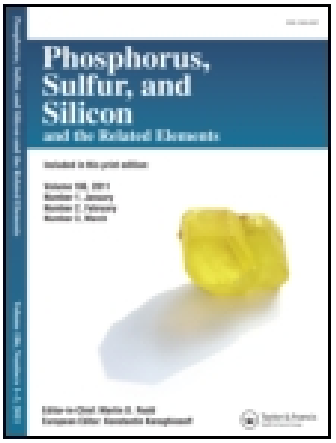

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A One-Pot Synthesis of Tetrahydro-2,5-dioxofuran-3-yl Alkylcarbamodithioates

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A ONE-POT SYNTHESIS OF TETRAHYDRO-2,5-DIOXOFURAN-3-YL ALKYLCARBAMODITHIOATES

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An efficient one-pot synthesis of tetrahydro-2,5-dioxofuran-3-yl alkylcarbamodithioates via three-component reaction of maleic anhydride, CS_2, and primary amines in good yields is described.

Keywords Carbamodithioate; maleic anhydride; one-pot synthesis; primary amine

INTRODUCTION

Organic carbamodithioates have received much attention due to their interesting chemistry and wide utility as radical precursors and intermediates in organic synthesis.[1–4] They also have a variety of applications in agriculture as pesticides, as well as in the rubber industry as vulcanization accelerators, and as antioxidants.[5–7] Because they have strong metal-binding capacity, they can act as inhibitors of enzymes and have a profound effect on biological systems.[8,9] Recently, a variety of reagents and catalysts including Mitsunobu's reagent,[10] solid $LiClO_4$/*N,N*-dimethylformamide,[11] dimethylsulfoxide,[12] and Cs_2CO_3/tetrabutylammonium iodide[13] have been used for the synthesis of carbamodithioates.

As part of our current studies on the development of new routes in approach to the synthesis of organosulfur compounds,[14–19] we describe an efficient synthesis of functionalized carbamodithioates containing a cyclic anhydride moiety. Similar reactions of amines, carbon disulfide, and maleimides with dithiocarbamic acids have been reported.[20]

RESULTS AND DISCUSSION

The reaction of primary amines (**1**), carbon disulfide, and maleic anhydride (**2**) in MeCN at room temperature produced tetrahydro-2,5-dioxofuran-3-yl alkylcarbamodithioates (**3**) in fairly good yields after purification (Scheme 1). Compounds **3a–3m** are stable, and they are recovered unchanged after reflux in toluene for 12 h. The structures of compounds **3a–3m** were determined by IR, ^{1}H NMR, and ^{13}C NMR spectral data.

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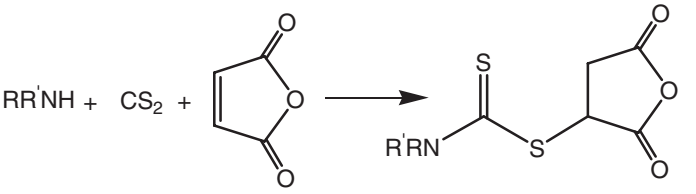


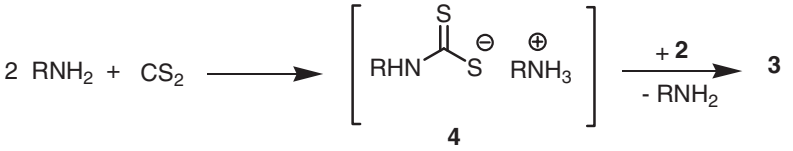
1,3	R	R'	Yield (%) of **3**
a	H	*i*-Pr	85
b	H	Et	87
c	H	*sec*-Bu	79
d	H	*n*-Bu	83
e	H	Cyclohexyl	92
f	H	$C_6H_5CH_2$	92
g	H	4-Cl-$C_6H_4CH_2$	60
h	H	4-Me-$C_6H_4CH_2$	89
i	H	4-MeO-$C_6H_4CH_2$	90
j	H	2-Furylmethyl	93
k	H	$CH_3CHOHCH_2$	93
l	H	Ph	11
m	Et	Et	20

Scheme 1 Formation of tetrahydro-2,5-dioxofuran-3-yl alkylcarbamodithioates (**3**).

The mass spectra of these compounds displayed molecular ion peaks at appropriate *m/z* values. The ^{1}H NMR spectra of **3a–3m** exhibited characteristic ABX systems for the CH_2-CH moieties. The proton-decoupled ^{13}C NMR spectra of **3a–3m** showed three distinct resonances for C=O and C=S groups. The reaction of aniline or diethylamine with CS_2 and maleic anhydride produced the corresponding carbamodithioate **3l** or **3m** in low yields.

Compounds **3c** and **3k** possess two stereogenic centers, and can exist as a mixture of two diastereoisomers. The NMR spectra of these compounds are consistent with the presence of two diastereoisomers (see the Experimental section). The methylene protons of the benzyl group in **3f–3i** are diastereotopic and exhibit characteristic AB systems.

A tentative mechanism for this transformation is proposed in Scheme 2. It is conceivable that the initial event is the formation of a xanthate salt **4** from two equivalents of the amine and CS_2. Nucleophilic attack of this salt on **2**, and subsequent elimination of RNH_2, leads to carbamodithioates **3**.



Scheme 2 A plausible mechanism for formation of carbamodithioates **3**.

In conclusion, we have described a convenient route to tetrahydro-2,5-dioxofuran-3-yl alkylcarbamodithioates from primary alkylamines, CS_2, and maleic anhydride. Mild conditions of dithiocarbonization, availability of the initial compounds, possibility of structural variations, and good preparative yields makes the developed synthesis of carbamodithioates a rather promising process.

EXPERIMENTAL

Maleic anhydride, CS_2, and amines **1** were obtained from Fluka and were used without further purification. Other procedural details are as follows: mp: Electrothermal-9100 apparatus; IR Spectra: Shimadzu IR-460 spectrometer. ^{1}H and ^{13}C NMR spectra: Bruker DRX-300 AVANCE instrument; in $CDCl_3$ at 300 and 75 MHz, respectively; δ in ppm, J in Hz; EI-MS (70 eV): Finnigan-MAT-8430 mass spectrometer, in m/z. Elemental analyses (C, H, N) were performed with a Heraeus CHN-O-Rapid analyzer.

General Procedure for the Preparation of Compounds 3

A mixture of amine **1** (2 mmol) in CS_2 (1.2 mL) was stirred for 1 h, then a solution of maleic anhydride (0.20 g, 2 mmol) in MeCN (2 mL) was added slowly. After completion of the reaction (1–2 h), as indicated by TLC (AcOEt/hexane, 2:1), the solvent was removed under reduced pressure, and the residue was purified by dry-flash chromatography using a 1:3 mixture of AcOEt/petroleum ether as eluent to afford pure dithiocarbamates **3**. Further purification was done by recrystallization from a mixture of $CHCl_3$/hexane.

Tetrahydro-2,5-dioxofuran-3-yl *iso*-propylcarbamodithioate (3a). Pale yellow crystals; yield: 0.44 g (85%); mp 164–165°C. IR (KBr) (ν_{max}/cm^{-1}): 3443, 1729, 1699, 1471, 1453, 1397, 1265, 1203. EI-MS: 233 (M^+, 9), 191 (12), 187 (11), 133 (15), 100 (100), 43 (18); Anal. Calcd. for $C_8H_{11}NO_3S_2$ (233.2): C, 41.19; H, 4.75; N, 6.01%. Found: C, 41.43; H, 4.81; N, 6.07%. ^{1}H NMR: δ = 1.43 (3H, d, ^3J 6.9, Me), 1.46 (3H, d, ^3J 6.9, Me), 3.08 (1H, dd, ^2J 18.0, ^3J 8.0, CH), 3.23 (1H, dd, ^2J 18.0, ^3J 3.9, CH), 3.65 (1H, br s, NH), 4.51 (1H, dd, ^3J 8.0, ^3J 3.9, CH), 5.23 (1H, sept, ^3J 6.9, CH). ^{13}C NMR: δ = 18.3 (Me), 18.6 (Me), 36.6 (CH_2), 43.9 (CH), 50.8 (CHN), 175.4 (C=O), 176.0 (C=O), 201.4 (C=S).

Tetrahydro-2,5-dioxofuran-3-yl ethylcarbamodithioate (3b). Pale yellow crystals; yield: 0.38 g (87%); mp 121–122°C. IR (KBr) (ν_{max}/cm^{-1}): 3423, 2569, 1718, 1708, 1436, 1328, 1250, 1121. EI-MS: 219 (M^+, 8), 191 (14), 173 (13), 133 (19), 86 (100), 29 (16); Anal. Calcd. for $C_7H_9NO_3S_2$ (219.2): C, 38.35; H, 4.14; N, 6.39%. Found: C, 38.62; H, 4.10; N, 6.48%. ^{1}H NMR: δ = 1.22 (3H, t, ^3J 7.2, Me), 3.1 (1H, dd, ^2J 18.1, ^3J 8.5, CH), 3.3 (1H, dd, ^2J 18.1, ^3J 3.6, CH), 4.07 (2H, q, ^3J 7.2, CH_2), 4.42 (1H, dd, ^3J 8.5, ^3J 3.6, CH), 10.6 (1H, br s, NH). ^{13}C NMR: δ = 12.2 (Me), 36.8 (CH_2), 40.4 (CH_2), 45.7 (CH_2N), 175.7 (C=O), 175.8 (C=O), 200.4 (C=S).

Tetrahydro-2,5-dioxofuran-3-yl *sec*-butylcarbamodithioate (3c). Pale yellow crystals; yield: 0.39 g (79%); mp 111–113°C. IR (KBr) (ν_{max}/cm^{-1}): 3435, 2568, 1727, 1717, 1460, 1387, 1284, 1115. EI-MS: 247 (M^+, 12), 201 (11), 191 (13), 133 (17), 114 (100), 57 (40); Anal. Calcd. for $C_9H_{13}NO_3S_2$ (247.3): C, 43.71; H, 5.30; N, 5.67%. Found: C, 43.52; H, 5.41; N, 5.70%. Major isomer (55%): ^{1}H NMR: δ = 0.89 (3H, t, ^3J 7.3, Me), 1.42 (3H, d, ^3J 6, Me), 1.83–1.85 (2H, m, CH_2), 3.07–3.10 (2H, m, CH_2), 4.28–4.30 (1H, m, CH), 5.06–5.08 (1H, m, CH). ^{13}C NMR: δ = 11.4 (Me), 16.6 (Me), 25.6 (CH_2), 36.6 (CH_2), 43.8 (CH), 56.6 (CH-N), 175.5 (C=O), 176.1 (C=O), 205.1 (C=S). Minor isomer

(45%): ^{1}H NMR: $\delta = 0.89$ (3H, t, ^3J 7.3, Me), 1.46 (3H, d, ^3J 6.0, Me), 2.13–2.15 (2H, m, CH_2), 3.24–3.26 (2H, m, CH_2), 4.28–4.30 (1H, m, CH), 5.06–5.08 (1H, m, CH). ^{13}C NMR: $\delta = 11.5$ (Me), 16.9 (Me), 25.7 (CH_2), 36,8 (CH_2), 43.9 (CH), 56.7 (CH-N), 175.5 (C=O), 176.1 (C=O), 205.1 (C=S).

Tetrahydro-2,5-dioxofuran-3-yl *n*-butylcarbamodithioate (3d). Pale yellow crystals; yield: 0.41g (83%); mp 139–140°C. IR (KBr) (ν_{max}/cm^{-1}): 3444, 2605, 1733, 1703, 1437, 1361, 1284, 1194. EI-MS: 247 (M^+, 7), 201 (12), 191 (15), 133 (18), 114 (100), 57 (25), 43 (18); Anal. Calcd. for $C_9H_{13}NO_3S_2$ (247.2): C, 43.71; H, 5.30; N, 5.67%. Found: C, 43.01; H, 5.42; N, 5.59%. ^{1}H NMR: $\delta = 0.95$ (3H, t, ^3J 7.2, Me), 1.36–1.38 (2H, m, CH_2), 1.61–1.63 (2H, m, CH_2), 3.07 (1H, dd, ^2J 18.0, ^3J 8.7, CH), 3.3 (1H, dd, ^2J 18.0, ^3J 3.9, CH), 4.00 (1H, t, ^3J 7.6, CH_2), 4.42 (1H, dd, ^3J 8.7, ^3J 3.9, CH), 10.98 (1H, br s, NH). ^{13}C NMR: $\delta = 14.0$ (Me), 20.4 (CH_2), 29.0 (CH_2), 36.9 (CH_2), 45.1 (CH), 45.6 (CH_2), 175.9 (C=O), 176.0 (C=O), 200.7 (C=S).

Tetrahydro-2,5-dioxofuran-3-yl cyclohexylcarbamodithioate (3e). Pale yellow crystals; yield: 0.50 g (92%); mp 186–187°C. IR (KBr) (ν_{max}/cm^{-1}): 3462, 2532, 1743, 1701, 1465, 1396, 1284, 1199. EI-MS: 273 (M^+, 9), 192 (100), 174 (36), 140 (17), 81 (17), 55 (34), 41 (25); Anal. Calcd. for $C_{11}H_{15}NO_3S_2$ (273.3): C, 48.34; H, 5.53; N, 5.13%. Found: C, 48.01; H, 5.65; N, 5.23%. ^{1}H NMR: $\delta = 1.20$–2.40 (10 H, m, 5 CH_2), 3.06 (1H, dd, ^2J 18.0, ^3J 8.3, CH), 3.23 (1H, dd, ^2J 18.0, ^3J 3.9, CH), 4.25 (1H, dd, ^3J 8.3, ^3J 3.9, CH), 4.8 (1H, br s, NH). ^{13}C NMR: $\delta = 25.4$ (CH_2), 26.3 (CH_2), 26.4 (CH_2), 27.7 (CH_2), 27.9 (CH_2), 36.6 (CH_2), 43.7 (CH), 58.8 (CHN), 176.2 (C=O), 176.9 (C=O), 201.7 (C=S).

Tetrahydro-2,5-dioxofuran-3-yl benzylcarbamodithioate (2f). Yellow crystals; yield: 0.49 g (92%); mp 139–140°C. IR (KBr) (ν_{max}/cm^{-1}): 3404, 2560, 1706, 1688, 1425, 1349, 1174, 1107. EI- MS: 281 (M^+, 11), 235 (7), 149 (21), 148 (70), 91 (100), 65 (18), 45 (8). Anal. Calcd. for $C_{12}H_{11}NO_3S_2$ (281.3): C, 51.23; H, 3.94; N, 4.98%. Found: C, 51.4; H, 4.11; N, 5.09%. ^{1}H NMR: $\delta = 3.06$ (1H, dd, ^2J 18, ^3J 8.0, CH), 3.32 (1H, dd, ^2J 18.0, ^3J 3.9, CH), 3.65 (1H, bs, NH), 4.46 (1H, dd, ^3J 8.0, ^3J 3.9, CH), 5.20 (2 H, ABq, $\nu\Delta_{AB} = 22$ Hz, $^2J_{AB}$ 14.2, CH_2), 7.30–7.42 (5H, m, CH-arom). ^{13}C NMR: δ 36.6 (CH_2), 45.7 (CH), 48.2 (CH_2N), 128.6 (CH), 129.0 (2CH), 129.3 (2CH), 134.9(C), 174.4 (C=O), 175.8 (C=O), 200.4 (C=S).

Tetrahydro-2,5-dioxofuran-3-yl 4-chlorobenzylcarbamodithioate (3g). Pale yellow crystals; yield: 0.38 g (60%); mp 139–140°C. IR (KBr) (ν_{max}/cm^{-1}): 3481, 2598, 1742, 1721, 1492, 1346, 1203, 1089. EI-MS: 317 (M^{+2}, 6), 315 (M^+, 20), 269 (8), 187 (11), 182 (68), 125 (100), 91 (10); Anal. Calcd. for $C_{12}H_{10}NO_3S_2Cl$ (315.7): C, 45.65; H, 3.51; N, 4.44%. Found: C, 45.23; H, 3.60; N, 4.52%. ^{1}H NMR: δ 3.10 (1H, dd, ^2J 18, ^3J 8.2, CH), 3.27 (1H, dd, ^2J 18.0, ^3J 3.9, CH), 4.44 (1H, dd, ^3J 8.2, ^3J 3.9, CH), 5.15 (2H, ABq, $\nu\Delta_{AB} = 7$ Hz, $^2J_{AB}$ 10.1, CH_2) 7.28 (d, ^3J 8.0, 2CH), 7.38 (2H, d, ^3J 8.0, 2CH), 10.17 (1H, br s, NH). ^{13}C NMR: $\delta = 36.5$ (CH_2), 45.7 (CH), 47.5 (CH_2N), 129.1 (2CH), 130.9 (2CH), 133.3 (C), 134.5 (C), 175.7 (C=O), 175.8 (C=O), 200.2 (C=S).

Tetrahydro-2,5-dioxofuran-3-yl 4-methylbenzylcarbamodithioate (3h). Pale yellow crystals; yield: 0.52 g (89%); mp 172–174°C. IR (KBr) (ν_{max}/cm^{-1}): 3481, 2590, 1747, 1720, 1492, 1346, 1272, 1196. EI-MS: 295 (M^+, 6), 249 (7), 162 (48), 105 (100), 91 (13); Anal. Calcd. for $C_{13}H_{13}NO_3S_2$ (295.3): C, 52.87; H, 4.44; N, 4.74%. Found: C, 52.95; H, 4.29; N, 4.87%. ^{1}H NMR: $\delta = 2.33$ (3 H, s, Me), 3.04 (1 H, dd, ^2J 18.0, ^3J 9.0, CH), 3.32 (1 H, dd, ^2J 18.0, ^3J 3.6, CH), 4.45 (1 H, dd, ^3J 9.0, ^3J 3.6, CH), 5.15 (2 H, ABq, $\nu\Delta_{AB} = 11$ Hz, $J_{AB} = 14.0$, CH_2), 7.12 (2 H, d, ^3J 7.8, 2 CH), 7.32 (2 H,

d, ^3J 7.8, 2 CH). ^{13}C NMR: $\delta = 21.6$ (Me), 36.7 (CH_2), 45.7 (CH), 48.0 (CH_2N), 129.3 (2 CH), 129.6 (2 CH), 132.0 (C), 138.4 (C), 174.8 (C=O), 175.8 (C=O), 200.4 (C=S).

Tetrahydro-2,5-dioxofuran-3-yl 4-methoxybenzylcarbamodithioate (3i). Pale yellow crystals; yield: 0.57 g (90%); mp 130–131°C. IR (KBr) (ν_{max}/cm^{-1}): 3370, 2560, 1721, 1712, 1428, 1300, 1252, 1193. EI-MS: 311 (M^+, 9), 265 (4), 178 (34), 121 (100), 91 (9.5), 77 (13.5); Anal. Calcd. for $C_{13}H_{13}NO_4S_2$ (311.3): C, 50.15; H, 4.21; N, 5.16%. Found: C, 49.85; H, 4.30; N, 5.23%. ^{1}H NMR: δ 3.03 (1 H, dd, ^2J 18.0, ^3J 8.7, CH), 3.27 (1 H, dd, ^2J 18.0, ^3J 3.6, CH), 3.8 (3 H, s, Me), 4.43 (1 H, dd, ^3J 8.7, ^3J 3.6, CH), 5.14 (2 H, ABq, $\nu\Delta_{AB} = 16.0$ Hz, J_{AB} 14.2, CH_2), 6.84 (2 H, d, ^3J 6.2, 2 CH), 7.4 (2 H, d, ^3J 6.2, 2 CH). ^{13}C NMR: δ 36.7 (CH_2), 45.8 (CH), 47.7 (CH_2), 55.6 (Me), 114.3 (2CH), 127.2 (CH), 131.0 (2CH), 159.8 (*C*-OMe), 175.0 (C=O), 175.8 (C=O), 200.5 (C=S).

Tetrahydro-2,5-dioxofuran-3-yl (furan-2-yl)methylcarbamodithioate (3j). Pale yellow crystals; yield: 0.46 g (93%); mp 105–106°C. IR (KBr) (ν_{max}/cm^{-1}): 3449, 2549, 1726, 1704, 1411, 1331, 1243, 1103. EI-MS: 271 (M^+, 9), 25 (9), 187 (11), 139 (78), 81 (100), 45 (8), 29 (18); Anal. Calcd. for $C_{10}H_9NO_4S_2$ (271.3): C, 44.27; H, 3.34; N, 5.16%. Found: C, 44.54; H, 3.42; N, 5.22%. ^{1}H NMR: $\delta = 3.07$ (1 H, dd, ^2J 18.0, ^3J 9.0, CH), 3.34 (1 H, dd, ^2J 18.0, ^3J 3.8, CH), 4.47 (1 H, dd, ^3J 9.0, ^3J 3.8, CH), 5.20 (2 H, ABq, $\nu\Delta_{AB} = 8$ Hz, J_{AB} 14.2, CH_2), 6.32–7.35 (3 H, m, CH-furyl), 7.28 (1 H, br s, NH). ^{13}C NMR: δ 36.7 (CH_2), 41.0 (CH), 45.7 (CH_2), 110.8 (CH), 110.9 (CH), 143.0 (CH), 147.9 (C), 175.3 (C=O), 175.4 (C=O), 199.8 (C=S).

Tetrahydro-2,5-dioxofuran-3-yl 2-hydroxypropylcarbamodithioate (3k). Pale yellow crystals; yield: 0.46 g (93%); mp 157–159°C. IR (KBr) (ν_{max}/cm^{-1}): 3436, 3380, 2349, 1735, 1704, 1456, 1368, 1262, 1208, 1131. EI-MS: 249 (M^+, 9), 231 (45), 203(8), 191 (10), 133 (17), 116 (100), 45 (28); Anal. Calcd. for $C_8H_{11}NO_4S_2$ (249.2): C, 38.55; H, 4.45; N, 5.62%. Found: C, 38.63; H, 4.52; N, 5.74%. Major isomer (80%): ^{1}H NMR: $\delta = 1.20$ (3 H, d, ^3J 6.0, Me), 3.15 (1 H, dd, ^2J 19, ^3J 6.3, CH), 3.27 (1 H, dd, ^2J 19.0, ^3J 3.9, CH), 3.34 (1 H, br s, OH), 3.77–3.80 (2 H, m, CH_2-N), 4.23–4.25 (1 H, m, CH-O), 4.60 (1 H, dd, ^3J 6.3, ^3J 3.9, CH). ^{13}C NMR: $\delta = 21.7$ (Me), 36.5 (CH_2), 46.8 (CH), 60.0 (CH_2), 63.4 (CH), 172.5 (C=O), 177.0 (C=O), 200.5 (C=S). Minor isomer (20%): ^{1}H NMR: $\delta = 1.16$ (3 H, d, ^3J 6.0, Me), 3.13 (1 H, dd, ^2J 19.0, ^3J 6.3, CH), 3.26 (1 H, dd, ^2J 19.0, ^3J 3.9, CH), 3.34 (1 H, br-s, OH), 3.86–3.90 (2 H, m, CH_2-N), 4.21–4.23 (1 H, m, CH-O), 4.55 (1 H, dd, ^3J 6.3, ^3J 3.9, CH), 4.87 (1 H, br s, NH). ^{13}C NMR: $\delta = 21.6$ (Me), 36.3 (CH_2), 46.7 (CH), 51.9 (CH_2), 63.3 (CH), 172.4 (C=O), 176.9 (C=O), 200.1 (C=S).

Tetrahydro-2,5-dioxofuran-3-yl phenylcarbamodithioate (3l). Pale Yellow oil; yield: 0.06 g (11%); IR (KBr) (ν_{max}/cm^{-1}): 3400, 2535, 1710, 1660, 1425, 1347, 1160. EI- MS: 268 (M^{+1}, 10), 267 (M^+, 8), 221 (9), 134 (100), 92 (23), 65 (16), 45 (8). Anal. Calcd. for $C_{11}H_9NO_3S_2$ (267.3): C, 49.43; H, 3.39; N, 5.24%. Found: C, 50.02; H, 4.04; N, 5.30%. ^{1}H NMR: $\delta = 3.32$ (1 H, dd, ^2J 8.0, ^3J 3.5, CH), 3.52 (1 H, br s, NH), 4.24 (1 H, dd, ^3J 5.5, ^3J 3.5, CH), 4.64 (1 H, dd, ^2J 8.0, ^3J 5.5, CH), 7.22–7.54 (5 H, m, CH-arom). ^{13}C NMR: δ 37.8 (CH_2), 43.5 (CH), 125.2 (CH), 129.5 (2CH), 129.8 (2CH), 135.0 (C), 173.4 (C=O), 175.9 (C=O), 200.1 (C=S).

Tetrahydro-2,5-dioxofuran-3-yl diethylcarbamodithioate (3m). Pale yellow crystals; yield: 0.10 g (20%); mp 118–119°C. IR (KBr) (ν_{max}/cm^{-1}): 3423, 2565, 1718, 1705, 1430, 1321, 1120. EI-MS: 247 (M^+, 7), 219 (24), 190 (100), 176 (13), 116 (23), 29 (16); Anal. Calcd. for $C_9H_{13}NO_3S_2$ (247.3): C, 43.72; H, 5.29; N, 5.67%. Found: C, 42.90; H, 5.20; N, 5.72%. ^{1}H NMR: $\delta = 1.19$ (3 H, t, ^3J 7.2, Me), 1.24 (3 H, t, ^3J 7.2,

Me), 3.15 (1 H, dd, ^2J 18.0, ^3J 8.4, CH), 3.37 (1H, dd, ^2J 18.0, ^3J 3.6, CH), 3.77 (2 H, q, ^3J 7.2, CH_2), 4.02 (2 H, q, ^3J 7.2, CH_2), 5.25 (1 H, dd, ^3J 8.4, ^3J 3.6, CH). ^{13}C NMR: δ = 13.2 (Me), 14.4 (Me) 36.4 (CH_2), 41.7 (CH_2), 43.2 (CH_2), 51.3 (CH), 171.3 (C=O), 171.4 (C=O), 193.8 (C=S).

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