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Supplementary material

New Facile One-Pot Synthesis of Isobutyl Thiocarbamate in Recycling Solvent Mixture

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1. Experimental part

1.1. Materials

All reagents and chemicals for the synthesis were purchased from commercial sources (Sigma, St. Louis, MO, USA). All solvents were of reagent purity and used without further purification. Isobutanol - iBuOH (98.0%), potassium hydroxide - KOH (85%), carbon disulfide – CS_2 (98.0%), xylene (99%), calcium hydroxide (≥95.0%) were purchased from Merck. Ammonium hydroxide (30-32%) and deuterated chloroform (99.8% of deuterium), were supplied from Sigma Aldrich. All amines: 70% ethyl amine – $EtNH_2$ (70.0%), propyl amine – $PrNH_2$ (99.0%), butyl amine – $BuNH_2$ (99.0%), isopropyl amine – $iPrNH_2$ (99.5%), isobutyl amine – $iBuNH_2$ (98.0%), isopentyl amine – $iPeNH_2$ (98.0%), sec-butyl amine – $sBuNH_2$ (98.5%), cyclopropyl amine – $cycPrNH_2$ (98.0%), cyclopentyl amine – $cycPeNH_2$ (98,0%), cyclohexyl amine – $cycHeNH_2$ (98,5%), diethyl amine – $diEtNH_2$ (99.5%), dipropyl amine – $diPrNH_2$ (96.6%), dibutyl amine – $diBuNH_2$ (97.5%) are provided from Fluka. Hydrochloric acid (37%), dichloromethane, and sodium sulfate were purchased from Merck.

1.2. Experimental results

Table S1. ^1H and ^{13}C NMR data, and results of elemental analysis of synthesized *N*-alkyl, *N*,*N*-dialkyl and *N*-cycloalkyl-*O*-isobutyl thiocarbamates.

Compound	^1H NMR (δ/ppm)	^{13}C NMR (δ/ppm)	Elementar analysis Counted/ found
iBuOC(S)NHEt	6.30 (1H, d, *J*=6.5, N-H), 4.12-4.28 (2H, q, OCH_2), 3.21-3.59 (2H, dq, NCH_2), 1.54-1.73 (1H, sex, $CH(CH_3)_2$, 0.82-1.07 (9H, m, $CH(CH_3)_2$,CH_2CH_3).	188.8 (C=S), 78.69 (OCH_2), 43.70 (NCH_2), 28.35 ($CH(CH_3)_2$), 19.24 ($CH(CH_3)_2$), 12.1 (CH_2CH_3).	%C 52.13; %H 9.38; %N 8.69; %O 9.92; %S 19.88. %C 52.18; %H 9.42; %N 8.66; %O 9.84; %S 19.90.
iBuOC(S)NHPr	6.40 (1H, d, *J*=6.6, N-H), 4.13-4.27 (2H, q, OCH_2), 3.20-3.57 (2H, dq, NCH_2), 1.94-2.15 (1H, sex, $CH(CH_3)_2$), 1.52-1.70 (2H, sex, CH_2CH_3), 0.90-1.05 (9H, m, $CH(CH_3)_2$,CH_2CH_3).	189.8 (C=S), 77.69 (OCH_2), 44.70 (NCH_2), 27.70 ($CH(CH_3)_2$), 22.25 (CH_2CH_3), 18.96 ($CH(CH_3)_2$), 12.0 (CH_2CH_3).	%C 60.78; %H 10.66; %N 6.44; %O 7.36; %S 14.75. %C 60.81; %H 10.70; %N 6.39; %O 7.34; %S 14.76.

iBuOC(S)NHBu	6.42 (1H, d, *J*=6.6, N-H), 4.15-4.29 (2H, q, OCH_2), 3.22-3.58 (2H, dq, NCH_2), 1.92-2.13 (1H, sex, $CH(CH_3)_2$), 1.52-1.70 (2H, sex, $CH_2CH_2CH_3$), 0.89-1.05 (9H, m, $CH(CH_3)_2$,CH_2CH_3).	189.7 (C=S), 77.85 (OCH_2), 42.70 (NCH_2), 32.17 (NCH_2CH_2), 28.22 ($CH(CH_3)_2$), 21.25 (CH_2CH_3), 19.21 ($CH(CH_3)_2$), 13.20 (CH_2CH_3).	%C 57.10; %H 10.12; %N 7.40; %O 8.45; %S 16.94. %C 57.15; %H 10.15; %N 7.39; %O 8.35; %S 16.96.
iBuOC(S)NHsBu	6.20 (1H, d, *J*=6.5, N-H), 4.12-4.26 (2H, dd, *J*=6.4, OCH_2), 3.76-3.90 (1H, qv, NCH), 1.92-2.19 (2H, m, CH_2CH_3), 1.42-1.69 (4H, m, $NCHCH_3$ i $CH(CH_3)_2$), 0.87-1.21 (9H, m, $CH(CH_3)_2$ i CH_2CH_3).	189.16 (C=S), 77.64 (OCH_2), 50.75 (NCH), 29.22 (CH_2CH_3), 28.84 ($CH(CH_3)_2$), 19.87 ($NCHCH_3$), 19.23 $CH(CH_3)CH_3$), 18.96 ($CH(CH_3)CH_3$), 10.20 (CH_2CH_3).	%C 63.62; %H 11.09; %N 5.71; %O 6.52; %S 13.07. %C 63.58; %H 11.16; %N 5.76; %O 6.46; %S 13.04.
iBuOC(S)NHiPr	6.22 (1H, d, *J*=6.8, N-H), 4.33-4.47 (1H, m, OCH_2), 4.15-4.28 (1H dd, *J*=6.7, NCH), 1.90-2.15 (1H, m, $CH_2CH(CH_3)_2$), 1.12-1.26 (6H, q, $NCH(CH_3)_2$), 0.94-1.01 (6H, t, $CH_2CH(CH_3)_2$).	189.12 (C=S), 77.66 (OCH_2), 45.23 (NCH), 27.66 ($CH_2CH(CH_3)_2$), 22.18 ($NCH(CH_3)CH_3$), 21.76($NCH(CH_3)CH_3$), 18.94 ($CH_2CH(CH_3)_2$).	%C 54.81; %H 9.78; %N 7.99; %O 9.13; %S 18.29. %C 54.69; %H 9.82; %N 8.06; %O 9.15; %S 18.28.
iBuOC(S)NHiBu	6.40 (1H, d, *J*=6.4, N-H), 4.19-4.28 (2H, q, OCH_2), 3.09-3.44 (2H, sex, NCH_2), 1.90-2.19 (1H, m, NCH_2CH), 1.74-1.89 (1H, qv, OCH_2CH), 0.93-1.01 (12H, m, $NCH_2CH(CH_3)_2$ i $OCH_2CH(CH_3)_2$).	189.94(C=S), 77.75(OCH_2), 50.40 (NCH_2), 28.28 (NCH_2CH), 27.73 (OCH_2CH), 20.05 ($NCH_2CH(CH_3)_2$), 19.96 ($OCH_2CH(CH_3)CH_3$), 18.97 ($OCH_2CH(CH_3)CH_3$)	%C 54.81; %H 9.78; %N 7.99; %O 9.13; %S 18.29 %C 54.75; %H 9.85; %N 8.11; %O 9.18; %S 18.11.
iBuOC(S)NHiPen	6.33 (1H, d, *J*=6.6, N-H), 4.17-4.27 (2H, q, OCH_2), 3.16-3.61 (2H, dq, NCH_2), 1.89-2.15 (1H, m, NCH_2CH_2CH), 1.63-1.76 (1H, m, OCH_2CH), 1.38-1.56 (2H, m, NCH_2CH_2), 1.62 (1H, m, N-CH_2CH_2CH) 0.87-1.01 (12H, m, $OCH_2CH(CH_3)_2$ i ($NCH_2CH_2CH(CH_3)_2$).	189.63 (C=S), 77.67 (OCH_2), 37.71 (NCH_2), 37.29 (NCH_2CH_2), 27.66 (OCH_2CH), 25.46 (NCH_2CH_2CH), 22.29 ($NCH_2CH_2CH(CH_3)CH_3$), 22.16 ($NCH_2CH_2CH(CH_3)CH_3$), 18.94 ($OCH_2CH(CH_3)_2$).	%C 59.07; %H 10.41; %N 6.89; % O 7.87; %S 15.77 %C 59.11; %H 10.38; %N 6.93; %O 7.82; %S 15.76.
iBuOC(S)NHcPr	6.42 (1H, d, *J*=6.8, N-H), 4.20-4.36 (2H, dd, *J*=6.7, OCH_2), 1.42-1.54 (2H, m, $CH(CH_3)_2$ i 1-Cpr), 0.82-0.97 (10H, m, $CH(CH_3)_2$ i 2-Cpr).	189.8 (C=S), 77.88 (OCH_2), 28.21 ($CH(CH_3)_2$), 25.21 (1-Cpr) 20.56 ($CH(CH_3)_2$)), 7.32 (2-Cpr).	%C 55.45; %H 8.73; %N 8.08; %O 9.23; %S 18.51. %C 55.34; %H 8.56; %N 8.21; %O 9.14; %S 18.65.

iBuOC(S)NHcPen	6.32 (1H, d, *J*=6.6, N-H), 4.16-4.27 (2H, dd, *J*=6.7, OCH_2), 1.46 (1H, m, $CH(CH_3)_2$) 1.81-2.15 (5H, m, 1- i 2-Cpent), 1.43-1.67 (7H, m, 3-Cpent i $CH(CH_3)_2$).	189.28(C=S), 77.78 (OCH_2), 56.43 (C1-Cpen), 37.78 (C2-Cpen), 32.31 (C2-Cpen), 27.75 ($CH(CH_3)_2$), 23.56 (C3-Cpen), 23.03 (C3-Cpen), 18.99 ($CH(CH_3)CH_3$), 18.74 ($CH(CH_3)CH_3$).	%C 57.10; %H 10.12; %N 7.40; %O 8.45; %S 16.94. %C 57.14; %H 10.04; %N 7.45; %O 8.42; %S 16.95.
iBuOC(S)NHcHex	6.23 (1H, d, *J*=6.4 N-H), 3.31 (2H, dd, *J*=6.9, OCH_2) 3.66-3.74 (1H, m, 1-Cheks), 1.87-2.12 (2H, m, 2-Cheks), 1.50-1.78 (3H, m, 4- i 2-Cheks), 1.05-1.44 (6H, m, 3-Cheks, 4-Cheks $CH(CH_3)_2$), 0.89-1.00 (6H, m, $CH(CH_3)_2$).	189.41(C=S), 77.62(OCH_2), 52.29 (C1-Chex), 32.39 (C2-Chex), 32.05 (C2-Cheks), 27.64 ($CH(CH_3)_2$), 25.27 (C4-Chex), 24.60 (C3-Chex), 24.47(C3-Chex), 18.97 ($CH(CH_3)_2$).	%C 59.66; %H 9.51; %N 6.96; %O 7.95; %S 15.93. %C 59.64; %H 9.58; %N 6.90; %O 7.97; %S 15.91.
iBuOC(S)N$(Et)_2$	4.18-4,20 (2H, d, *J*=6.5, OCH_2), 3.24-3.62 NCH_2 (2H, sex, NCH_2), 1.8-2.09 (1H, sex, $CH(CH_3)_2$), 0.87-1.16 (12H, qv, $N(CH_2CH_3)_2$) i$CH(CH_3)_2$).	187.76 (C=S), 77.64 (OCH_2), 42.79 $N(CH_2CH_3)_2$), 28.20 ($CH(CH_3)_2$), 19.18($CH(CH_3)_2$), 12.18 (CH_2CH_3).	%C 57.10; %H 10.12; %N 7.40; %O 8.45; %S 16.94. %C 57.12; %H 10.18; %N 7.48; %O 8.50; %S 16.91.
iBuOC(S)N$(Pr)_2$	4,.19-4.22 (2H, d, *J*=6.5, OCH_2), 3.74 (4H, sex, NCH_2), 1.97-2.11 (1H, qv, CH), 1.52-1.82 (4H, m, 2(CH_2CH_3)), 0.87-1.00 (12H, qv, $N(CH_2CH_2CH_3)_2$) i $CH(CH_3)_2$).	187.76 (C=S), 77.64 (OCH_2), 27.79 ($CH(CH_3)_2$), 21.20 ($N(CH_2CH_2CH_3)_2$), 20.90 (NCH_2CH_2) 19.83 ($CH(CH_3)CH_3$), 19.08($CH(CH_3)CH_3$), 11.18 (CH_2CH_3), 11.07 (CH_2CH_3).	%C 61.35; %H 9.83; %N 6.50; %O 7.43; %S 14.89. %C 61.39; %H 9.78; %N 6.48; %O 7.50; %S 14.85.
iBuOC(S)N$(Bu)_2$	4.19-4.22 (2H, d, *J*=6.8, OCH_2), 3.70-3.77 (4H, t, $N(CH_2)$, 3.38-3.45 (4H, t, $N(CH_2)$ $CH_2)_2$), 1.23-1.40 (5H, qv, 2(CH_2CH_3) i $CH(CH_3)_2$), 0.90-1.00 (12H, m, $CH(CH_3)_2$ i 2(CH_2CH_3)).	187,59(C=S), 77.64 (OCH_2), 48.65 ($N(CH_2)_2$) 29.99 ($N(CH_2)_2(CH_2)_2$), 28.70 ($CH(CH_3)_2$), 20.05 (($CH_2)_2(CH_3)_2$), 19.97($CH(CH_3)CH_3$), 19.10 ($CH(CH_3)CH_3$), 13.71 ($CH_2CH_2CH_3$), 13.66($CH_2CH_2CH_3$).	%C 57.10; %H 10.12; %N 7.40; %O 8.45; %S 16.94. %C 57.14; %H 10.16; %N 7.35; %O 8.43; %S 16.92.

s singlet; d doublet; dd double doublet, t triplet; q quartet, dq double quartet; qv quintet; sex sextet;hept heptet, m multiplet; Cpr cyclopropane ring; Cpent cyclopentane ring; Numeration of the cyclohexane ring (numbering begins with the nitrogen and oxygen atom).

Table S2. FTIR and MS data of *N*-alkyl, *N,N*-dialkyl and *N*-cycloalkyl-*O*-isobutyl thiocarbamate.

Compound	IR, ν_{max} cm^{-1}	MS m/z
iBuOC(S)NHEt	3255 (νNH), 3045, 2951 ($\nu_{as}CH_3$), 2935 ($\nu_{as}CH_2$), 2897 (ν_sCH_3 and ν_sCH_2), 2799 (ν_sCH_3 and ν_sCH_2), 1701, 1514 ($\delta_{as}CH_3$), 1434 (δ_sCH_3), 1397, 1329, 1260, 1203, 1119 (νC=S), 1032 (νC-N), 988 (γC-H), 884, 777.	170,15
iBuOC(S)NHPr	3259 (νNH), 3051, 2963 ($\nu_{as}CH_3$), 2935 ($\nu_{as}CH_2$), 2875 (ν_sCH_3 and ν_sCH_2), 2727, 1705, 1524 ($\delta_{as}CH_3$), 1464 (δ_sCH_3), 1406, 1332, 1262, 1199, 1124 (νC=S), 1037 (νC-N), 989 (γC-H), 891, 780 .	175,29
iBuOC(S)NHBu	3239 (νNH), 3045, 2945 ($\nu_{as}CH_3$ and ν_sCH_3), 2854 (ν_sCH_3 and ν_sCH_2), 2721, 1699, 1512 ($\delta_{as}CH_3$), 1450 (δ_sCH_3), 1396, 1321, 1270, 1206, 1119 (νC=S), 1037 (νC-N), 983 (γC-H), 776, 725.	185,45
iBuOC(S)NHsBu	3246 (νNH), 2966 ($\nu_{as}CH_3$), 2934 ($\nu_{as}CH_2$), 2876 (ν_sCH_3 and ν_sCH_2), 2727, 1701, 1519 ($\delta_{as}CH_3$), 1456 (δ_sCH_3), 1403, 1330, 1272, 1202, 1123 (νC=S), 1049 (νC-N), 1022, 998 (γC-H), 782, 723.	189,32
iBuOC(S)NHiPr	3248 (νNH), 2969 ($\nu_{as}CH_3$), 2935 ($\nu_{as}CH_2$), 2874 (ν_sCH_3 and ν_sCH_2), 1700, 1519 ($\delta_{as}CH_3$), 1475 (δ_sCH_3), 1403, 1368, 1314, 1205, 1120 (νC=S), 1039 (νC-N), 996 (γC-H), 728.	175,29
iBuOC(S)NHiBu	3252 (νNH), 2961 ($\nu_{as}CH_3$), 2934 ($\nu_{as}CH_2$), 2873 (ν_sCH_3 and ν_sCH_2), 1522 ($\delta_{as}CH_3$), 1468 (δ_sCH_3), 1406, 1385, 1332, 1201, 1126 (νC=S), 1030 (νC-N), 1090, 997 (γC-H).	189,32
iBuOC(S)NHiPent	3259 (νNH), 2959 ($\nu_{as}CH_3$), 2935 ($\nu_{as}CH_2$), 2873 (ν_sCH_3 and ν_sCH_2), 1522 ($\delta_{as}CH_3$), 1469 (δ_sCH_3), 1405, 1367, 1326, 1243, 1194, 1125 (νC=S), 1046 (νC-N), 989 (γC-H).	203,34
iBuOC(S)NHcPr	3244 (νNH), 2967 ($\nu_{as}CH_3$), 2938 ($\nu_{as}CH_2$), 2876 (ν_sCH_3 and ν_sCH_2), 1526 ($\delta_{as}CH_3$), 1474 (δ_sCH_3), 1412, 1392, 1338, 1212, 1134 (νC=S), 1060 (νC-N), 987 (γC-H).	173,28
iBuOC(S)NHcPen	3251 (νNH), 2960 ($\nu_{as}CH_3$), 2872 (ν_sCH_3 and ν_sCH_2), 2111, 1698, 1653, 1519 ($\delta_{as}CH_3$), 1470 (δ_sCH_3), 1404, 1347, 1293, 1203, 1168, 1088 (νC=S), 1050 (νC-N), 991 (γC-H), 964, 738, 653.	201,33
iBuOC(S)NHcHex	3399, 3244 (νNH), 2933 ($\nu_{as}CH_3$ and $\nu_{as}CH_2$), 2855 (ν_sCH_3 and ν_sCH_2), 2662, 2103, 1700, 1515 ($\delta_{as}CH_3$), 1450 (δ_sCH_3), 1403, 1347, 1251, 1172 (νC=S), 1015, 970 (γC-H), 892, 744.	215,36
iBuOC(S)N(Et)$_2$	2957 ($\nu_{as}CH_3$), 2928 ($\nu_{as}CH_2$), 2736, 1698, 1490 ($\delta_{as}CH_3$), 1459 (δ_sCH_3), 1413, 1349, 1313, 1276, 1393, 1217, 1160 (νC=S), 1033 (νC-N), 956 (γC-H), 891, 750.	216,93
iBuOC(S)N(Pr)$_2$	2964 ($\nu_{as}CH_3$), 2934 ($\nu_{as}CH_2$), 2875 (ν_sCH_3 and ν_sCH_2), 2730, 1700, 1499 ($\delta_{as}CH_3$), 1465 (δ_sCH_3), 1422, 1370, 1318, 1285, 1247, 1226, 1172 (νC=S), 1044 (νC-N), 965 (γC-H), 938, 894, 751.	217,37
iBuOC(S)N(Bu)$_2$	2960 ($\nu_{as}CH_3$), 2933 ($\nu_{as}CH_2$), 2873 (ν_sCH_3 and ν_sCH_2), 2732, 1735, 1690, 1652, 1501 ($\delta_{as}CH_3$), 1466 (δ_sCH_3), 1423, 1274, 1210, 1170 (νC=S), 1042 (νC-N), 965 (γC-H), 916.	245,42

Table S3. ^{1}H and ^{13}C NMR data, and results of elemental analysis of *O,O*-dialkyl dixanthates intermediates.

Compound	^{1}H NMR (δ/ppm)	^{13}C NMR (δ/ppm)	Elementar analysis Counted/ found
$(iBuOCS)_2S_2$	3.31 (4H, dd, *J*=6.9, OCH_2) 1.45 (2H, m, $CH(CH_3)_2$) 0.89-1.00 (6H, m, $CH(CH_3)_2$)	172.8 (C=S), 75.69 (OCH_2), 27.35 (CH), 19.24 ($CH(CH_3)_2$)	%C 40.54; %H 6.25; %O 10.52; %S 42.69; %C 40.24; %H 6.08; %O 10.72; %S 42.97;
$(EtOCS)_2S_2$	3.64 (2H, q, OCH_2) 1.32 (6H, m, CH_3)	172.0 (C=S), 69.7 (OCH_2), 14.0 (CH_3)	%C 29.55; %H 5.98; %O 10.82; %S 43.65; %C 29.73; %H 4.16; %O 13.20; %S 52.91;

Table S4. FTIR and MS data of *O,O*-dialkyl dixanthates intermediates.

Compound	IR, ν_{max} cm^{-1}	MS m/z
$(iBuOCS)_2S_2$	3045, 2951 ($\nu_{as}CH_3$), 2897 (ν_sCH_3 and ν_sCH_2), 2799 (ν_sCH_3 and ν_sCH_2), 1514 ($\delta_{as}CH_3$), 1434 (δ_sCH_3), 1397, 1329, 1260, 1203, 1119, 1015 (νC=S), 988 (γC-H), 884, 777 (νC-S), 700 (δC-S-S-C)	298.019
$(EtOCS)_2S_2$	2976 ($\nu_{as}CH_3$), 2932 (ν_sCH_3 and ν_sCH_2), 2870 (ν_sCH_3 and ν_sCH_2), 1450 ($\delta_{as}CH_3$), 1448, 1370, 1350 (δ_sCH_3), 1276, 1145, 1119, 1015 (νC=S), 890 (γC-H), 884, 786 (νC-S), 710 (δC-S-S-C)	241.956

FTIR spectra

Figure S1. FTIR spectrum of N-ethyl-O-isobutyl thiocarbamate.

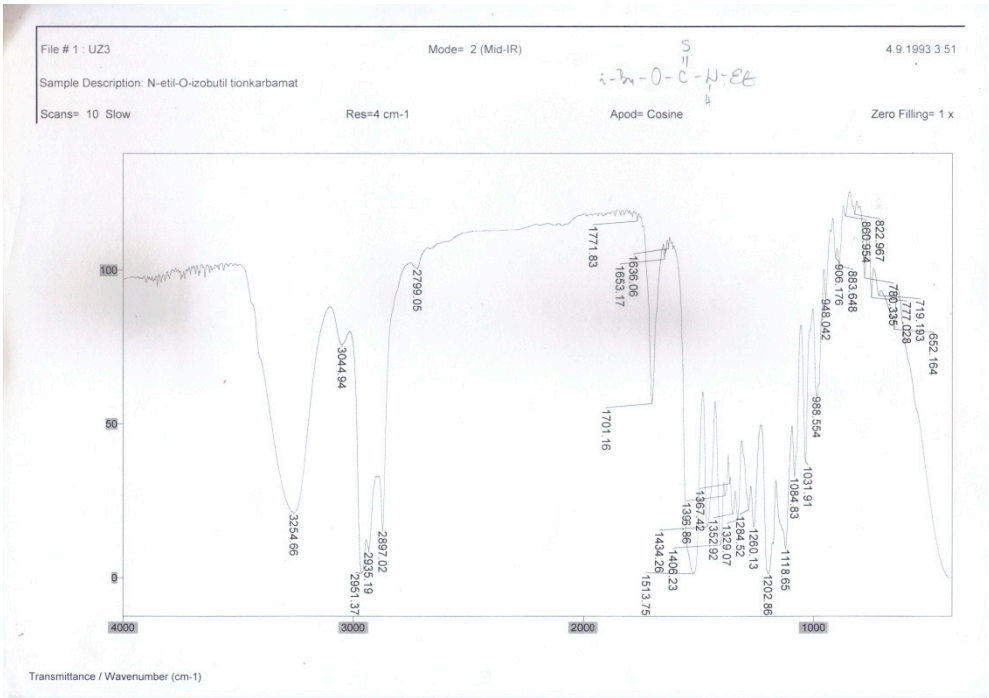


Figure S2. FTIR spectrum of N-propyl-O-isobutyl thiocarbamate.

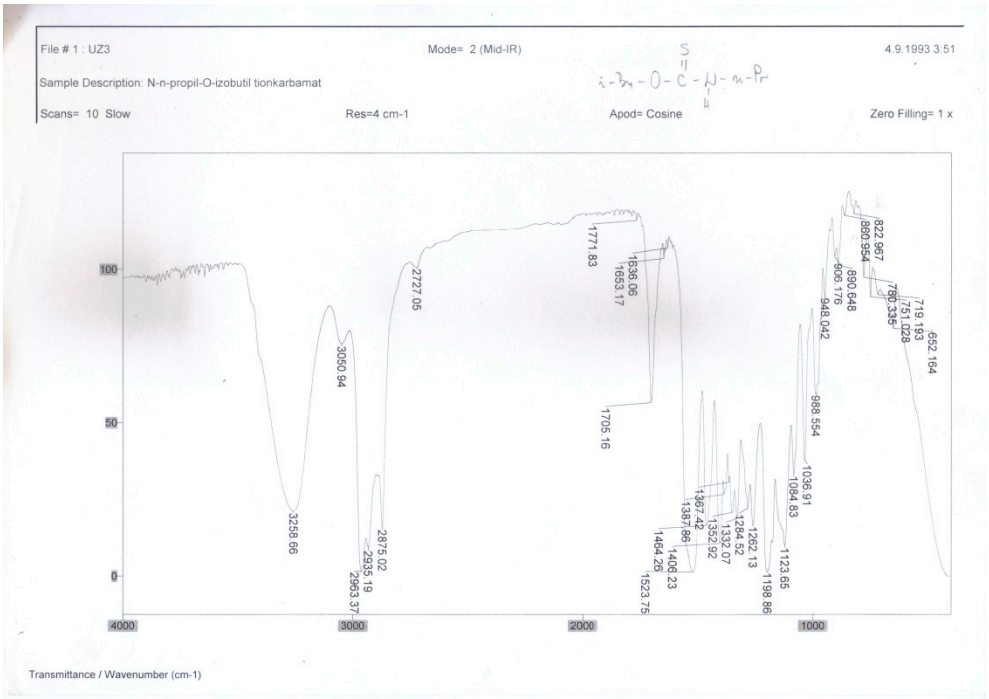


Figure S3. FTIR spectrum of N-butyl-O-isobutyl thiocarbamate.

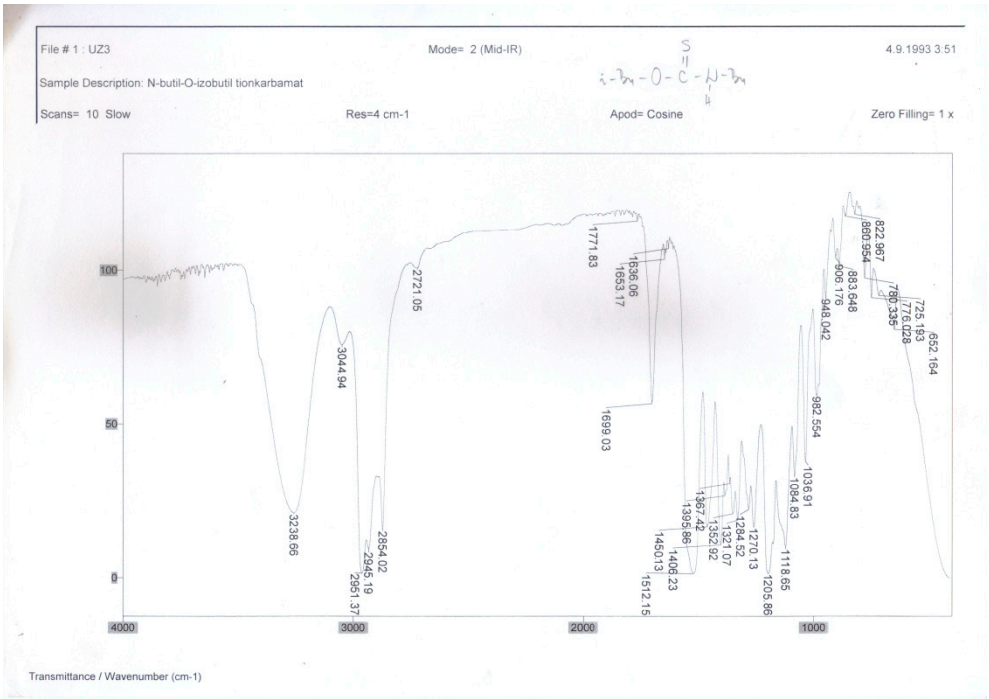


Figure S4. FTIR spectrum of N-*sec* butyl-O-isobutyl thiocarbamate.

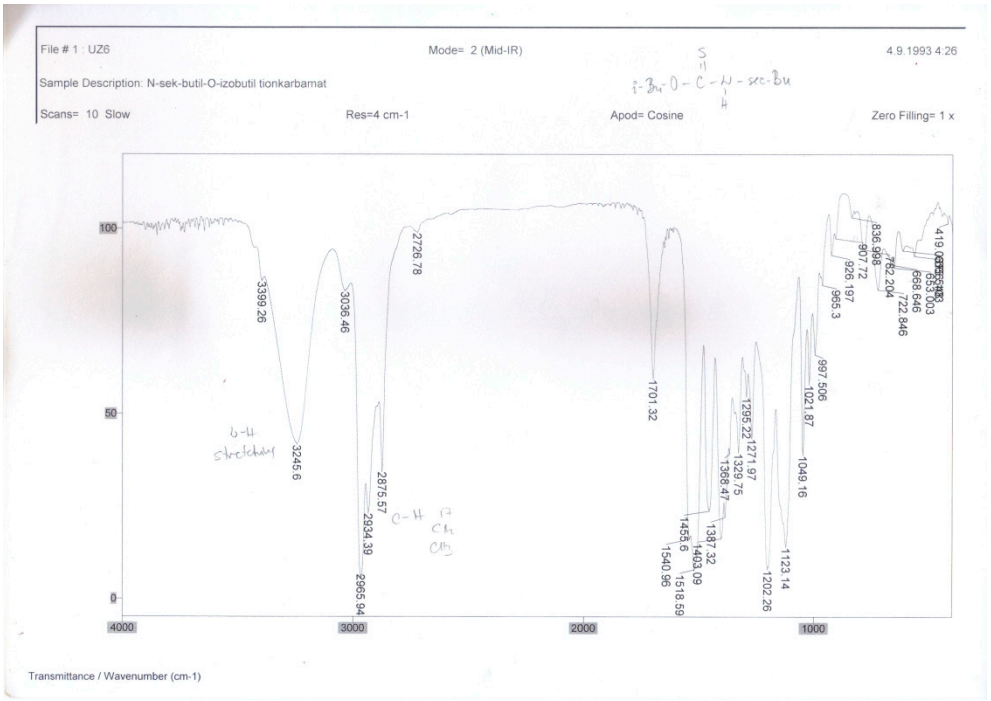


Figure S5. FTIR spectrum of N-isopropyl-O-isobutyl thiocarbamate.

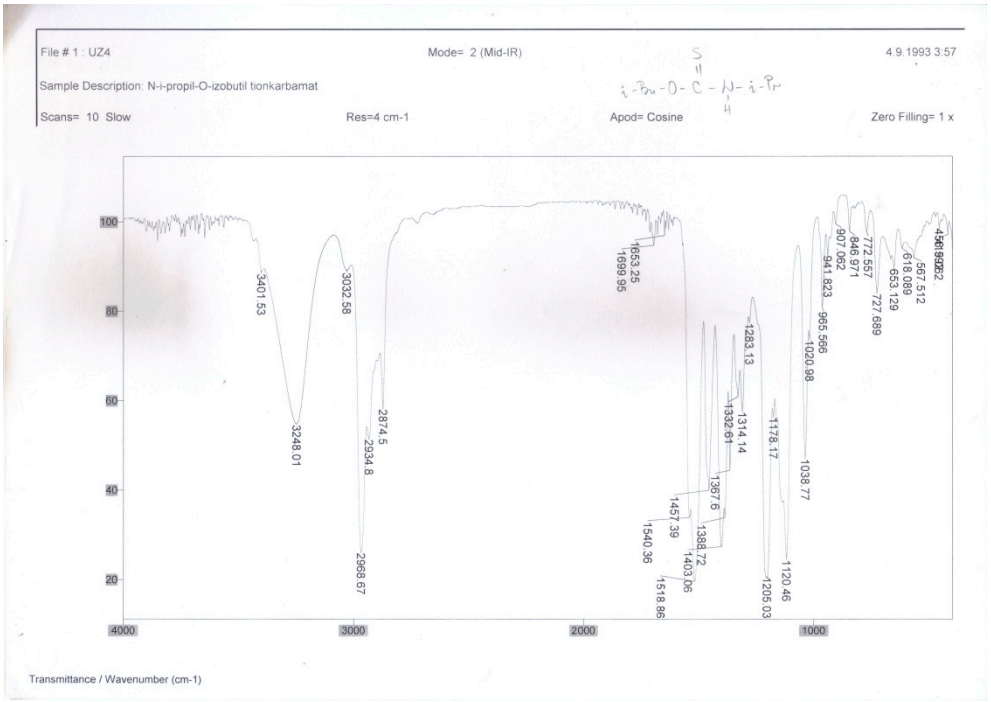


Figure S6. FTIR spectrum of N-isobutyl-O-isobutyl thiocarbamate.

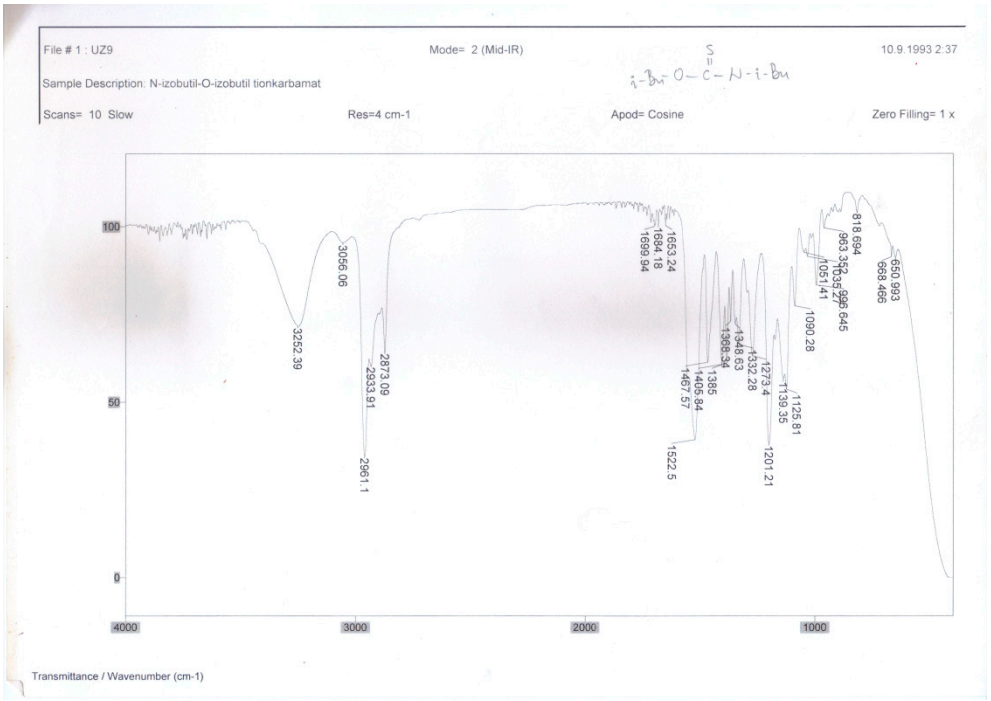


Figure S7. FTIR spectrum of N-isopentyl-O-isobutyl thiocarbamate.

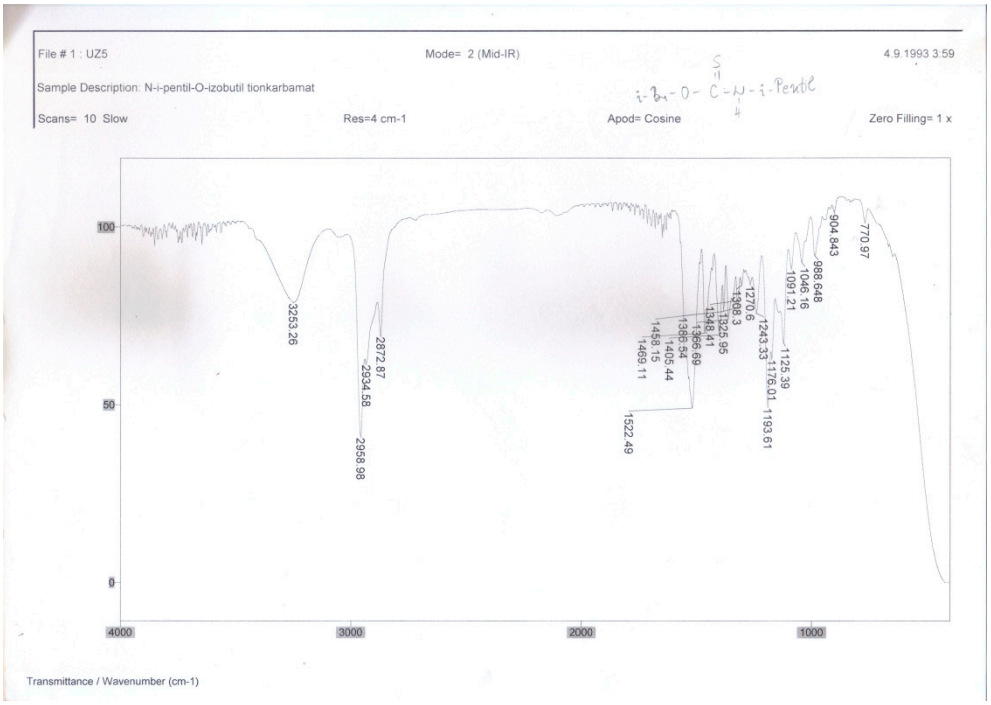


Figure S8. FTIR spectrum of N-cyclopentyl-O-isobutyl thiocarbamate.

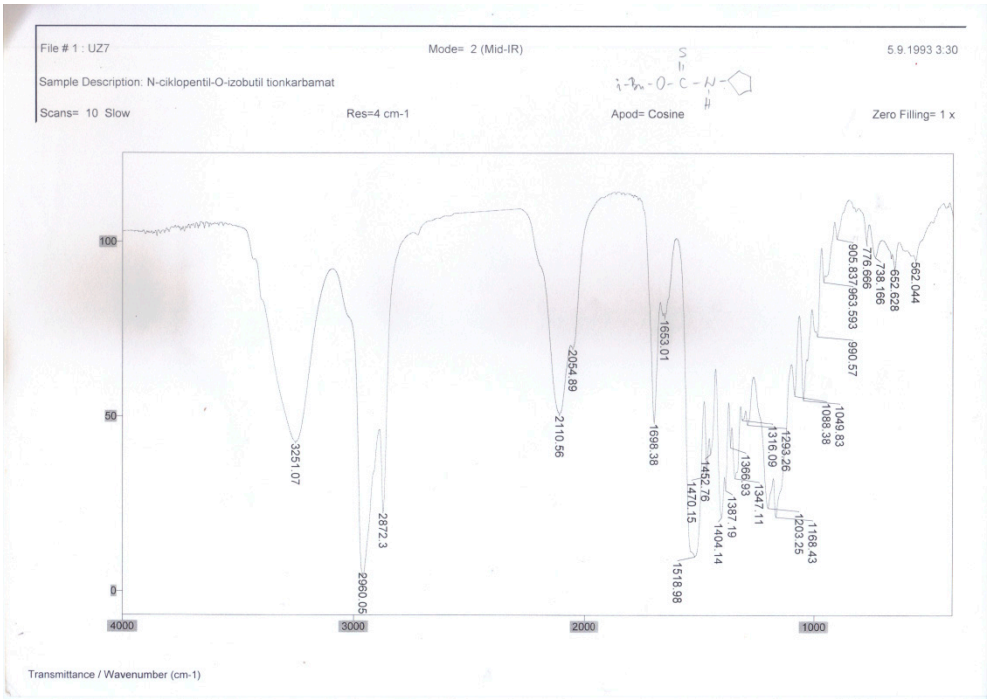


Figure S9. FTIR spectrum of N-cyclohexyl-O-isobutyl thiocarbamate.

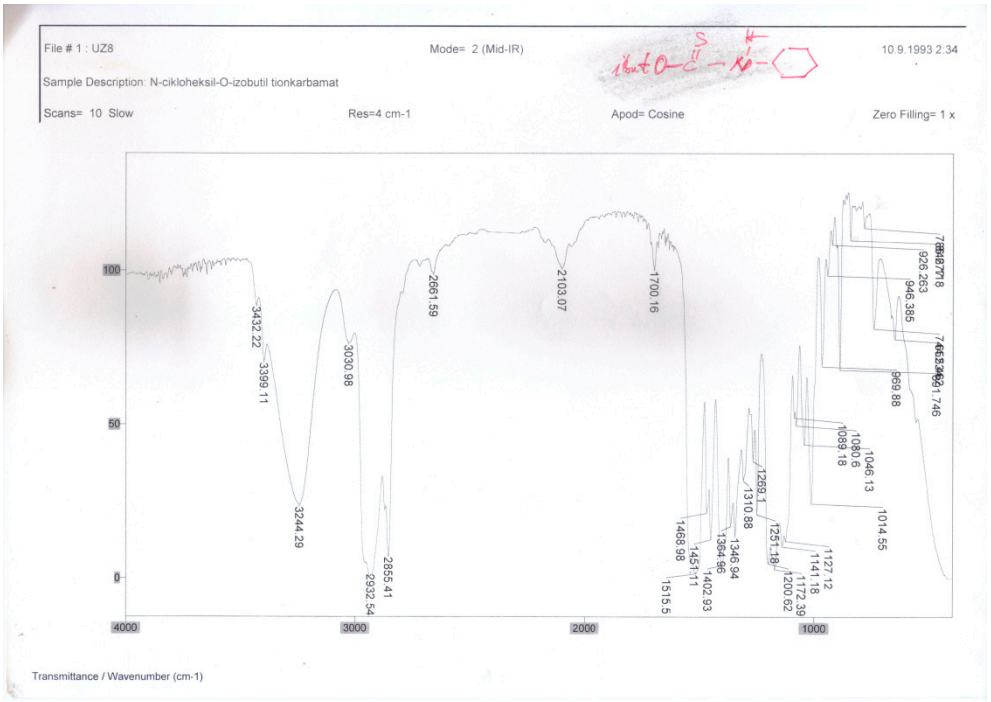


Figure S10. FTIR spectrum of N,N-dipropyl-O-isobutyl thiocarbamate.

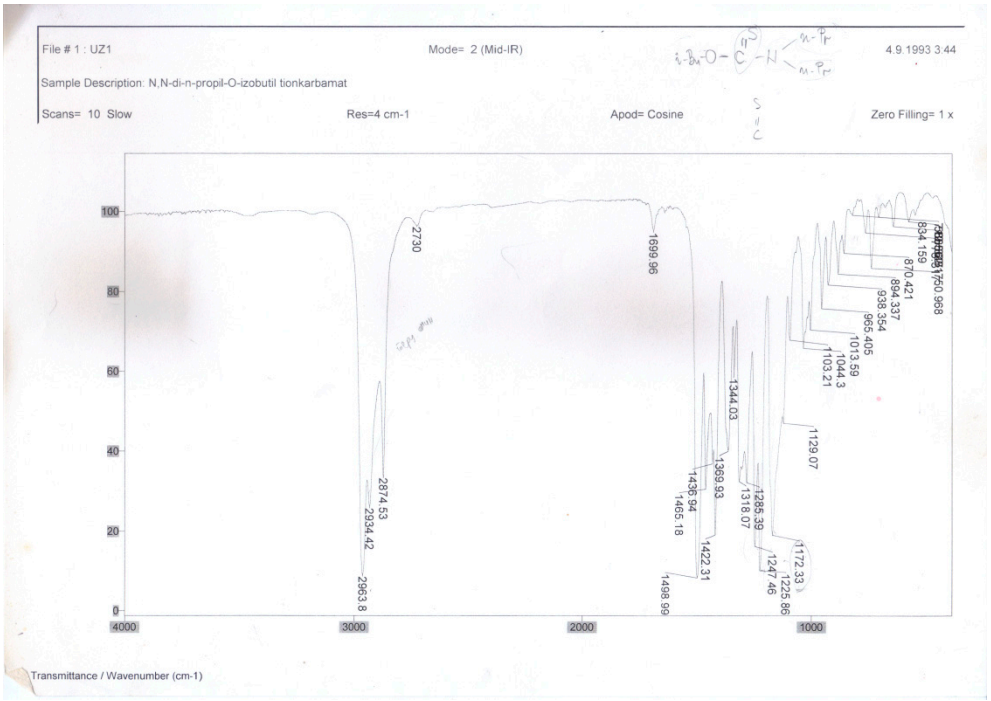


Figure S11. FTIR spectrum of N-propyl-O-ethoxy thiocarbamate.

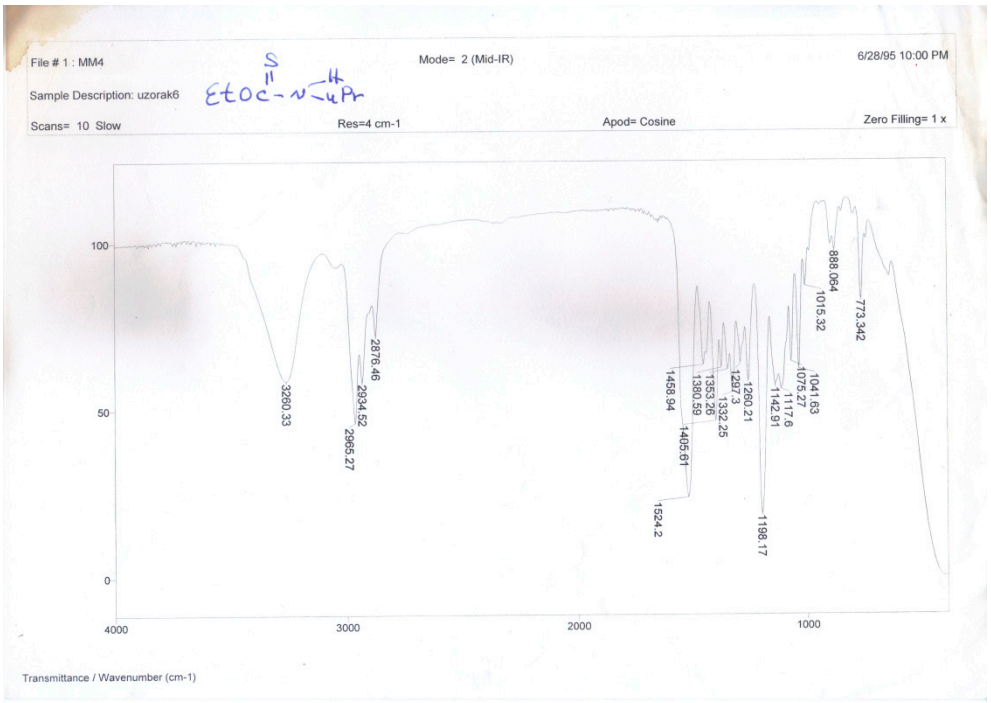
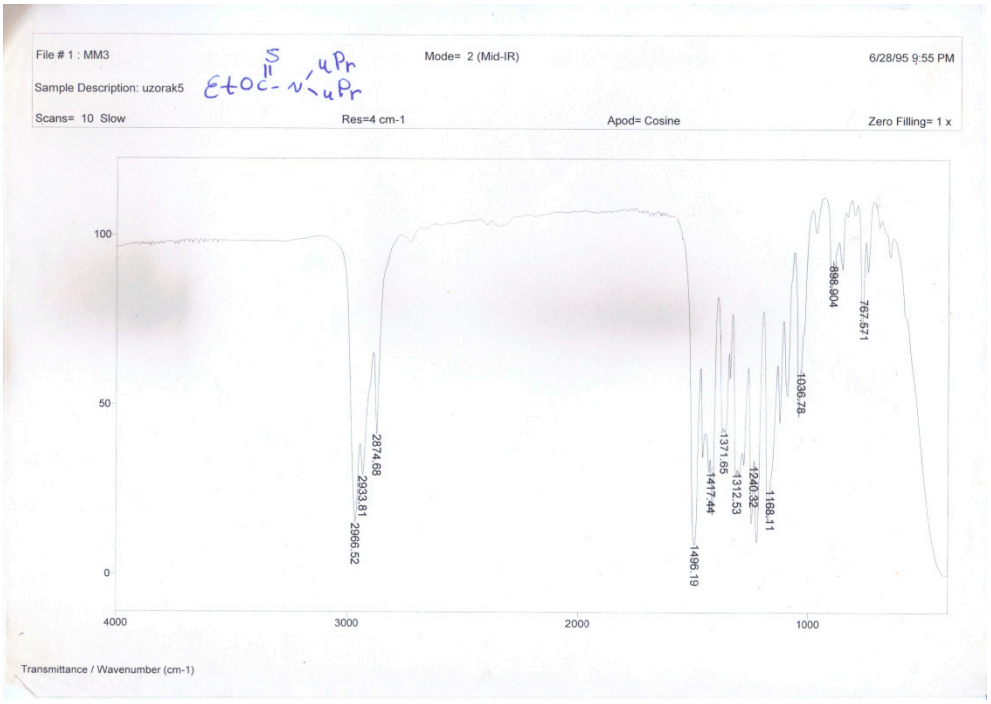


Figure S12. FTIR spectrum of N,N-dipropyl-O-ethoxy thiocarbamate.



^{13}C NMR spectra of synthesized thiocarbamates

Figure S13. ^{13}C NMR spectra of N-propyl-O-isobutyl thiocarbamate.

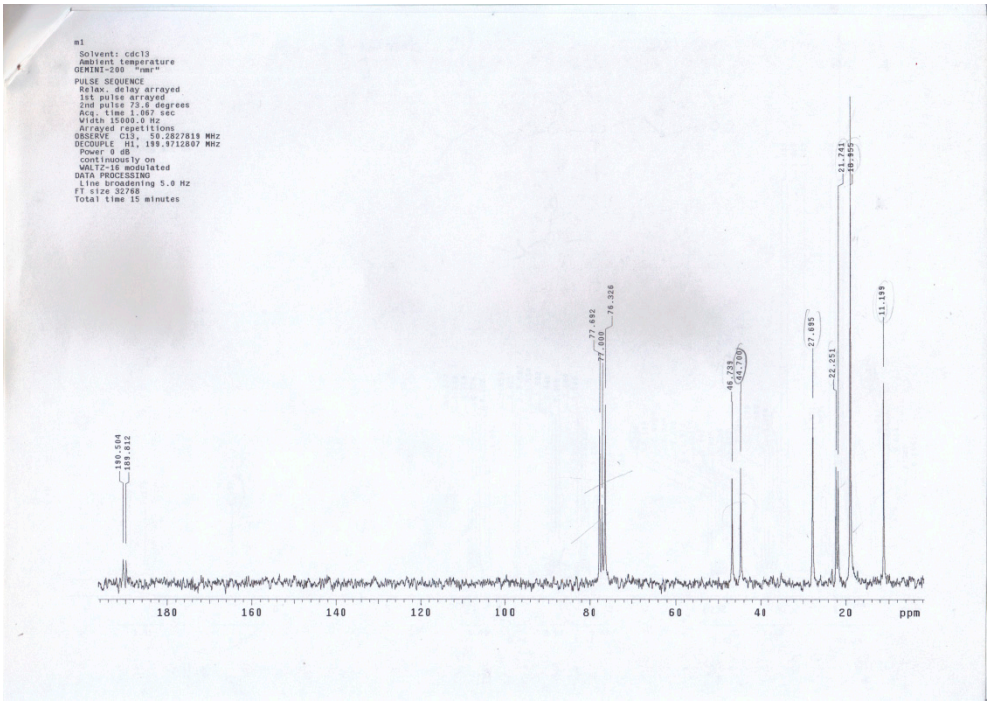


Figure S14. ^{13}C NMR spectra N-sec butyl-O-isobutyl thiocarbamate.

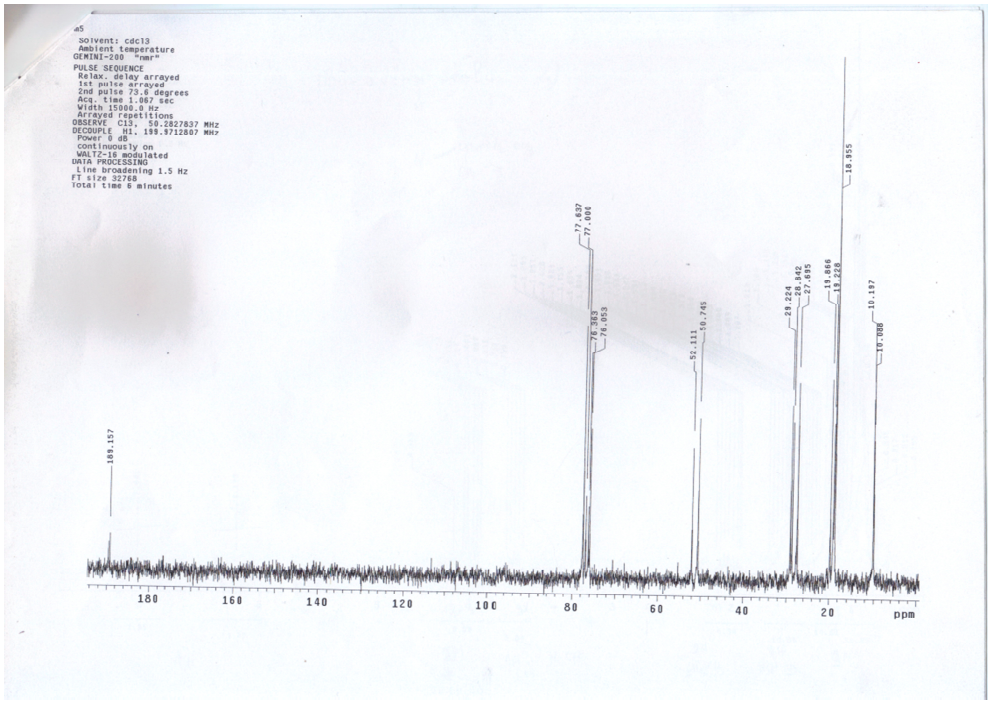


Figure S15. ^{13}C NMR spectra of N-isopropyl-O-isobutyl thiocarbamate.

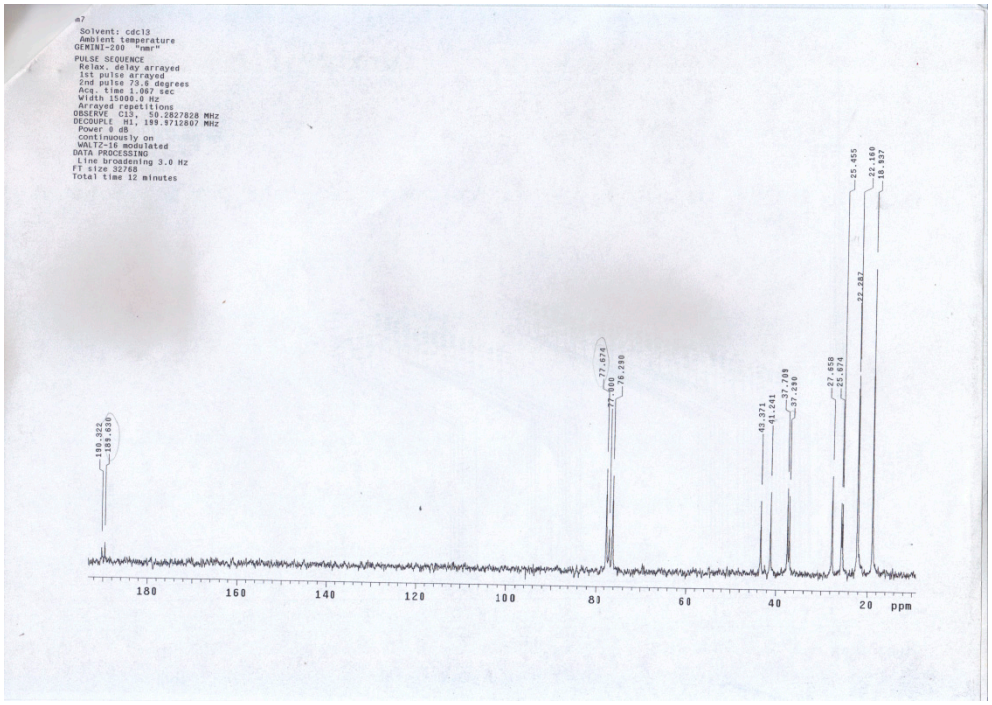


Figure S16. ^{13}C NMR spectra of N-isobutyl-O-isobutyl thiocarbamate.

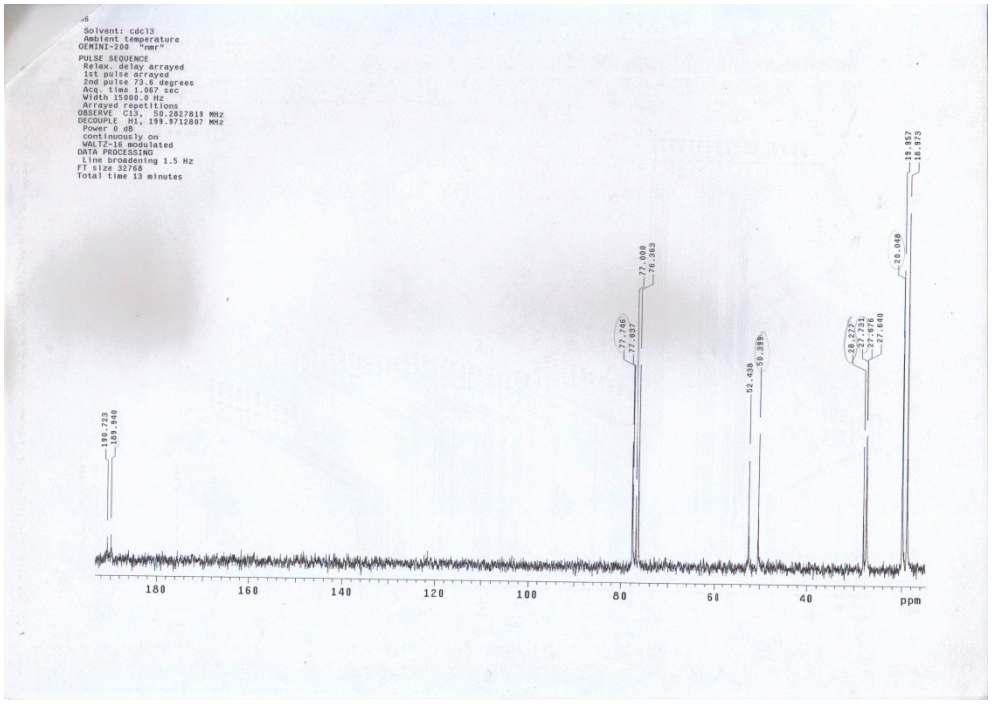


Figure S17. ^{13}C NMR spectra of N-isopentyl-O-isobutyl thiocarbamate.

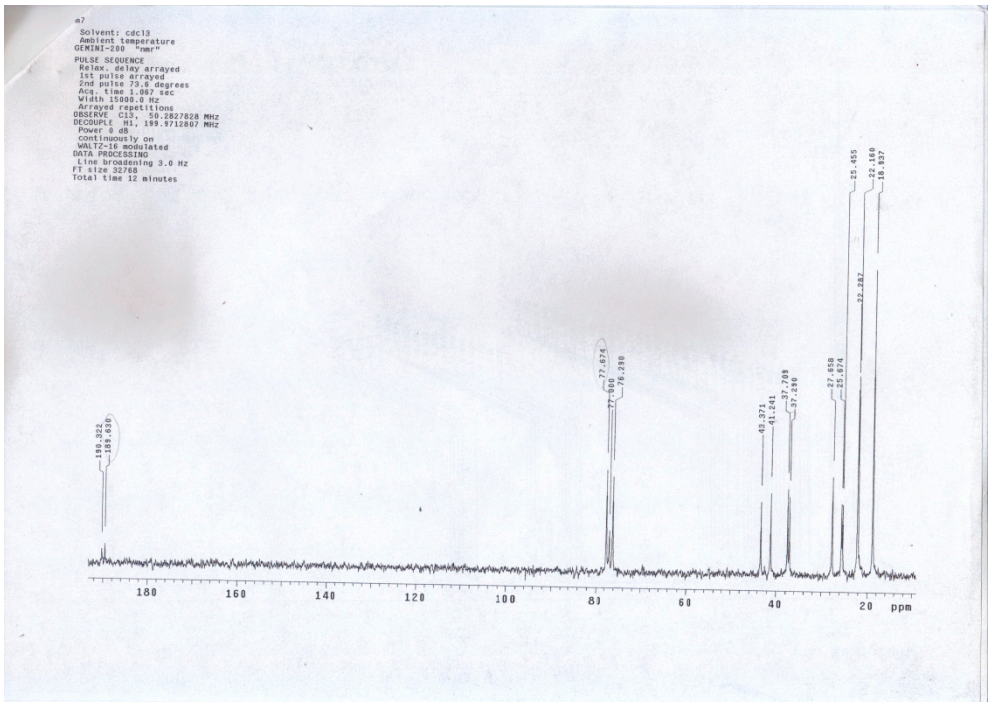


Figure S18. ^{13}C NMR spectra of N-cyclopentyl-O-isobutyl thiocarbamate.

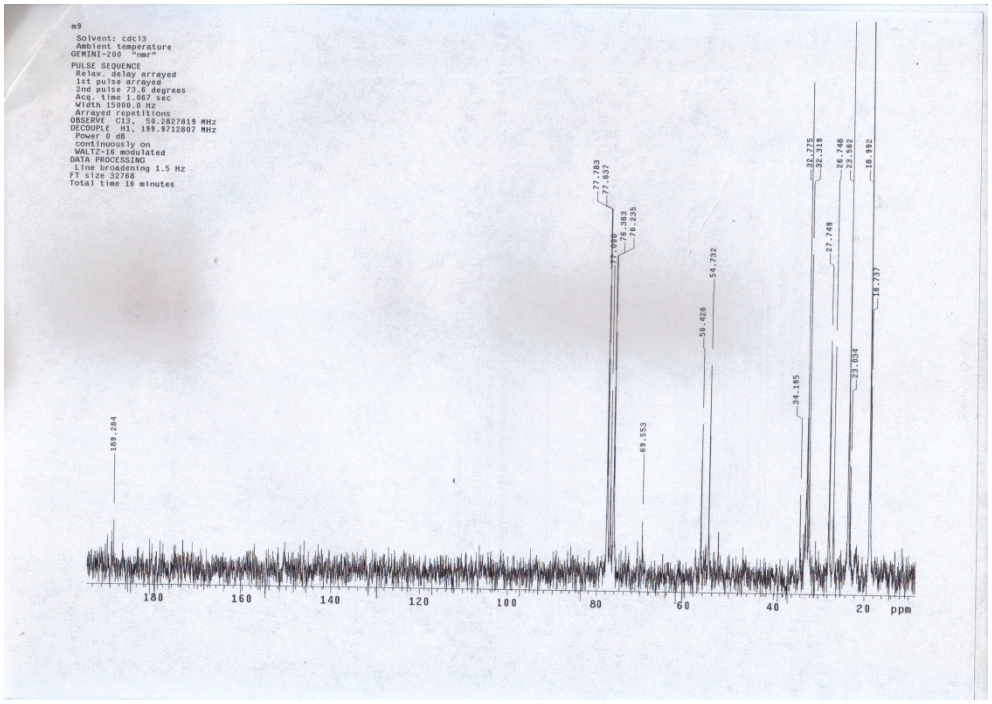


Figure S19. ^{13}C NMR spectra N-cyclohexyl-O-isobutyl thiocarbamate.

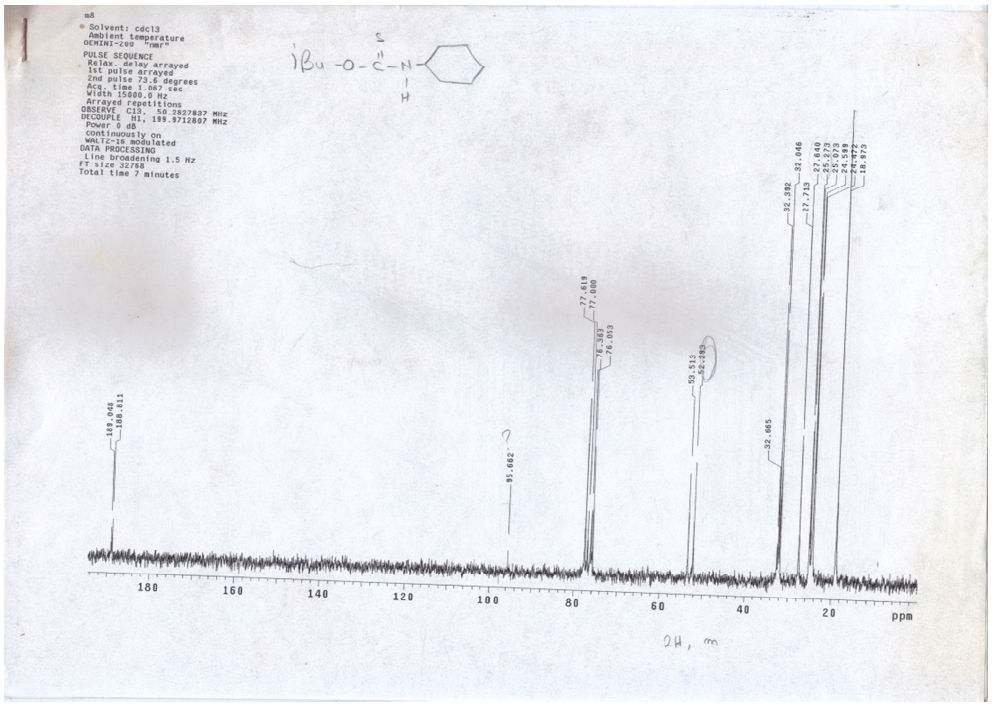


Figure S20. ^{13}C NMR spectra of N,N-dipropyl-O-isobutyl thiocarbamate.

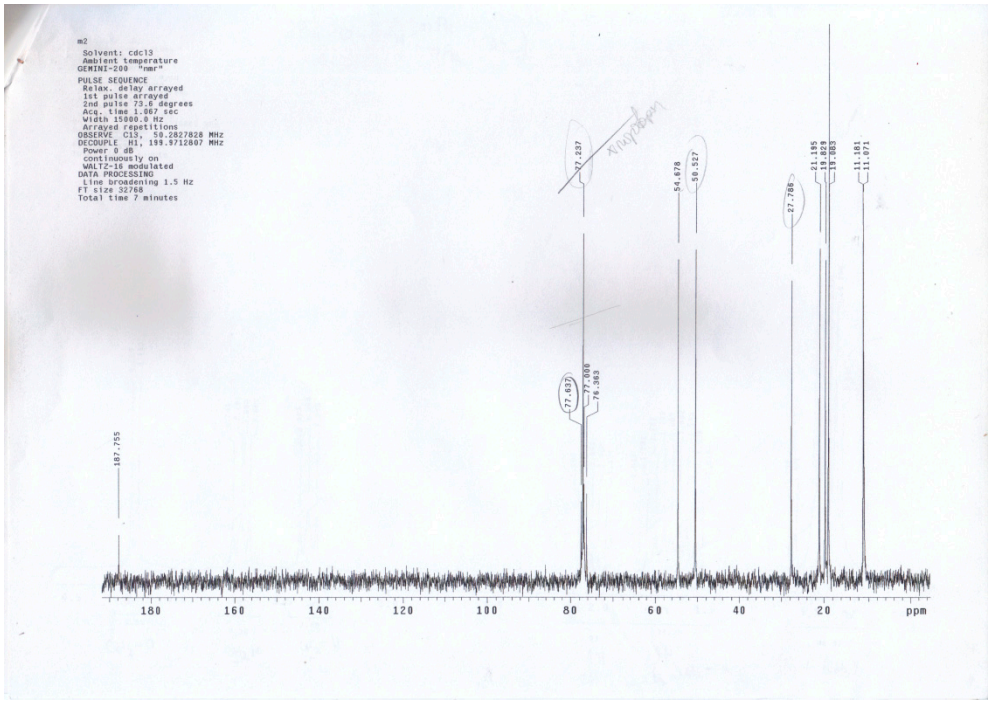


Figure S21. ^{13}C NMR spectra of N,N-dibutyl-O-isobutyl thiocarbamate.

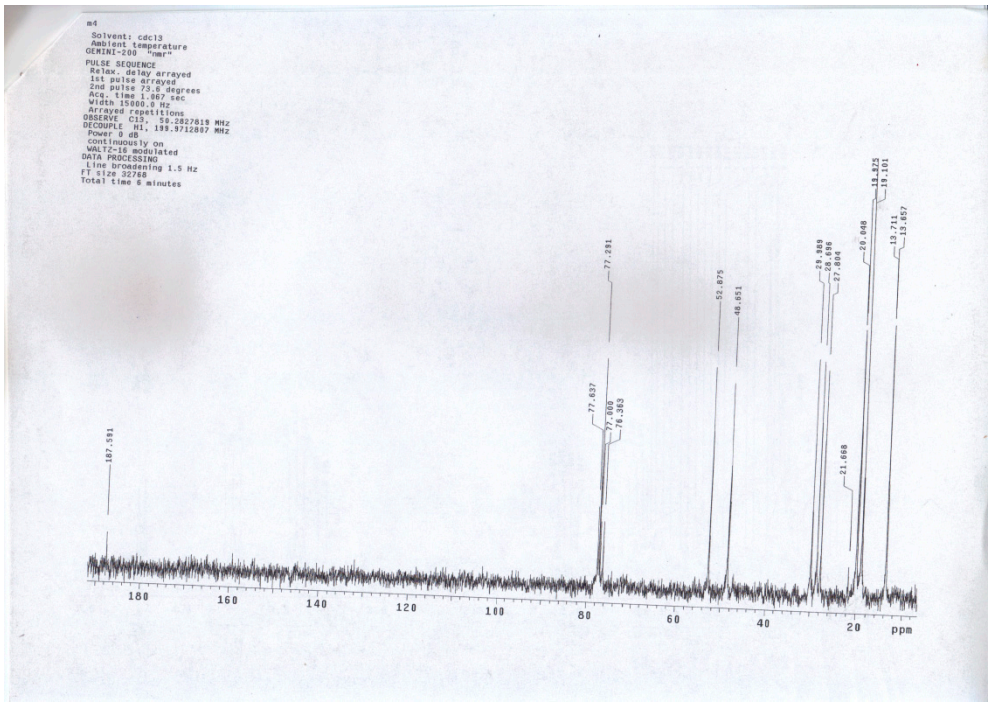


Figure S22. ^{13}C NMR spectra of N,N-dipropyl-O-isobutyl thiocarbamate.

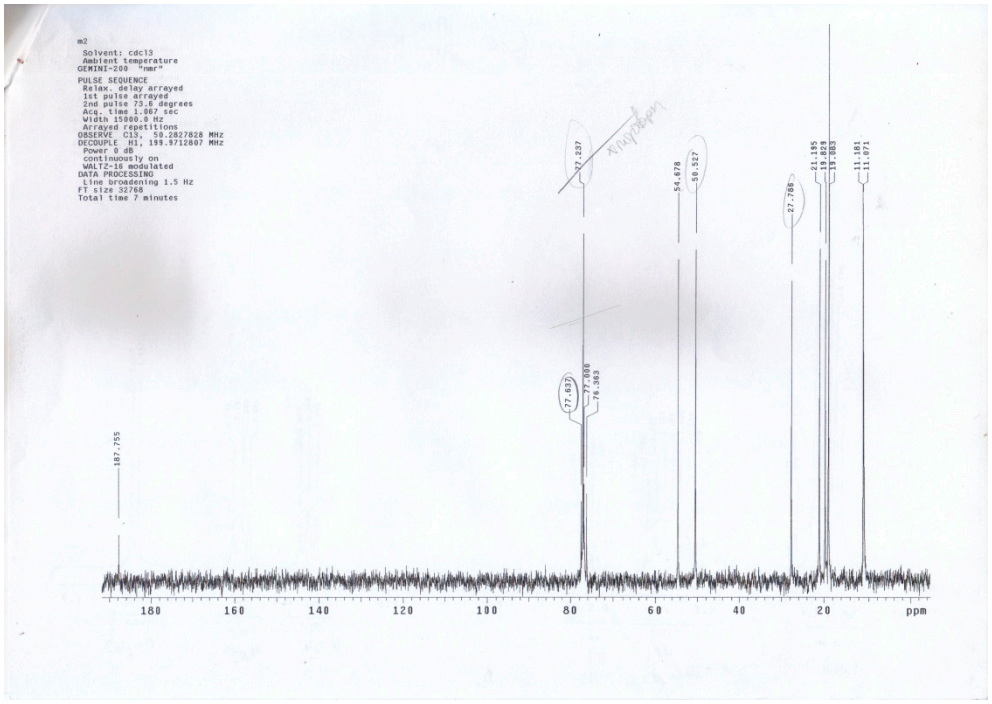


Figure S23. GC-MS chromatogram for N-isobutyl-O-isobutyl thiocarbamate.

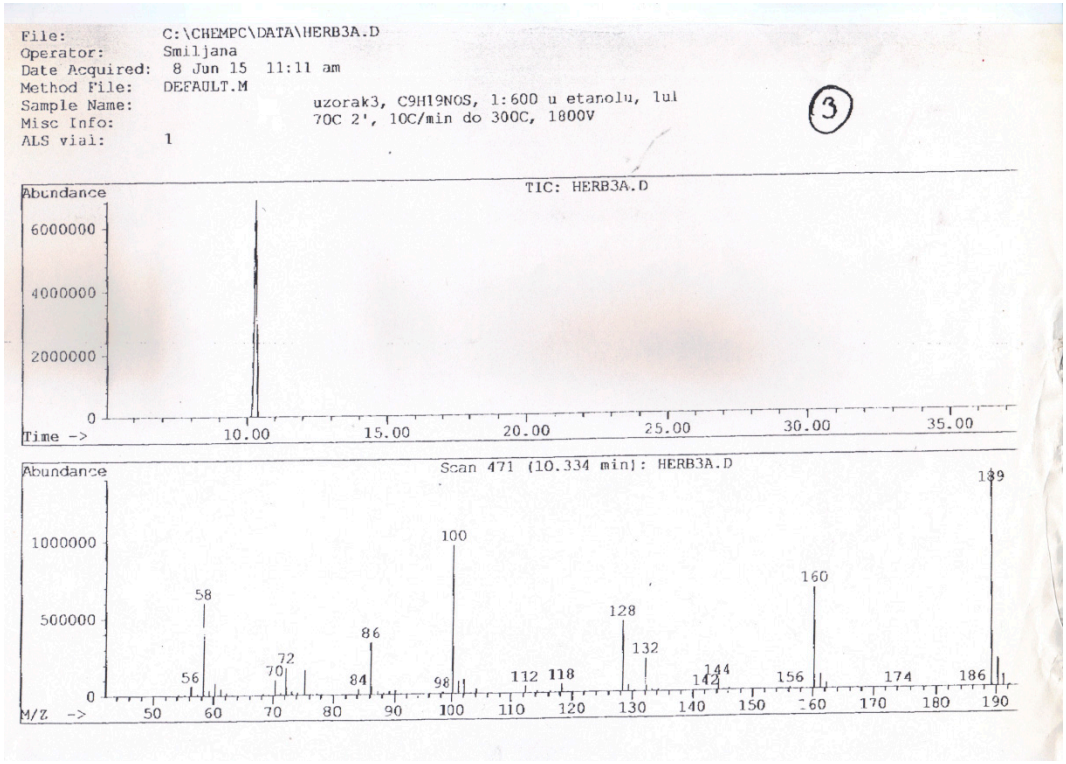


Figure S24. GC-MS chromatogram for N-butyl-O-isobutyl thiocarbamate.

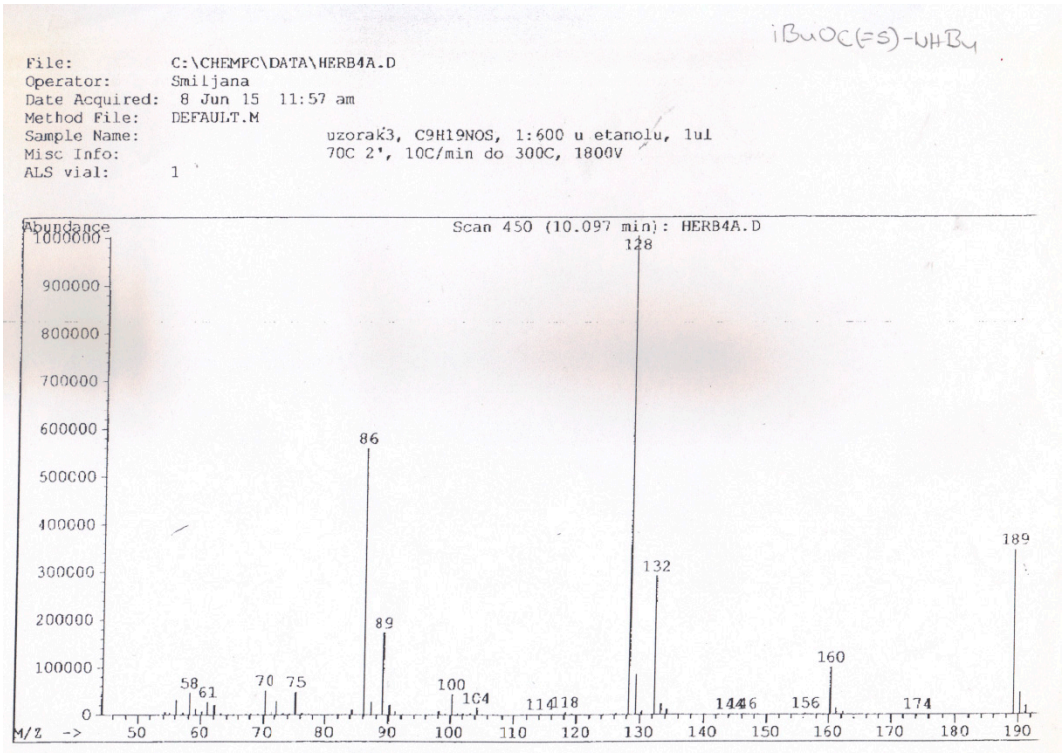


Figure S25. GC chromatogram for N-ethyl-O-isobutyl thiocarbamate (method B).

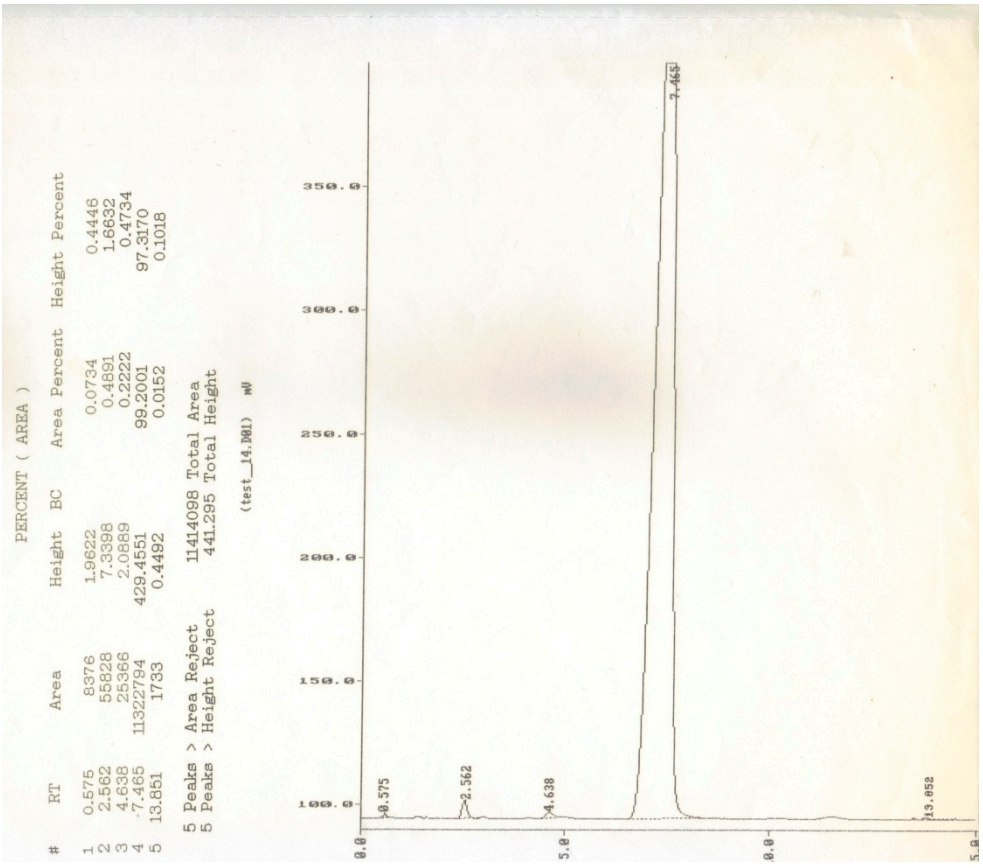


Figure S26. GC chromatogram for N-ethyl-O-isobutyl thiocarbamate (method A).

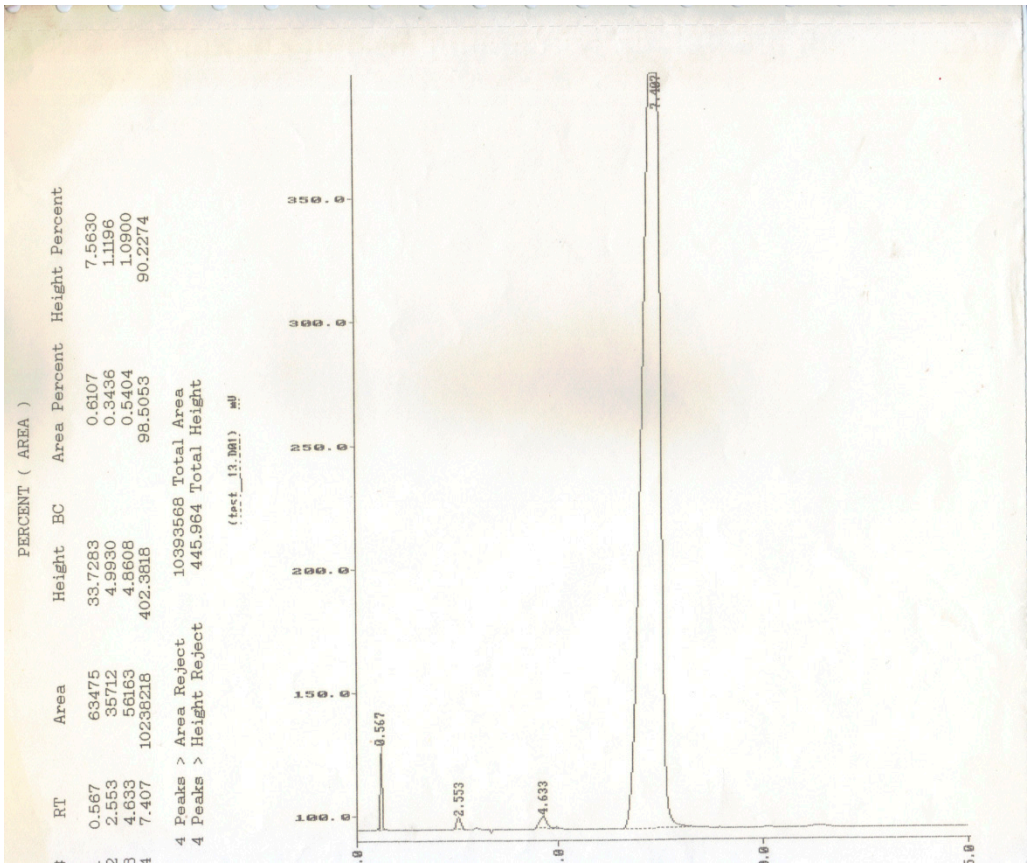


Figure S27. GC chromatogram for N-ethyl-O-isobutyl thiocarbamate (method C).