar*–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 14.1$, 23.4, 26.7, 39.6, 41.1, 112.5, 119.3, 129.6, 142.2, 223.5 ppm; MS: $m/z = 366$.

Hexyl 2-butylhydrazinecarbodithioate (**12**, $C_{11}H_{24}N_2S_2$)
Yield 95%; mp yellow oil; IR (neat): $\bar{\nu} = 674$, 1208 cm^{-1}; ^1H NMR ($CDCl_3$): $\delta = 0.96$ (t, 6H, CH_3), 1.29 (m, 4H, $CH_2CH_2CH_2CH_3$), 1.33 (t, 2H, CH_2CH_3), 1.55 (m, 2H, $NHCH_2CH_2$), 1.96 (m, 2H, SCH_2CH_2), 2.0 (br, 2H, NH), 2.65 (t, 2H, $NHCH_2$), 2.87 (t, 2H, SCH_2) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 13.7$, 14.1, 20.2, 23.1, 28.6, 31.5, 32.6, 49.9, 223.1 ppm; MS: $m/z = 248$.

n-Octyl 2-phenylhydrazinecarbodithioate (**13**, $C_{15}H_{24}N_2S_2$)
Yield 96%; mp yellow oil; IR (neat): $\bar{\nu} = 679$, 1211 cm^{-1}; ^1H NMR ($CDCl_3$): $\delta = 0.96$ (t, 3H, CH_3), 1.29 (m, 8H, CH_2), 1.33 (m, 2H, CH_2CH_3), 1.96 (m, 2H, SCH_2CH_2), 2.0 (br, H, NH), 2.88 (t, 2H, SCH_2), 4.0 (br, H, $Ph \cdot NH$), 6.65–7.20 (m, 5H, *Ar*–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 14.5$, 23.10, 28.9, 30.5, 31.5, 32.5, 112.2, 129.6, 118.9, 142.2, 223.6 ppm; MS: $m/z = 296$.

Decyl 2-butylhydrazinecarbodithioate (**14**, $C_{15}H_{32}N_2S_2$)
Yield 98%; mp yellow oil; IR (neat): $\bar{\nu} = 673$, 1220 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), 1.55 (m, 2H, $CH_2CH_2CH_3$), 1.96 (m, 2H, SCH_2CH_2), 2.0 (br, 2H, $NH \cdot NH$), 2.65 (m, 2H, $NHCH_2$), 2.87 (t, 2H, SCH_2) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 13.7$, 14.5, 20.3, 23.1, 28.9, 30.6, 30.9, 31.5, 32.5, 222.1 ppm; MS: $m/z = 304$.

1-Propylbutyl 2-phenylhydrazinecarbodithioate
(**15**, $C_{14}H_{22}N_2S_2$)
Yield 85%; mp yellow oil; IR (neat): $\bar{\nu} = 675$, 1210 cm^{-1}; ^1H NMR ($CDCl_3$): $\delta = 0.97$ (s, 3H, CH_3), 1.33 (m, 4H, CH_2CH_3), 1.92 (m, 4H, $CHCH_2$), 2.0 (br, H, NH), 2.52 (m, H, C*H*–S), 4.1 (br, H, NH–*Ar*), 6.66–7.22 (m, 5H, *Ar*–H) ppm; ^{13}C NMR

($CDCl_3$): $\delta = 14.5, 20.1, 38.4, 40.8, 112.5, 118.3, 129.6, 143.3, 222.1$ ppm; MS: $m/z = 282$.

1-Phenylethyl 2-phenylhydrazinecarbodithioate
(**16**, $C_{15}H_{16}N_2S_2$)
Yield 82%; mp yellow oil; IR (neat): $\bar{\nu} = 678, 1210\,cm^{-1}$; ^1H NMR ($CDCl_3$): $\delta = 1.69$ (d, 3H, CH_3), 2.2 (br, H, NH), 3.98 (m, H, *CH*–S), 4.2 (br, H, NH–*Ar*), 6.66–7.22 (m, 10H, *Ar*–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 23.4, 41.1, 112.5, 118.9, 126.5, 128.5, 129.7, 141.3, 142.5, 222.1$ ppm; MS: $m/z = 288$.

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