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# An efficient, basic resin mediated, one-pot synthesis of dithiocarbazates from the corresponding alkyl halides

Devdutt Chaturvedi[a]; Nisha Mishra[b]; Virendra Mishra[b]
[a] Medicinal and Process Chemistry Division, Central Drug Research Institute, Lucknow, Uttar Pradesh, India [b] Department of Chemistry, B. S. A. College, Synthetic Research Laboratory, Mathura, Uttar Pradesh, India

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# An efficient, basic resin mediated, one-pot synthesis of dithiocarbazates from the corresponding alkyl halides

Devdutt Chaturvedi[a]*, Nisha Mishra[b] and Virendra Mishra[b]

[a]*Medicinal and Process Chemistry Division, Central Drug Research Institute, Lucknow, Uttar Pradesh, India;* [b]*Synthetic Research Laboratory, Department of Chemistry, B. S. A. College, Mathura, Uttar Pradesh, India*

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A quick and efficient, one-pot synthesis of dithiocarbazates was accomplished in high yields by reaction of various alkyl halides with substituted hydrazines using Amberlite IRA 400 (basic resin)/$CS_2$ system. The reaction conditions are mild with extremely simple work-up procedures than the reported methods.

**Keywords:** alkyl halides; Amberlite IRA 400; carbon disulfide; substituted hydrazines; dithiocarbazates

## 1. 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 (*3–6*), agrochemicals (*7–10*), intermediates in organic synthesis (*11–13*), protection of amino groups in peptide synthesis (*14–17*), linkers in solid phase organic synthesis (*18, 19*), and as donor ligands in complexation reactions with transition metals (*20–22*). To satisfy the demand, their synthesis has been changed from the use of costly and toxic chemicals such as thiophosgene (*23*) and its derivatives (*24, 25*) directly or indirectly, to the abundantly available cheap and safe reagents like $CS_2$. Moreover, their formation using $CS_2$ employed harsh reaction conditions using strong bases, high reaction temperatures, and longer reaction times (*26, 27*). Thus, we were prompted to embark on the improved procedures. Our group (*28–40*) has been engaged for 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 $CO_2$ and $CS_2$, respectively. More recently (*41–47*), 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 (*48*) for the tetrahydropyranylation of alcohols and phenols. In the present communication, we report herein efficient, one-pot,

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

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synthesis of dithiocarbazates from a variety of primary, secondary, and tertiary alkyl halides and substituted hydrazines using basic resin/$CS_2$ system.

## 2. Results and discussion

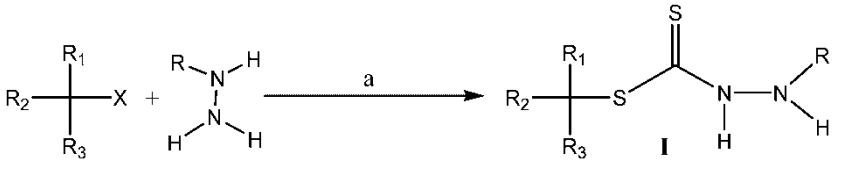
In connection with our ongoing interest pertaining to the use of Amberlite IRA 400 (basic resin) for the synthesis of carbamates, dithiocarbamates, and dithiocarbonates (xanthates) (*41–47*), we now wish to report a simple and effective one-pot procedure for the preparation of dithiocarbazates from a variety of primary, secondary, and tertiary alkyl halides and substituted hydrazines employing basic resin/$CS_2$ system. Thus, a mixture of substituted hydrazine and $CS_2$ in dry dimethyl sulfoxide (DMSO) and Amberlite IRA 400 (basic resin) was added. The reaction was stirred for 30 min at room temperature and then corresponding alkyl halide was added. Reaction was further continued until the completion of the starting materials checked by thin layer chromatography (TLC) (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 successful and the desired products isolated and further confirmed by various spectroscopic and analytical techniques. Since the products were simply obtained by concentration of organic layer in *vacuo* after filtration of basic resin from the reaction mixture which 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, indicates the necessity of basic resin in caring out the reaction. The whole reaction conditions are shown in Scheme 1.



Table 1. Conversion of various alkyl halides into dithiocarbazates of general formula I.

| Entry | $R_1$ | $R_2$ | $R_3$ | $X$ | $R$ | Time (h) | Isolated yield (%) |
|---|---|---|---|---|---|---|---|
| 1 | $n$-$C_3H_7$ | H | H | Br | 4-MeO-Ph | 2 | 92 |
| 2 | $PhCH_2CH_2$ | H | H | Br | Ph | 2 | 95 |
| 3 | $PhCH_2$ | H | H | Cl | Ph | 2.5 | 85 |
| 4 | Ph | H | H | Cl | Bn | 3 | 90 |
| 5 | $C_2H_5$ | Me | H | Br | Bn | 3 | 88 |
| 6 | 4-MeO·Ph | H | H | Cl | 3-$NO_2$·Ph | 3 | 83 |
| 7 | $C_3H_7$ | H | H | Br | 4-$NO_2$·Ph | 3 | 84 |
| 8 | $C_3H_7$ | H | H | Br | 2,4-$NO_2$·Ph | 4 | 78 |
| 9 | $C_3H_7$ | H | H | Br | Naphthyl | 3 | 83 |
| 10 | $C_4H_9$ | $C_4H_9$ | H | Br | Ph | 3 | 89 |
| 11 | $C_4H_9$ | $C_4H_9$ | $C_4H_9$ | Br | Ph | 3 | 87 |
| 12 | $C_5H_{11}$ | H | H | Cl | Bn | 2.5 | 94 |
| 13 | $C_7H_{15}$ | H | H | Cl | Ph | 2.5 | 92 |
| 14 | $C_9H_{19}$ | H | H | Cl | Bn | 2 | 98 |
| 15 | $C_3H_7$ | $C_3H_7$ | H | Br | Ph. | 3 | 85 |
| 16 | Ph | $CH_3$ | H | Br | Ph | 3.5 | 82 |

Note: All products were characterized by IR, NMR and ms.




Scheme 1. Reagents and conditions: (a) Amberlite IRA 400, $CS_2$, dry DMSO, rt, 2–4 h.

Thus, we screened various 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 have developed a convenient and efficient protocol for one-pot, three component coupling of various hydrazines with a variety of primary, secondary, and *tert*. alkyl halides via $CS_2$ bridge using basic resin (Amberlite IRA 400). This method generates the corresponding dithiocarbazates 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 general method for the formation of carbon-sulfur bonds essential to various organic syntheses.

## 3. Experimental

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



### 3.1. *Typical experimental procedure*

To a stirred solution of substituted hydrazine (3 mmol) in anhydrous DMSO (5 mL) was slowly added, carbon disulfide (8 mmol) and basic resin (5 mmol) at room temperature. The mixture was then stirred for 0.5 h at which point the required alkyl halide (3 mmol) was added over a period of 5 min. The reaction mixture was further continued until the completion of reaction (Table 1) under an argon atmosphere. The reaction mixture was filtered to remove 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 afford desired compound.

### 3.2. *Data for Dithiocarbazates*

*Butyl 2-(4-methoxyphenyl)hydrazinecarbodithiolate* (1)

IR $\ddot{v}(cm^{-1}) = 675, 1210$; $^1H$ 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); $^{13}C$ 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; analysis: $C_{12}H_{18}N_2OS_2$, Calcd: C, 53.30; H, 6.71; N, 10.36; S, 23.72; Obsd: C, 53.24; H, 6.65; N, 10.33; S, 23.58.

*3-Phenylpropyl 2-phenylhydrazinecarbodithiolate* (2)

IR $\ddot{v}(cm^{-1}) = 676, 1205$; $H^1$ NMR ($CDCl_3$) $\delta = 2.05$ (s, NH), 2.30 (m, 2H, $PhCH_2CH_2CH_2$-S)), 2.56 (t, 2H, $J = 7.2$ Hz, $PhCH_2$), 2.87 (t, 2H, $PhCH_2CH_2CH_2S$), 4.03 (m, H, Ph*NH*), 6.66–7.12 (m, 10H, Ar-H); $^{13}C$ 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; analysis: $C_{16}H_{18}N_2S_2$, Calcd: C, 63.54; H, 6.00; N, 9.26; S, 21.20; Obsd: C, 63.35; H, 6.26; N, 9.17; S, 21.28.

*2-Phenylethyl 2-phenylhydrazine carbodithiolate* (3)

IR $\ddot{v}(cm^{-1}) = 673, 1203$; $H^1$ NMR ($CDCl_3$) $\delta = 2.10$ (s, H, NH), 3.20 (2H, t, $J = 6.5$, Hz, $PhCH_2CH_2S$), 3.24 (m, 2H, $J = 7.2$ Hz, $PhCH_2$), 4.52 (m, H, Ph*NH*), 6.69–7.15 (m, 10H, Ar-H); $^{13}C$ 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; analysis: $C_{15}H_{16}N_2S_2$, Calcd: C, 62.46; H, 5.59; N, 9.71; S, 22.23; Obsd: C, 62.70; H, 6.64; N, 9.59; S, 22.10.

*Benzyl 2-butylhydrazinecarbodithiolate* (4)

IR $\ddot{v}(cm^{-1}) = 676, 1207$; $H^1$ NMR ($CDCl_3$) $\delta = 1.05$ (t, 3H, $CH_3$), 1.33 (m, 2H, $CH_2CH_3$), 1.56 (m, 2H, $CH_2CH_2CH_3$), 2.05 (br, NH), 2.65 (m, 2H, $NHCH_2$), 4.13 (s, 2H, $PhCH_2$), 7.06–7.15 (m, 5H, Ar-H); $^{13}C$ 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; analysis: $C_{12}H_{18}N_2S_2$, Calcd: C, 56.65; H, 7.13; N, 11.01; S, 25.21; Obsd: C, 56.46; H, 7.35; N, 11.27; S, 25.12.

*sec-Butyl 2-butylhydrazinecarbodithiolate* (5)

IR $\ddot{v}(cm^{-1}) = 682, 1214$; $H^1$ NMR ($CDCl_3$) $\delta = 0.99$ (t, 3H, $CH_3$), 1.05 (t, 3H, $CH_3$), 1.35 (m, 2H, $CH_2CH_3$), 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, *CH*-S), $^{13}C$ 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; analysis: $C_9H_{20}N_2S_2$, Calcd: C,49.05; H, 9.15; N, 12.71; S, 29.10; Obsd: C, 49.33; H, 9.01; N, 12.75; S, 29.32.

*4-Methoxybenzyl 2-(3-nitrophenyl)hydrazinecarbodithiolate* (6)

IR $\ddot{v}(cm^{-1})$ = 678, 1211; $H^1$ NMR ($CDCl_3$) $\delta$ = 2.05 (br, H, *NH*PhOMe), 3.73 (s, 3H, $OCH_3$), 4.06 (br, H, $NHPhNO_2$), 6.65–7.66 (m, 8H, Ar-H); $^{13}C$ 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; analysis: $C_{15}H_{15}N_3O_3S_2$, Calcd: C, 51.56; H, 4.33; N, 12.03; S, 18.35; Obsd: C, 51.23; H, 4.50; N, 12.24; S, 18.03.

*Butyl 2-(4-nitrophenyl)hydrazinecarbodithiolate* (7)

IR $\ddot{v}(cm^{-1})$ = 666, 1203; $H^1$ NMR ($CDCl_3$) $\delta$ = 0.96 (t, 3H, $CH_3$), 1.33 (m, 2H, $CH_2CH_3$), 1.96 (m, 2H, $SCH_2CH_2$), 2.05 (br, H, N*H*), 2.87 (t, 2H, $SCH_2$), 4.04 (br, $NHArNO_2$), 6.92–8.15 (m, 4H, Ar-H); $^{13}C$ 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; analysis: $C_{11}H_{15}N_3O_2S_2$, Calcd: C, 46.29; H, 5.30; N, 14.72; S, 22.47; Obsd: C, 46.45; H, 5.17; N, 14.47; S, 22.21.

*Butyl 2-(2,4-dinitrophenyl)hydrazinecarbodithiolate* (8)

IR $\ddot{v}(cm^{-1})$ = 670, 1212; $H^1$ NMR ($CDCl_3$) $\delta$ = 0.94 (t, 3H, $CH_3$), 1.32 (m, 2H, $CH_2CH_3$), 1.95 (m, 2H, $SCH_2.CH_2$), 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); $^{13}C$ 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; analysis: $C_{11}H_{14}N_4O_4S_2$, Calcd: C, 39.99; H, 4.27; N, 16.96; S, 19.41; Obsd: C, 40.22; H, 4.05; N, 16.76, S, 19.50.

*Butyl 2-(naphth-2-yl) hydrazinecarbodithiolate* (9)

IR $\ddot{v}(cm^{-1})$ = 677, 1209; $^1$H NMR ($CDCl_3$) $\delta$ = 0.95 (t, 3H, $CH_3$), 1.33 (m, 2H, $CH_2CH_3$), 1.97 (m, 2H, $SCH_2.CH_2$), 2.05 (br, H, N*H*), 2.84 (t, 2H, S$CH_2$), 4.05 (br, *NH*Ar$NO_2$), 6.76–7.55 (m, 7H, Ar-H); $^{13}$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; analysis: $C_{15}H_{18}N_2S_2$, Calcd: C, 62.03; H, 6.25; N, 9.64; S, 22.08; Obsd: C, 62.44; H, 6.33; N, 9.53; S, 22.25.

*1-Butylpentyl 2-phenylhydrazinecarbodithiolate* (10)

IR $\ddot{v}(cm^{-1})$ = 677, 1212; $^1$H 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, CH$CH_2$), 2.05 (br, N*H*), 2.52 (t, H, S*CH*), 4.05 (br, H, *NH*Ar), 6.66–7.18 (m, 5H, Ar-H); $^{13}$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; analysis: $C_{16}H_{26}N_2S_2$, Calcd: C, 61.89; H, 8.44; N, 9.02; S, 20.65; Obsd: C, 61.77; H, 8.54; N, 9.22; S, 20.46.

*1,1-Dibutylpentyl 2-phenylhydrazinecarbodithiolate* (11)

IR $\ddot{v}(cm^{-1})$ = 669, 1210; $^1$H NMR ($CDCl_3$) $\delta$ = 0.96 (t, 6H, $CH_3$), 1.29 (m, 4H, $CH_2CH_2C$), 1.33 (m, 4H, $CH_2CH_3$), 1.88 (m, 4H, CH$CH_2$), 2.04 (br, H, N*H*), 4.0 (br, H, *NH*-Ar), 6.67–7.19 (m, 5H, Ar-H); $^{13}$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; analysis: $C_{20}H_{34}N_2S_2$, Calcd: C, 65.52; H, 9.35; N, 7.64; S, 17.49; Obsd: C, 65.27; H, 9.11; N, 7.44; S, 17.49.

*Hexyl 2-butylhydrazinecarbodithiolate* (12)

IR $\ddot{v}(cm^{-1})$ = 674, 1208; $^1$H 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, NH$CH_2$), 2.87 (t, 2H, S$CH_2$), $^{13}$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; analysis: $C_{11}H_{24}N_2S_2$, Calcd: C, 53.18; H, 9.74; N, 11.28; S, 25.81; Obsd: C, 53.33; H, 9.54; N, 11.39; S, 25.64.

*n-Octyl 2-phenylhydrazinecarbodithiolate* (13)

IR $\ddot{v}(cm^{-1})$ = 679, 1211; $^1$H 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, S$CH_2$), 4.0 (br, H, Ph.*NH*), 6.65–7.20 (m, 5H, Ar-H), $^{13}$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; analysis: $C_{15}H_{24}N_2S_2$, Calcd: C, 60.76; H, 8.16; N, 9.45; S, 21.63; Obsd: 60.55; H, 8.33 N, 9.30; S, 21.77.

*Decyl 2-butylhydrazinecarbodithiolate* (14)

IR $\ddot{v}(cm^{-1})$ = 673, 1220; $^1$H 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.NH), 2.65 (m, 2H, NH$CH_2$), 2.87 (t, 2H, S$CH_2$), $^{13}$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; analysis: $C_{15}H_{32}N_2S_2$, Calcd: C, 59.15; H, 10.59; N, 9.20; S, 21.06; Obsd: C, 59.30; H, 10.34; N, 9.21; S, 21.24.

*1-Propylbutyl 2-phenylhydrazinecarbodithiolate* (15)

IR $\ddot{v}(cm^{-1}) = 675, 1210$; $^1H$ 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); $^{13}C$ NMR ($CDCl_3$) $\delta = 14.5, 20.1, 38.4, 40.8, 112.5, 118.3, 129.6, 143.3, 222.1$ ppm; MS: $m/z = 282$; analysis: $C_{14}H_{22}N_2S_2$, Calcd: C, 59.53; H, 7.85; N, 9.92; S, 22.70; Obsd: C, 59.75; H, 7.66; N, 9.92; S, 22.44.

*1-Phenylethyl 2-phenylhydrazinecarbodithiolate* (16)

IR $\ddot{v}(cm^{-1}) = 678, 1210$; $^1H$ 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), $^{13}C$ 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: $m/z = 288$; analysis: $C_{15}H_{16}N_2S_2$, Calcd: C, 62.46; H, 5.59; N, 9.71; S, 22.23; Obsd: C, 62.33; H, 5.46; N, 9.99; S, 22.36.

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