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An efficient, basic resin mediated, one-pot synthesis of dithiocarbamate esters through alcoholic tosylates

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An efficient, basic resin mediated, one-pot synthesis of dithiocarbamate esters through alcoholic tosylates

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A novel process for the one-step conversion of alcoholic tosylates into dithiocarbamates as protected amines was developed using basic resin (amberlyte IRA 400) in presence of carbon disulfide. Dithiocarbamates of different amines were isolated in very good to excellent yields. This protocol is mild, chemoselective and efficient compared to other existing methods.

Keywords: Basic resin; Carbon disulfide; Alcoholic tosylates; Dithiocarbamates; Thiocarbamation

1. Introduction

Organic dithiocarbamates have been frequently used as agrochemicals [1, 2], pharmaceuticals [3–5], intermediates in organic synthesis [6, 7], for the protection of amino groups in peptide synthesis [8], as linkers in solid phase organic synthesis [9], radical precursors in free radical chemistry [10, 11] and recently in the synthesis of ionic liquids [12]. To satisfy this demand, their synthesis have been changed from the use of harmful and toxic chemicals like dithiophosgene [13] and its derivatives [14] directly or indirectly, to the abundantly available, cheap and safe reagents like CS_2. However, their formation using CS_2 sometimes employs harsh reaction conditions such as use of strong base, high reaction temperatures and long reaction times [15–17]. Thus, we were prompted to embark on the improved procedures. Our group [18–20] has been engaged for several years in the development of new methodologies for the preparation of carbamates and dithiocarbamates using cheap, abundantly available and safe reagents. Recently, we have reported [21] a high yielding, one-pot, novel synthesis of dithiocarbamates from corresponding alkyl halides using the Triton-B/CS_2 system. We have also reported [22] the use of a basic resin in the tetrahydropyranylation of alcohols and phenols. Furthermore, use of basic resin has also been reported for synthesis of carbamates through alcoholic tosylates using gaseous CO_2 [23]. In the present communication, we report herein

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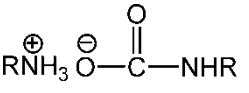
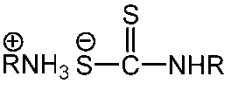
a chemoselective, highly efficient, one-pot, novel synthesis of dithiocarbamates using a basic resin/CS_2 system starting from alcoholic tosylates.

In our recent carbamate report [23], where 2 molar equivalents of amine were reacted with carbon dioxide, we suggested the formation of monoalkylammoniumalkyl carbamate ion (**A**). It has been observed that the nucleophilicity of **A** could be enhanced by using a basic catalyst. The $\mathbf{O}^-$ of **A** could then attack towards alkylating agents to afford carbamates in high yields. By adopting a similar approach monoalkylammoniumalkyl dithiocarbamate ion (**B**) should be obtained using CS_2. The nucleophilicity of **B** should be enhanced by a using basic resin *i.e.* Amberlyte IRA 400.

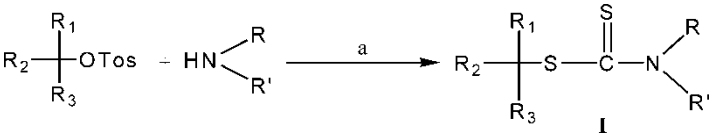


(A)



(B)

The nucleophilic $\mathbf{S}^-$ ion of **B** would then attack the electrophilic carbon of the alcoholic tosylates, leading to the formation of dithiocarbamates as shown in scheme 1. Moreover, due to the higher reactivity of carbon disulfide vs. carbon dioxide, the reaction at hand was tried at room temperature. The reaction proved successful and the products were isolated and characterized by various spectroscopic and analytical techniques.



I

SCHEME 1 Reagants and conditions: (a) Amberlyte IRA 400, CS2, dry DMSO, rt, 2–4 h.

The alcoholic tosylates of different alcohols (primary, secondary, tertiary) were prepared by reacting alcohols with p-toluenesulfonyl chloride by following the standard procedure [24]. Thus different alcoholic tosylates were reacted with different primary/secondary (aliphatic, aromatic, cyclic) amines using the basic resin/CS_2 system in dry dimethylsulfoxide (DMSO) at room temperature for 2–4 h to afford dithiocarbamates (70–98%) as shown in table 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 at room temperature.

In conclusion, we have developed a convenient and efficient protocol for one-pot, three components coupling of various amines with variety of alcoholic tosylates via CS_2 bridge using a basic resin (Amberlyte IRA 400). This method generates the corresponding dithiocarbamates in good to excellent yields. Furthermore, this method exhibits substrate versatility, mild reaction conditions and experimental convenience. This synthetic protocol developed in our laboratory is believed to offer a more general method for the formation of carbon-sulfur bonds, essential to numerous organic syntheses.

2. Experimental

Chemicals were procured from Merck, Aldrich and Fluka chemical companies. Amberlyte IRA 400 (basic resin) was also obtained from Merck. IR spectra (4000–200 cm^{-1}) were recorded on

a Bomem MB-104 FTIR spectrophotometer where as ^{1}H NMRs were scanned on a AC-300F NMR (300 MHz) instrument using $CDCl_3$ as solvent and TMS as internal standard. Elemental analyses were made by Carlo-Erba EA1110 CNNO-S analyzer and agreed favorably with calculated values.

2.1 *Typical experimental procedure*

A mixture of Amberlyte IRA 400 (6 mmol) and carbon disulfide (6 mmol) was taken in 40 mL dry DMSO and was allowed to stirred for 20 min. at room temperature. Amine (5 mmol) was added and stirred continuously at room temperature for 1 h. Now the corresponding alcoholic tosylates (2 mmol) was added. The reaction was further continued till the completion of reaction (cf. table 1). The reaction mixture was filtered and filtrate was poured into 50 mL distilled water and extracted with ethyl acetate (60 mL) thrice. Organic layer was washed with 0.1 N HCl (50 mL), saturated solution of $NaHCO_3$ (50 mL), brine (60 mL), dried (Na_2SO_4) and then concentrated to get the desired compound.

2.2 *S-2-Phenylethyl N-n-butyl dithiocarbamate (1)*

IR (Neat): $\ddot{v} = 659$ (C−S), 1086 (C=S), 1467 (Ar), 2884 (CH), 2927 (CH), 3398 (NH) cm^{-1}; ^{1}H NMR ($CDCl_3$): $\delta = 0.89$–0.93 (t, 3H, CH_3), 1.28–1.34 (m, 2H, CH_2CH_3), 1.54–1.57 (m, 2H, $CH_2CH_2CH_3$), 2.2 (br, H, N*H*), 2.63–2.65 (m, 2H, CH_2NH), 3.18–3.20 (t, 2H, PhCH$_2$ CH_2), 3.23–3.25 (t, 2H, PhCH_2), 7.08–7.21 (m, 5H, Ar-H of phenyl ring) ppm; Ms: m/z = 253.

2.3 *S-3-Phenylpropyl N-n-hexyl dithiocarbamate (2)*

IR (Neat): $\ddot{v} = 669$ (C−S), 1116 (C=S), 1512 (Ar), 2864 (CH), 2937 (CH), 3408 (NH) cm^{-1}; ^{1}H NMR ($CDCl_3$): $\delta = 0.88$–0.92 (t, 3H, CH_3), 1.28–1.30 (m, 4H, CH_2CH_2 of n-hexyl group), 1.32–1.35 (m, 2H, CH_2CH_3), 1.54–1.57 (m, 2H, NHCH$_2CH_2$), 2.2 (br, H, N*H*) 2.27–2.29

Table 1. Conversion of the alcoholic tosylates into dithiocarbamates of general formula **I**.

Entry	R_1	R_2	R_3	R	R′	Time (h)	Yields (%)
1	Benzyl	H	H	*n*-Butyl	H	3	93
2	2-Phenethyl	H	H	*n*-Hexyl	H	2.5	96
3	2-Phenethyl	H	H	*i*-Propyl	*i*-Propyl	3.5	84
4	*n*-Propyl	H	H	*n*-Octyl	H	2.5	94
5	*n*-Butyl	H	H	Cyclohexyl	H	3.5	85
6	2-Naphthyloxyethyl	H	H	R&R,= Morpholinyl		3.5	86
7	2-Naphthyloxyethyl	H	H	R&R,= Pyrrolidinyl		3.5	83
8	*n*-Butyl	*n*-Butyl	H	*n*-Octyl	H	2.5	82
9	*n*-Butyl	*n*-Butyl	*n*-Butyl	*n*-Dodecyl	H	3	80
10	*n*-Hexyl	H	H	Phenyl	H	4	70
11	*n*-Heptyl	H	H	Benzyl	H	3	82
12	*n*-Heptyl	H	H	*n*-Dodecyl	H	2	98
13	2-Naphthyloxyethyl	H	H	*n*-C_4H_9	H	3	86
14	2-Naphthyloxypropyl	H	H	*n*-Octyl	H	2.5	95
15	$R_1 = R_2 =$ *c*-hexyl		H	*n*-Butyl	H	3	81
16	$R_1 = R_2 =$ *c*-hexyl		CH_3	*n*-Hexyl	H	3	83
17	CH_3	CH_3	CH_3	*n*-Dodecyl	H	4	75
18	CH_3	CH_3	H	*n*-Octyl	H	3.5	76

Note: All the products were characterized by IR, NMR and Mass spectroscpic and analytical data

(m, 2H, $PhCH_2CH_2CH_2$), 2.54–2.56 (t, 2H, $PhCH_2$), 2.63–2.66 (t, 2H, CH_2NH), 2.84–2.86 (m, 2H, S−CS−NH CH_2), 7.08–7.21 (m, 5H, Ar−H of phenyl ring) ppm; Ms: m/z = 295.

2.4 *S-3-Phenylpropyl N,N-di-isopropyl dithiocarbamate (3)*

IR (Neat): $\ddot{v}$ = 658 (C−S), 1096 (C=S), 1502 (Ar), 2854 (CH), 2927 (CH) cm^{-1}; ^1H NMR ($CDCl_3$): δ = 0.90–0.94 (t, 3H, CH_3), 1.43–1.45 (m, 2H, CH_2CH_3 of di isopropyl group), 2.53–2.55 (t, 2H, NCH_2), 3.17–3.20 (t, 2H, $PhCH_2CH_2$), 3.24–3.26 (t, 2H, $PhCH_2$), 7.08–7.21 (m, 5H, Ar-H of phenyl ring) ppm; Ms: m/z = 281.

2.5 *S-n-Butyl N-n-octyl dithiocarbamate (4)*

IR (Neat): $\ddot{v}$ = 648 (C−S), 1086 (C=S), 2864 (CH), 2917 (CH), 3388 (NH) cm^{-1}; ^1H NMR ($CDCl_3$): δ = 0.89–0.92 (t, 3H, CH_3), 1.26–1.30 (m, 8H, CH_2 of n-octyl group), 1.32–1.34 (m, 2H, CH_2CH_3), 1.54–1.57 (m, 2H, NH−CH_2CH_2), 1.93–1.96 (m, 2H, S−CH_2CH_2), 2.0 (br, NH), 2.63–2.65 (t, 2H, CH_2NH), 2.85–2.87 (t, 2H, CH_2S) ppm; Ms: m/z = 261.

2.6 *S-n-Pentyl N-cyclohexyl dithiocarbamate (5)*

IR (Neat): $\ddot{v}$ = 650 (C−S), 1088 (C=S), 2862 (CH), 2919 (CH), 3389 (NH) cm^{-1}; ^1H NMR ($CDCl_3$): δ = 0.89–0.92 (t, CH_3), 1.28–1.30 (m, CH_2 of n-pentyl group), 1.32–1.34 (m, CH_2CH_3), 1.43–1.46 (m, CH_2 of cyclohexyl ring), 1.64–1.67 (m, CH_2 of cylohexyl ring), 1.93–1.96 (m, S−CH_2CH_2 of cyclopentyl group), 2.1 (br, NH), 2.55–2.58 (m, CH of cyclohexyl ring), 2.85–2.87 (t, CH_2S) ppm. ^{13}C NMR δ = 199.3, 49.3, 32.8, 32.5, 31.5, 31.1, 27.2, 22.8, 22, 14.5 ppm. Ms: m/z = 245.

2.7 *S-3-(2-Naphthyloxy) propyl morpholinodithiocarbamate (6)*

IR(KBr): $\ddot{v}$ = 671 (C−S), 1129 (C=S), 1477 (Ar), 1528 (Ar), 1610 (Ar), 2884 (CH), 2937 (CH) cm^{-1}; ^1H NMR ($CDCl_3$): δ = 2.34–2.38 (m, naphthyl−O−$CH_2CH_2CH_2$−), 2.83–2.87 (t, S−CH_2 of naphthyl), 2.89–2.93 (m, NCH_2 of morpholine ring), 3.65–3.69 (t, −O−CH_2−of morpholine ring), 4.05–4.09 (t, CH_2−O−naphthyl), 6.97–7.64 (m, Ar−H of naphthyloxy ring) ppm; Ms: m/z = 347.

2.8 *S-3-(2-Naphthyloxy) propyl pyrrodinodithiocarbamate (7)*

IR (KBr): $\ddot{v}$ = 673 (C−S), 1126 (C=S), 1474 (Ar), 1522 (Ar), 1606 (Ar), 2884 (CH), 2937 (CH) cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.58–1.60 (m, CH_2 of pyrolidine ring), 2.35–2.38 (m, naphthyl−O−$CH_2CH_2CH_2$−S−C=S), 2.8 (t, CH_2N of pyrrolidine ring), 2.83–2.87 (t, CH_2−S−C=S), 4.01–4.04 (t, CH_2−O−naphthyl), 6.97–7.64 (m, Ar−H of naphthyloxy)ppm; Ms: m/z = 331.

2.9 *S-5-nonyl N-n-octyl dithiocarbamate (8)*

IR (KBr): $\ddot{v}$ = 648 (C−S), 1086 (C=S), 2864 (CH), 2917 (CH), 3388 (NH) cm^{-1}; ^1H NMR ($CDCl_3$): δ = 0.90–0.93 (t, CH_3 of n-butyl and n-octyl group), 1.27–1.30 (m, CH_2 of n-butyl and n-octyl group), 1.32–1.34 (m, CH_2CH_3 of n-butyl and n-octyl group), 1.55–1.57 (m, $NHCH_2CH_2$ of n-octyl group), 1.90–1.92 (m, CH · CH_2), 2.2 (br, NH), 2.63–2.65 (t, CH_2NH)

ppm.^{13}C NMR $\delta = 200.22$, 47.33, 41.75, 35.93, 30.55, 31.44, 30.04, 28.94, 27.40, 23.45, 14.45 ppm. Ms: m/z = 331.

2.10 *S-(5-n-butyl)-5-nonyl N-n-dodecyl dithiocarbamate (9)*

IR (KBr): $\ddot{v} = 648$ (C−S), 1086 (C=S), 2864 (CH), 2917 (CH), 3388 (NH) cm^{-1}; ^{1}H NMR ($CDCl_3$): $\delta = 0.89$–0.92 (t, CH_3 of n-butyl and n-dodecyl group), 1.26–1.30 (m, CH_2 of n-dodecyl group), 1.32–1.34 (m, CH_2CH_3 of n-butyl group), 1.55–1.57 (m, $NHCH_2CH_2$ of n-dodecyl group), 1.85–1.88 (m, $CH \cdot CH_2$), 2.0 (br, NH), 2.63–2.65 (t, CH_2NH) ppm; ^{13}C NMR $\delta = 200.24$, 47.35, 41.70, 39.93, 32.55, 31.45, 30.04, 27.42, 23.45,14.45 ppm. Ms: m/z = 443.

2.11 *S-n-Heptyl N-phenyl dithiocarbamate (10)*

IR (KBr): $\ddot{v} = 649$ (C−S), 1083 (C=S), 2860 (CH), 2915 (CH), 3390 (NH) cm^{-1}; ^{1}H NMR ($CDCl_3$): $\delta = 0.90$–0.92 (t, CH_3), 1.28–1.31 (m, CH_2 of n-heptyl group), 1.32–1.34 (m, CH_2CH_3), 2.86–2.88 (t, CH_2S), 4.0 (br, NH), 6.46–7.05 (m, aromatic protons) ppm; Ms: m/z = 267.

2.12 *S-n-Octyl N-benzyl dithiocarbamate (11)*

IR (KBr): $\ddot{v} = 653$ (C−S), 1087 (C=S), 2863 (CH), 2918 (CH), 3395 (NH) cm^{-1}; ^{1}H NMR ($CDCl_3$): $\delta = 0.88$–0.91 (t, CH_3), 1.29–1.32 (m, CH_2 of n-Octyl group), 1.33–1.36 (m, CH_2CH_3), 2.2 (br, NH), 2.85–2.87 (t, CH_2S), 3.91–3.93 (m, benzylic CH_2), 7.05–7.17 (m, aromatic protons) ppm; Ms: m/z = 295.

2.13 *S-n-Octyl N-dodecyl dithiocarbamate (12)*

IR (KBr): $\ddot{v} = 646$ (C−S), 1085 (C=S), 2866 (CH), 2920 (CH), 3393 (NH) cm^{-1}; ^{1}H NMR ($CDCl_3$): $\delta = 0.87$–0.90 (t, CH_3 of n-octyl and dodecyl group), 1.29–1.32 (m, CH_2 of n-Octyl and dodecyl group), 1.33–1.36 (m, CH_2CH_3 of octyl and dodecyl group), 1.55–1.57 (m, $NHCH_2CH_2$ of n-dodecyl group), 2.1 (br, NH), 2.65–2.67 (m, $NHCH_2$ of dodecyl group), 2.86–2.88 (t, CH_2S), ppm; Ms: m/z = 295.

2.14 *S-3-(2-Naphthyloxy) propyl N-n-butyl dithiocarbamate (13)*

IR (KBr): $\ddot{v} = 670$ (C−S), 1114 (C=S), 1474 (Ar), 1510 (Ar), 1609 (Ar), 2874 (CH), 2937 (CH), 3418 (NH) cm^{-1}; ^{1}H NMR ($CDCl_3$): $\delta = 0.93$–0.97 (t, CH_3), 1.30–1.34 (m, CH_2CH_3), 1.53–1.56 (m, $CH_2 \cdot CH_2 . CH_3$), 1.70–1.72 (m, naphthyl−O−CH_2CH_2), 1.95–1.98 (m, S−CH_2CH_2), 2.0 (br, NH), 2.63–2.66 (m, $NHCH_2$), 2.84–2.88 (t, CH_2−S−C=S), 4.01–4.04 (t, CH_2−O−naphthyl), 6.97–7.64 (m, Ar−H of naphthyloxy) ppm; Ms: m/z = 347.

2.15 *S-4-(2-Naphthyloxy) butyl N-n-octyl dithiocarbamate (14)*

IR (KBr): $\ddot{v} = 662$ (C−S), 1109 (C=S), 1464 (Ar), 1512 (Ar), 1604 (Ar), 2864 (CH), 2927 (CH), 3391 (NH) cm^{-1}; ^{1}HNMR ($CDCl_3$): $\delta = 0.93$–0.96 (t, CH_3), 1.27–1.29 (m, $CH_2CH_2CH_2CH_2$ CH_2CH_3 of octyl group), 1.30–1.34 (m, CH_2CH_3 of octyl group), 1.53–1.56 (m, $CH_2 \cdot CH_2 \cdot N$), 2.1 (br, NH), 2.64–2.66 (m, $NHCH_2$), 3.27–3.30

(t, CH_2−S−C=S), 4.70–4.72 (t, CH_2−O−naphthyl), 6.97–7.64 (m, Ar−H of naphthyloxy) ppm; Ms: m/z = 375.

2.16 *S-Cyclohexyl N-n-butyl dithiocarbamate (15)*

IR (Neat): $\ddot{v} = 648$ (C−S), 1086 (C=S), 2864 (CH), 2917 (CH), 3388 (NH) cm^{-1}; ^1H NMR ($CDCl_3$): $\delta = 0.89$–0.92 (t, 3H, CH_3), 1.32–1.34 (m, 2H, CH_2CH_3), 1.43–1.46 (m, 6H, CH_2 of cyclohexyl ring), 1.55–1.58 (m, 2H, CH_3CH_2 CH_2), 2.05–2.10 (t, 4H, CH · CH_2 of cyclohexyl), 2.2 (br, NH), 2.45–2.48 (m, CH of cyclohexyl ring), 2.63–2.66 (t, 2H, CH_2S) ppm; Ms: m/z = 231.

2.17 *S-Cyclohexyl N-n-hexyl dithiocarbamate (16)*

IR (Neat): $\ddot{v} = 652$ (C−S), 1093 (C=S), 2870 (CH), 2927 (CH), 3396 (NH) cm^{-1}; ^1H NMR ($CDCl_3$): $\delta = 0.90$–0.93 (t, 3H, CH_3), 1.32–1.34 (m, 4H, $CH_2CH_2CH_3$), 1.44–1.47 (m, 6H, CH_2 of cyclohexyl ring), 1.50 (s, 3H, CH_3 of cyclohexyl part) 1.55–1.58 (m, 2H, CH_3CH_2 CH_2), 2.03–2.07 (t, 4H, CH · CH_2 of cyclohexyl), 2.3 (br, NH), 2.64–2.67 (t, 2H, CH_2S) ppm; Ms: m/z = 273.

2.18 *S-t-butyl N-n-dodecyl dithiocarbamate (17)*

IR (Neat): $\ddot{v} = 648$ (C−S), 1086 (C=S), 2864 (CH), 2917 (CH), 3388 (NH) cm^{-1}; ^1H NMR ($CDCl_3$): $\delta = 0.93$–0.96 (t, 3H, CH_3 of n-dodecyl group), 1.26–1.30 (m, 16H, CH_2 of n-dodecyl group), 1.32–1.34 (m, 2H, CH_2CH_3 of n-dodecyl group), 1.41 (s, 9H, CH_3 of tert. butyl group), 1.53–1.56 (m, 2H, CH2 of dodecyl), 2.0 (br, NH), 2.63–2.65 (t, 2H, CH_2NH) ppm; Ms: m/z = 317.

2.19 *S-2-propyl N-n-octyl dithiocarbamate (18)*

IR (Neat): $\ddot{v} = 649$ (C−S), 1089 (C=S), 2867 (CH), 2921(CH), 3392 (NH) cm^{-1}; ^1H NMR ($CDCl_3$): $\delta = 0.92$–0.95 (t, 3H, CH_3 of n-octyl group), 1.26–1.30 (m, 8H, CH_2 of n-octyl group), 1.32–1.34 (m, 2H, CH_2CH_3 of n-octyl group), 1.34–1.38 (d, 6H, CH_3 of isopropyl group), 1.53–1.56 (m, 2H, CH_2 of octyl), 2.1 (br, NH), 2.63–2.65 (t, 2H, CH_2NH), 2.87–2.90 (m, H, CH of isopropyl group) ppm; Ms: m/z = 247.

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