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Devdutt Chaturvedi[a]; Nisha Mishra[b]; Virendra Mishra[b]
[a] Medicinal and Process Chemistry Division, Central Drug Research Institute, U.P., India [b] Synthetic Research Lab, Department of Chemistry, B. S. A. College, U.P., India

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An efficient, basic resin mediated, one-pot, synthesis of dithiocarbamates by Michael addition of dithiocarbamate anion to α, β-unsaturated compounds

DEVDUTT CHATURVEDI*†, NISHA MISHRA‡ and VIRENDRA MISHRA‡

†Medicinal and Process Chemistry Division, Central Drug Research Institute, Lucknow-226001, U.P., India
‡Synthetic Research Lab, Department of Chemistry, B. S. A. College, Mathura-281004, U.P., India

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A highly efficient, one-pot, synthesis of dithiocarbamates was accomplished in high yields by the Michael addition of dithiocarbamate anion to α, β-unsaturated compounds using Amberlite IRA 400 (basic resin). The reaction conditions are mild with extremely simple work-up procedures than the reported methods.

Keywords: Michael addition; Amberlite IRA 400; Carbon disulfide; Amines; α, β-Unsaturated olefins

1. Introduction

Organic dithiocarbamates have widely been used as agrochemicals [1, 2], pharmaceuticals [3–5], intermediates in organic synthesis [6, 7], protection of amino groups in peptide synthesis [8], linkers in solid phase organic synthesis [9], radical precursors in free radical chemistry [10–14] and in the synthesis of ionic liquids [15]. Also, different transition metal complexes of dithiocarbamates have been synthesized for various studies primarily because of their applications as organic superconductors [16–19]. To satisfy their demand, their synthesis has been adapted from the use of costly and toxic chemicals like thiophosgene [20] and its derivatives [21] directly or indirectly, to the abundantly available cheap and safe reagents like CS_2. Moreover, their formation using CS_2 employed harsh reaction conditions such as use of strong bases, higher reaction temperatures and longer reaction times [22–25]. Synthesis of β-substituted ethyl dithiocarbamates were reported by Guo [26] and Azizi *et al.* [25] through Michael addition reaction but both of the methods require product purification through chromatographic techniques. Thus, we were prompted to embark on improved procedures. Our group [27–37] has been engaged from past several years for the development of new methodologies for the preparation of carbamates, dithiocarbamates and related compounds

*Corresponding author. Email: ddchaturvedi002@yahoo.co.in

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using cheap, abundantly available, and safe reagents like CO_2 and CS_2 respectively. More recently [38–41], we found that Amberlite IRA 400 (basic resin) is the best reagent for the synthesis of carbamates, dithiocarbamates and dithiocarbonates (Xanthates). Furthermore, use of basic resin has also been reported [42] for the tetrahydropyranylation of alcohols and phenols. In the present communication, we report here an efficient, one-pot, synthesis of dithiocarbamates by Michael addition of dithiocarbamate ion to activated olefins using basic resin/CS_2 system.

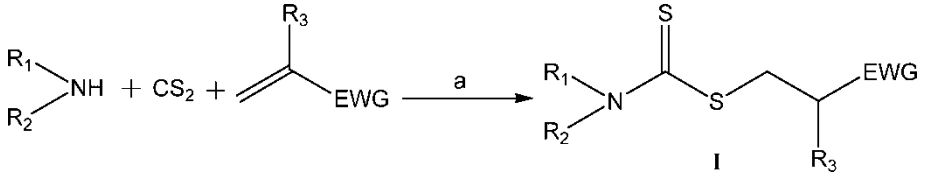
2. Results and discussion

In connection with our ongoing interest pertaining to the use of Amberlite IRA 400 (basic resin) for the synthesis of carbamates, dithiocarbamates and dithiocarbonates (Xanthates) [33–36], we now wish to report a simple and effective one-pot procedure for a Michael addition of dithiocarbamate anion to α, β-unsaturated activated olefins mediated by basic resin. Thus, a mixture of amine and CS_2 were dissolved in dry DMSO and Amberlite IRA 400 (basic resin) was added in it. It is proposed that the dithiocarbamate ion produced will add via Michael type addition to corresponding activated alkene to afford dithiocarbamates in high yields (75–98%) at room temperature in 2–4 h, as mentioned in table 1. The reaction proved to be successful and upon isolation the structures of the desired products were confirmed by various spectroscopic and analytical techniques. That the products were simply obtained by concentration of organic layer after filtration of basic resin from the reaction mixture indicates the novelty of the method among the reported procedures. Reactions have also been tried without using Amberlite resin, but no products could be observed, indicating the necessity of basic resin in carrying out the reaction (scheme 1).

Table 1 shows that primary and secondary amines can react with carbon disulfide and different kind of α, β-unsaturated activated olefins to give the corresponding β-EWG ethyl dithiocarbamates. This reaction only works well with electron withdrawing groups (EWG), such as −CN, $-CONH_2$, C(O)OMe and $COCH_3$. The reactions were carried out in one-pot and complete within 2–4 h. However, when hindered α, β-carbonyl compounds with methyl or methylene groups at the β-position were used, low yields of the products were obtained. We tried several solvents like *n*-heptane, *n*-hexane, acetonitrile, benzene, toluene, methanol,

Table 1. Conversion of activated olefins into dithiocarbamates of general formula **I**.

Entry	R_1	R_2	R_3	EWG	Time (h)	Isolated yield (%)
1	C_2H_5	C_2H_5	H	$COCH_3$	2	92
2	C_2H_5	C_2H_5	H	$COOCH_3$	2	95
3	C_2H_5	C_2H_5	H	CN	2.5	85
4	C_2H_5	C_2H_5	H	$CONH_2$	3	90
5	C_2H_5	C_2H_5	CH_3	$COOCH_3$	2.5	79
6	CH_2Ph	H	H	$COOCH_3$	3	86
7	CH_2Ph	H	H	CN	3	84
8	*t*-C_4H_9	H	H	COOMe	2.5	90
9	R_1=R_2=Pyrrolidine		H	$COCH_3$	2.5	87
10	R_1=R_2=Piperidine		H	$COCH_3$	2.5	83
11	R_1=R_2=Morpholinyl		H	$COOCH_3$	4	81
12	R_1=R_2=Piperidine		H	$CONH_2$	3	88
13	R_1=R_2=Pyrrolidine		CH_3	COOMe	3	98
14.	Ph.CH(CH_3)	H	H	$COCH_3$	2.5	78
15	Ph.CH(CH_3)	H	H	COOMe	2.5	81
16	Ph.CH(CH_3)	H	H	CN	3	75



SCHEME 1 Reagents and Conditions: (a) Amberlite IRA 400, Dry DMSO, rt, 2-4 h.

dichloromethane, chloroform, DMSO, dimethylformamide, hexamethylphosphoric triamide of which dry DMSO proved to be 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 α, β-unsaturated olefins via CS_2 bridge using 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.

Note: All the products were characterized by IR, NMR and Mass spectroscopic data.

3. Experimental

Chemicals were purchased from Merck, Aldrich and Fluka chemical companies. Amberlite IRA 400 (basic resin) was also purchased from Merck. Reactions were carried out under an atmosphere of nitrogen. IR spectra (4000–200 cm^{-1}) were recorded on a Bomem MB-104–FTIR spectrophotometer. NMRs were obtained on a AC-300F, NMR (300MHz), instrument using $CDCl_3$ and some other deuterated solvents with TMS as internal standard. Elemental analysis were made by Carlo-Erba EA 1110-CNNO-S analyzer and agreed favorably with calculated values.

3.1 *Typical experimental procedure*

To a stirred solution of amine (3 mmol) in anhyd. DMSO (5 mL) was slowly added, carbon disulfide (8 mmol) and basic resin (5 mmol) at room temperature. Then the mixture was stirred for 0.5 h at which point α, β-unsaturated olefin (3 mmol) was added over a period of 5 min. The reaction mixture was further continued until the completion of reaction (cf table 1) under argon. The reaction mixture was filtered to remove the resin. The filtrate was poured into water (20 mL) and organic layer was extracted with EtOAc (3 × 10 mL). The organic layer was washed with 0.1 N HCl (20 mL), saturated solution of sodium bicarbonate (25 mL), brine (30 mL) and dried (Na_2SO_4) and concentrated to get the desired compound.

3.2 *Data for dithiocarbamates*

3.2.1 Compound (1). IR $\ddot{\nu}$ (cm^{-1}) = 1712 (C=O), 1220 (C=S); ^{1}H NMR ($CDCl_3$) δ = 1.01–1.06 (6H, m), 1.94 (3H, s), 2.72 (2H, t, J = 6.6 Hz), 3.23 (2H, t, J = 6.6 Hz, CH_2N), 3.52 (2H, q, J = 7.1 Hz SCH_2), 3.73 (2H, q, J = 7.1 Hz); ^{13}CNMR ($CDCl_3$), δ = 11.8, 12.7, 30.1, 30.6, 43.6, 46.9, 49.6, 195.2, 206.9 ppm; MS: m/z = 219; Analysis: $C_9H_{17}NOS_2$, Calcd: C, 49.28; H, 7.81; N, 6.39; S, 29.23; Obsd: C, 49.72; H, 7.55; N, 6.12; S, 29.12.

3.2.2 Compound (2). IR $\ddot{\nu}$ (cm^{-1}) = 1730 (C=O), 1205 (C=S); ^{1}H NMR ($CDCl_3$) δ = 0.93–0.99 (6H, m), 2.47 (2H, t, J = 6.4 Hz), 3.20 (2H, t, J = 6.5 Hz, CH_2N), 3.44 (3H, s), 3.45 (2H, q, J = 6.9 Hz), 3.70 (2H, q, J = 6.9 Hz); ^{13}CNMR ($CDCl_3$), δ = 11.7, 12.9, 31.7, 33.9, 46.9, 49.6, 51.6, 171.6, 194.4 ppm; MS: m/z = 235; Analysis: $C_9H_{17}NO_2S_2$, Calcd: C, 45.93; H, 7.28; N, 5.95; S, 27.25; Obsd: C, 45.49; H, 7.45; N, 6.17; S, 27.11.

3.2.3 Compound (3). IR $\ddot{\nu}$ (cm^{-1}) = 2248 (CN), 1203 (C=S), ^{1}H NMR ($CDCl_3$) δ = 0.92–0.96 (6H, m), 2.53 (2H, t, J = 6.7 Hz), 3.12 (2H, t, J = 6.2, Hz), 3.42 (2H, q, J = 6.7 Hz), 3.67 (2H, t, J = 6.7 Hz); ^{13}CNMR ($CDCl_3$), δ = 9.4, 11.7, 18.2, 32.0, 47.2, 49.9, 118.6, 192.7 ppm; MS: m/z = 202; Analysis: $C_8H_{14}N_2S_2$, Calcd: C, 47.49; H, 6.97; N, 13.84; S, 31.69; Obsd: C, 47.73; H, 6.64; N, 13.55; S, 32.10.

3.2.4 Compound (4). IR $\ddot{\nu}$ (cm^{-1}) = 3380, 1680, 1201; ^{1}H NMR (DMSO/$CDCl_3$) δ = 1.04 (3H, t, J = 7.2 Hz), 1.14 (3H, t, J = 7.1 Hz), 2.46 (2H, J = 7.2 Hz), 3.38 (2H, t, J = 7.0 Hz, SCH_2), 3.60 (2H, q, J = 7.2 Hz, CH_2N), 3.81 (2H, q, J = 7.2 Hz, CH_2N), 4.98–5.41 (2H, br, NH_2); ^{13}CNMR (DMSO/$CDCl_3$), δ = 12.4, 34.6, 35.8, 45.3, 174.8, 197.5 ppm; MS: m/z = 220; Analysis: $C_8H_{16}N_2OS_2$, Calcd: C, 43.60; H, 7.32; N, 12.71; S, 29.10; Obsd: C, 43.89; H, 7.07; N, 12.45; S, 29.42.

3.2.5 Compound (5). IR $\ddot{\nu}$ (cm^{-1}) = 1735, 1210; ^{1}H NMR (C_6D_6) δ = 1.10 (6H, br), 1.28 (3H, d, J = 7.2 Hz), 3.00 (1H, m), 3.34 (1H, d, J = 7.2 Hz, SCH_2), 3.46 (1H, d, J = 7.2 Hz, SCH_2), 3.53 (2H, q, J = 7.2 Hz, CH_2CN), 3.63 (3H, s), 3.78 (2H, q, J = 7.2 Hz, CH_2N), ^{13}C NMR (C_6D_6) δ = 12.7, 15.8, 35.7, 40.4, 45.8, 52.6, 173.8, 196.7 ppm; MS: m/z = 249; Analysis: $C_{10}H_{19}NO_2S_2$, Calcd: 48.16; H, 7.68; N, 5.62; S, 25.71; Obsd: C, 48.62; H, 7.45; N, 5.49; S, 25.48.

3.2.6 Compound (6). IR $\ddot{\nu}$ (cm^{-1}) = 1736, 1215; ^{1}H NMR (CD_3NO_2) δ = 2.82 (2H, t, J = 7.1 Hz), 3.53 (2H, t, J = 7.1 Hz), 3.73 (3H, s), 4.97 (2H, d, J = 4.8 Hz, CH_2NH), 7.40 (5H, m), 7.14 and 8.04 (each 1H, br, together NH and SH) ppm; ^{13}C NMR (CD_3NO_2) δ = 31.2, 34.8, 48.5, 52.1, 126.7, 126.9, 127.2, 142.3, 173.5, 199.8 ppm; MS: m/z = 269; Analysis: $C_{12}H_{15}NO_2S_2$, Calcd: C, 53.50; H, 5.61; N, 5.20; S, 23.81; Obsd: C, 53.77; H, 5.33; N, 5.57; 23.43.

3.2.7 Compound (7). IR $\ddot{\nu}$ (cm^{-1}) = 2251, 1203, 747; ^{1}H NMR (acetone-d_6) δ = 2.89 (2H, t, J = 7.1 Hz, CH_2CN), 3.55 (2H, t, J = 7.1 Hz, SCH_2), 4.95 (2H, d, J = 5 Hz, CH_2NH), 7.34 (5H, m), 7.14 and 9.48 (each 1H, br, together NH and SH); ^{13}C NMR (acetone-d_6) δ = 19.2, 48.6, 55.9, 118.3, 126.7, 126.9, 127.2, 142.3, 199.8 ppm; MS: m/z = 236; Analysis: $C_{11}H_{12}N_2S_2$, Calcd: C, 55.90; H, 5.12; N, 11.85; S, 27.13; Obsd: C, 55.66; H, 5.37; N, 11.47; S, 27.55.

3.2.8 Compound (8). IR $\ddot{\nu}$ (cm^{-1}) = 1735 (C=O), 1207 (C=S); ^{1}H NMR ($CDCl_3$) δ = 1.43 (9H, s), 2.60(2H, t, J = 6.1 Hz), 3.28 (2H, t, J = 6.2 Hz), 3.56 (3H, s); ^{13}CNMR ($CDCl_3$) δ = 27.9, 28.4, 34.9, 52.0, 57.6, 172.6, 149.6 ppm; MS: m/z = 235; Analysis: $C_9H_{17}NO_2S_2$, Calcd: C, 45.93; H, 7.28; N, 5.95; S, 27.25; Obsd: C, 45.57; H, 7.55; N, 6.23, S, 27.08.

3.2.9 Compound (9). IR $\ddot{\nu}$ (cm^{-1}) = 1710 (C=O), 1222 (C=S); ^{1}H NMR (CD_3NO_2) δ = 2.06 (2H, t, J = 7.2 Hz), 2.18 (2H, t, J = 7.2 Hz), 2.21 (3H, s), 2.94 (2H, t, J = 7.1 Hz), 3.43 (2H, t, J = 7.1 Hz), 3.69 (2H, t, J = 7.2 Hz), 3.69 (2H, t, J = 7.2 Hz), 3.89 (2H, t, J = 7.2 Hz); ^{13}CNMR (CD_3NO_2) δ = 23.8, 25.9, 29.9, 43.1, 50.7, 55.1, 191.7, 202.3 ppm; MS: m/z = 217; Analysis: $C_9H_{15}NOS_2$, Calcd: C, 49.73; H, 6.96; N, 6.44; S, 29.51; Obsd: C, 49.53; H, 6.82; N, 6.61; S, 29.69.

3.2.10 Compound (10). IR $\ddot{\nu}$ (cm^{-1}) = 1708 (C=O), 1212 (C=S); ^{1}H NMR (CD_3NO_2) δ = 1.6 (6H, m), 2.05 (3H, s), 2.82 (2H, t, J = 7.2 Hz), 3.35 (2H, t, J = 7.2 Hz), 3.78 (2H, br, CH_2CN), 4.16 (2H, br, CH_2CN), ^{13}C NMR (CD_3NO_2) δ = 24.8, 25.1, 30.1, 43.1, 50.7, 55.6, 191.1, 202.6 ppm; MS: m/z = 231; Analysis: $C_{10}H_{17}NOS_2$, Calcd: C, 51.91; H, 7.41; N, 6.05; S, 27.72; Obsd: C, 51.69; H, 7.62; N, 6.44; S, 27.33.

3.2.11 Compound (11). IR $\ddot{\nu}$ (cm^{-1}) = 1735 (C=O), 1210 (C=S); ^{1}H NMR (C_6D_6) δ = 2.73 (2H, t, J = 6.8 Hz), 3.36 (5H, br, CH_2N and OCH_3), 3.51 (4H, s, CH_2OCH_2), 3.59 (4H, m, CH_2N and SCH_2); ^{13}C NMR (C_6D_6) δ = 31.8, 34.1, 44.1, 48.3, 51.4, 51.8, 52.9, 53.8, 172.4, 196.1 ppm; MS: m/z = 249; Analysis: $C_9H_{15}NO_3S_2$, Calcd: C, 43.35; H, 6.06; N, 5.62; S, 25.72; Obsd: C, 43.56; H, 6.28; N, 5.40; S, 25.49.

3.2.12 Compound (12). IR $\ddot{\nu}$ (cm^{-1}) = 3358, 1648, 1220; ^{1}H NMR (CD_3NO_2) δ = 1.78 (6H, br), 2.70 (2H, t, J = 7.2 Hz), 3.54 (2H, t, J = 7.2 Hz, SCH_2), 3.96 (2H, br, CH_2N), 4.31 (2H, br, CH_2N), 6.0 and 6.33 (together 2H, br, NH_2), ^{13}C NMR (CD_3NO_2) δ = 24.8, 25.6, 34.8, 35.7, 50.2, 174.8, 200.5 ppm; MS: m/z = 232; Analysis: $C_9H_{16}N_2OS_2$, Calcd: C, 46.52; H, 6.94; N, 12.06; S, 27.60; Obsd: C, 46.26; H, 7.19; N, 12.27; S, 27.42.

3.2.13 Compound (13). IR $\ddot{\nu}$ (cm^{-1}) = 1735, 1211; ^{1}H NMR (C_6D_6) δ = 1.28 (3H, d, J = 7.2 Hz), 1.88–2.00 (4H, m), 2.81 (1H, m, CHCO), 3.38 (1H, d, J = 7.0 Hz, SCH_2), 3.46 (1H, d, J = 7.2 Hz, SCH_2), 3.59 (2H, t, J = 7.2 Hz, CH_2N), 3.63 (3H, s, OCH_3), 3.83 (3H, t, J = 7.2, CH_2N); ^{13}C NMR (C_6D_6) δ = 15.8, 25.2, 35.7, 40.2, 52.4, 52.8, 173.6, 199.4 ppm; MS: m/z = 247; Analysis: $C_{10}H_{17}NO_2S_2$, Calcd: C, 48.55; H, 6.93; N, 5.66; S, 25.92; Obsd: 48.77; H, 6.70; N, 5.89; S, 25.68.

3.2.14 Compound (14). IR $\ddot{\nu}$ (cm^{-1}) = 1712 (C=O), 1220 (C=S); ^{1}H NMR ($CDCl_3$) δ = 1.58 (3H, d, J = 6.8 Hz), 2.14 (3H, s), 2.89 (2H, t, J = 6.1 Hz), 3.40 (2H, t, J = 6.1 Hz), 5.81 (1H, q, J = 6.8 Hz), 7.26–7.35 (5H, m), 8.18 (1H, br, s, NH); ^{13}CNMR ($CDCl_3$), δ = 21.3, 29.0, 30.4, 43.3, 56.4, 127.0, 128.2, 129.3, 141.7, 196.8, 207.6 ppm; MS: m/z = 267; Analysis: $C_{13}H_{17}NOS_2$, Calcd: C, 58.39; H, 6.41; N, 5.24; S, 23.98; Obsd: C, 58.76; H, 6.22; N, 5.05; S, 24.33.

3.2.15 Compound (15). IR $\ddot{\nu}$ (cm^{-1}) = 1732 (C=O), 1210 (C=S); ^{1}H NMR ($CDCl_3$) δ = 1.59 (3H, d, J = 7.1 Hz), 2.77 (2H, t, J = 6.5 Hz), 3.50 (2H, t, J = 6.5 Hz), 3.67 (3H, s), 5.82 (1H, q, J = 7.1 Hz), 7.27–7.36 (5H, m), 7.97 (1H, br, NH); ^{13}CNMR ($CDCl_3$) δ = 21.2, 30.2, 34.1, 52.3, 56.3, 141.7, 173.0, 196.3 ppm; MS: m/z = 283; Analysis: $C_{13}H_{17}NO_2S_2$, Calcd: C, 55.09; H, 6.05; N, 4.94; S, 22.63; Obsd: C, 54.71; H, 6.43; N, 5.22; S, 22.36.

3.2.16 Compound (16). IR $\ddot{\nu}$ (cm^{-1}) = 2250 (CN), 1204 (C=S); ^{1}H NMR ($CDCl_3$) $\delta = 1.57$ (3H, d, $J = 6.8$ Hz), 2.73 (2H, t, $J = 6.7$ Hz), 3.40 (2H, t, $J = 6.7$ Hz), 5.79 (1H, q, $J = 6.7$ Hz), 7.29–7.37 (5H, m), 8.37 (1H, br, s, NH); ^{13}CNMR ($CDCl_3$) $\delta = 18.2, 21.8, 31.5, 57.5, 119.3, 127.1, 128.8, 129.5, 141.7, 194.9$ ppm; MS: $m/z = 250$; Analysis: $C_{12}H_{14}N_2S_2$, Calcd: C, 57.56; H, 5.64; N, 11.19; S, 25.61; Obsd: C, 57.23; H, 5.97; N, 11.44; S, 25.36.

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