

Facile three-component synthesis of dithiocarbamate derivatives with potent antimicrobial activity

Mohamed S. Behalo* and Aly A. Aly

Chemistry Department, Faculty of Science, Benha University, PO Box 13518 Egypt

A one-pot three-component synthesis of 3-oxo-3-(phenoxathiin-2-yl)-1-phenyl/(4-chlorophenyl)propyl alkyl/aryl-dithiocarbamate was achieved from the reaction of 3-phenyl/(4-chlorophenyl)-1-(phenoxathiin-2-yl)propenones, amine and carbon disulfide. The antimicrobial activities of some compounds were also screened against some selected bacteria and fungi.

Keywords: dithiocarbamates, phenoxathiin, antimicrobial activity

The chemistry of dithiocarbamates has attracted the interest of many researchers in the last years. There are few general approaches involving amines, carbon disulfide and alkyl halide, epoxide or Michael acceptors to afford dithiocarbamates^{1–6} which have demonstrated a broad spectrum of applications including agrochemical, medicinal, and rubber industry.^{7–10}

On the other hand, phenoxathiin derivatives were found to be active as potential antihypertensive,¹¹ antimicrobial,^{12,13} antitumour¹⁴ and anti-inflammatory agents¹⁵ and have promising fluorescent properties.^{16,17} Several phenoxathiin pyridinium derivatives were prepared and acted as effective antifungal agents against *Aspergillus* and *Candida*.¹⁸

In previous work, we reported three components synthesis of dithiocarbamates and heterocycles from azoalkene systems, carbon disulfide and primary amine.¹⁹ In our ongoing research programme aimed at the synthesis of biologically active compounds,^{20–22} we now report one pot synthesis of dithiocarbamates having bulky phenoxathiin moiety substituted at position-2 utilising carbon disulfide, amine and phenoxathiinylchalcones as Michael acceptors in order to enhance their biological and pharmacological activities.

Results and discussion

The starting chalcones, 3-phenyl/(4-chlorophenyl)-1-(phenoxathiin-2-yl)propenones (**1a,b**) were prepared by the

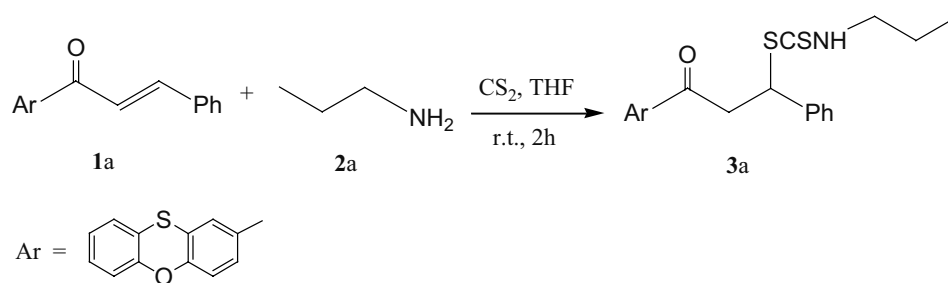
reaction of 2-acetylphenoxathiin and benzaldehyde and 4-chlorobenzaldehyde.²³

The reaction of 3-phenyl-1-(phenoxathiin-2-yl)propenone (**1a**) (1 mmole), carbon disulfide (3 mmole) and propylamine (**2a**) (1.5 mmole) in THF at room temperature afforded 3-oxo-3-(phenoxathiin-2-yl)-1-phenylpropyl propyldithiocarbamate (**3a**) in good yield, Scheme 1. The structure of compound **3a** was assigned on the basis of the elemental analysis and spectroscopic data (¹H NMR, ¹³C NMR, MS and IR).

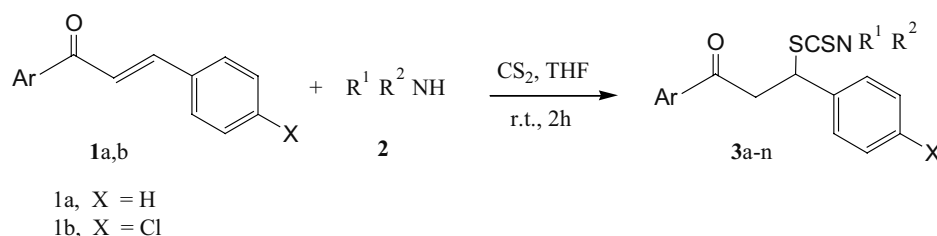
From these results and in order to generalise this three components synthesis, chalcones **1a,b** were allowed to react with different amines viz. benzylamine, cyclohexylamine, 1-amino-2-acetaldahyde diethylacetal, diethylamine and carbon disulfide under the same reaction conditions, it was found that the reaction afforded the corresponding dithiocarbamate derivatives **3a–n**, Scheme 2. The spectral data and the elemental analysis of the products are all consistent with the assigned structures.

The reaction was also carried out in different organic solvents such as ethanol, acetonitrile, dimethylformamide, diethyl ether and finally solvent free condition. In these solvents, the reaction proceeded from low to high yield to give the corresponding dithiocarbamates.

On the other hand, aromatic amines such as aniline and *p*-tolidine did not participate well in the reaction due to isolation of low yield.



Scheme 1



Scheme 2

* Correspondent. E-mail: mohamedbehalo@hotmail.com

Conclusion

The objectives of the present study were to synthesise and investigate the biological activities of new phenoxathiin-containing compounds. This aim has been verified by an efficient three components synthesis of dithiocarbamate derivatives **3a-n** at room temperature from the reaction of phenoxathiinylchalcones **1a,b**, carbon disulfide and amines.

Biological activities

Some of the newly synthesised target compounds were evaluated *in vitro* for their antibacterial activity against *Bacillus subtilis*, *Rhodococcus equii* as Gram positive bacteria and *Pseudomonas aeruginosa* and *Escherichia coli* as Gram negative bacteria. They were also evaluated *in vitro* for their antifungal potential against *Candida albican* and *Fusarium solami*. Agar diffusion method was used for the determination of the preliminary antibacterial and antifungal activity.²⁴ Ampicillin trihydrate and terbinafene were used as a reference drugs.

The results were recorded for each tested compound as the average diameter of inhibition zones (r) of bacterial or fungal growth around the disks in mm at 100 µg concentration using dimethylsulfoxide (10 ml) as a solvent.

The results depicted in Table 2 revealed that most of the tested compounds displayed variable inhibitory effect on the growth of the tested organisms. Regarding the activity of the dithiocarbamate series against both Gram positive and Gram negative bacteria, the results showed that compounds **3c**, **3f** and **3h** have a significant antibacterial effect. Compounds **3c** and **3i** showed the highest activity against *Bacillus Subtilis* while **3a** showed the lowest. On the other hand, the tested compounds have no effect against *Fusarium solami* except compounds **3c** and **3j**, they showed antifungal activity similar to the reference drug Terbinafene.

Experimental

All reactions were monitored by TLC using Merck Platen Kieselgel 60 F 25G. Chromatographic purification were performed on columns packed with Merck silica gel 60, melting points are uncorrected. IR spectra in KBr were recorded using a Perkin-Elmer 298 spectrophotometer. Mass spectra were obtained using a Shimadzu GCMS-QP 1000 EX mass spectrometer. ¹H and ¹³C NMR spectra were obtained using 400 MHz and 100 MHz respectively in the institute of organic chemistry, faculty of science and technology, university of Urbino, Urbino. Italy. Microanalytical data was carried out in the microanalytical centre, university of Cairo, Egypt.

General procedure for the synthesis of dithiocarbamates **3a-n**

To stirred solution of amine **2** (1.5 mmole) in THF (20 mL), CS₂ (3 mmole) was added. The chalcone **1** (1 mmole) was added and the reaction mixture was stirred at room temperature for 2 h. Excess carbon disulfide was removed and the crude product was purified by column chromatography on silica gel and ethyl acetate/cyclohexane as eluent.

3-Oxo-3-(phenoxathiin-2-yl)-1-phenylpropylpropyldithiocarbamate (3a): ¹H NMR (DMSO-*d*₆), δ ppm = 1.07–1.11 (t, 3H, CH₃), 1.95–2.11 (m, 2H, CH₂), 3.32–3.37 (t, 2H, CH₂N), 3.46–3.49 (d, 2H, CH₂CO), 4.85–4.93 (dd, 1H, CHS), 6.97–7.53 (m, 12H, ArH), 12.60 (s, 1H, NH, exchangeable); ¹³C NMR: 14.03(CH₃), 22.17 (CH₂), 39.92 (CHS), 47.56 (CH₂CO), 52.41 (CH₂N), 117.18, 117.42, 117.94, 124.89, 126.58, 126.65, 127.18, 128.22, 128.95, 130.13, 131.13, 135.17, 151.91, 156.87 (phenoxathiin and phenyl carbons), 191.10 (CO), 199.13 (CS); IR, 1680 (CO), 3187 (NH), 2923, 2854 (CH₂), 1218 (CS) cm⁻¹; MS: *m/z*: 465 (M⁺); Anal. Calcd for C₂₅H₂₃NO₂S₃ (465.65): C, 64.49; H, 4.98; N, 3.01. Found: C, 64.66; H, 5.12; N, 3.21%.

3-Oxo-3-(phenoxathiin-2-yl)-1-phenylpropylcyclohexyldithiocarbamate (3b): ¹H NMR (DMSO-*d*₆), δ ppm = 1.39–1.67 (m, 6H, 3CH₂), 1.98–2.29 (m, 4H, 2CH₂), 3.79–3.84 (m, 1H, CHN), 4.19–4.28 (d, 2H, CH₂CO), 5.06 (dd, 1H, CHS), 6.78–8.05 (m, 12H, ArH), 11.10 (s, 1H, NH, exchangeable); ¹³C NMR: 22.17, 28.86, 32.27 (5CH₂), 39.78 (CHS), 42.72 (CH₂CO), 52.28 (CHN), 117.60, 118.28, 125.15, 125.94, 126.35, 126.95, 127.63, 128.32, 129.25, 131.54, 133.52, 144.93, 150.98, 159.08 (phenoxathiin and phenyl carbons), 193.42 (CO), 196.22 (CS); IR, 1681 (CO), 3350 (NH), 2925, 2850 (CH₂),

Table 1 Reaction of chalcones **1a,b** with carbon disulfide and amines

Entry	Chalcone 1	Amine, R ¹ R ² NH 2	Product 3	M.p./°C	Yield/%
1	1a	Propylamine	a	182 – 184	85
2		Cyclohexylamine	b	177 – 179	91
3		Diethylamine	c	196 – 198	88
4		1-Amino-2-acetaldahyde diethylacetal	d	206 – 208	68
5		Benzylamine	e	212 – 214	83
6		Aniline	f	194 – 196	45
7		<i>p</i> -Tolidine	g	202 – 204	49
8	1b	Propylamine	h	195 – 197	82
9		Cyclohexylamine	i	186 – 188	83
10		Diethylamine	j	214 – 216	82
11		1-Amino-2-acetaldahyde diethylacetal	k	183 – 185	75
12		Benzylamine	l	206 – 208	74
13		Aniline	m	180 – 182	39
14		<i>p</i> -Tolidine	n	191 – 193	46

Table 2 Responses of various micro-organisms to some synthesised compounds

Compound	<i>B. subtilis</i>	<i>R. equii</i>	<i>P. aeruginosa</i>	<i>E. Coli</i>	<i>C. albican</i>	<i>F. solami</i>
3a	10	16	0	0	14	0
3b	16	18	15	15	12	0
3c	30	28	20	18	14	8
3d	18	0	0	0	0	0
3e	20	16	0	0	8	0
3f	20	20	16	28	10	0
3h	12	18	15	26	8	0
3i	28	0	0	18	8	0
3j	22	0	0	0	6	12
Ampicillin trihydrate	30	30	30	30	NT	NT
Terbinafene	NT	NT	NT	NT	11	11

Numbers in the table represent the inhibition zone diameter (r mm) of either fungal growth or bacterial cells for each compound; r > 20 mm, high active; r > 12 mm, moderately active; r > 6 mm, slightly active; 0 no inhibition was observed; and NT means not tested.

1216 (CS) cm^{-1} ; MS: m/z : 505 (M^+); Anal. Calcd for $\text{C}_{28}\text{H}_{27}\text{NO}_2\text{S}_3$ (505.71): C, 66.50; H, 5.38; N, 2.77. Found: C, 66.82; H, 5.47; N, 2.68%.

3-Oxo-3-(phenoxathiin-2-yl)-1-phenylpropyldithiocarbamate (3c): ^1H NMR ($\text{DMSO}-d_6$), δ ppm = 1.13–1.24 (2t, 6H, 2CH_3), 3.66–3.69 (d, 2H, CH_2CO), 4.01–4.17 (2q, 4H, 2CH_2), 5.82 (dd, 1H, CHS), 6.72–7.98 (m, 12H, ArH), 10.47 (s, 1H, NH, exchangeable); ^{13}C NMR 11.25 (2CH_3), 45.68 (CHS), 47.16 (2CH_2), 47.93 (CH_2CO), 117.94, 118.11, 118.80, 121.56, 122.37, 122.85, 125.13, 128.28, 128.49, 131.12, 134.22, 136.18, 154.76, 156.87 (phenoxathiin and phenyl carbons), 191.22 (CO), 195.23 (CS); IR, 1704 (CO), 3378 (NH), 2980, 2844 (CH_2), 1232 (CS) cm^{-1} ; MS: m/z : 479 (M^+); Anal. Calcd for $\text{C}_{26}\text{H}_{25}\text{NO}_2\text{S}_3$ (479.68): C, 65.10; H, 5.25; N, 2.92. Found: C, 65.27; H, 5.39; N, 2.88%.

3-Oxo-3-(phenoxathiin-2-yl)-1-phenylpropyl(2,2-diethoxyethyl)dithiocarbamate (3d): ^1H NMR ($\text{DMSO}-d_6$), δ ppm = 1.24–1.35 (2t, 6H, 2CH_3), 2.25–2.26 (d, 2H, CH_2N), 4.05 (d, 2H, CH_2CO), 4.07 (dd, 1H, CHS), 4.15–4.31 (2q, 4H, 2CH_2), 5.06–5.10 (m, 1H, OCH), 7.20–7.95 (m, 12H, ArH), 8.75 (s, 1H, NH, exchangeable); IR, 1688 (NH), 3345 (NH), 2935, 2820 (CH_2), 1315 (CS) cm^{-1} ; MS: m/z : 539 (M^+); Anal. Calcd for $\text{C}_{28}\text{H}_{29}\text{NO}_4\text{S}_3$ (539.72): C, 62.31; H, 5.42; N, 2.60. Found: C, 62.45; H, 5.53; N, 2.49%.

3-Oxo-3-(phenoxathiin-2-yl)-1-phenylpropylbenzylidithiocarbamate (3e): ^1H NMR ($\text{DMSO}-d_6$), δ ppm = 3.72 (s, 2H, CH_2), 3.78–3.82 (d, 2H, CH_2CO), 4.67–4.71 (m, 1H, CH), 6.93–7.88 (m, 17H, ArH), 9.38 (s, 1H, NH, exchangeable); IR, 1685 (CO), 3335 (NH), 2945, 2830 (CH_2), 1240 (CS) cm^{-1} ; MS: m/z : 513 (M^+); Anal. Calcd for $\text{C}_{29}\text{H}_{23}\text{NO}_2\text{S}_3$ (513.68): C, 67.81; H, 4.51; N, 2.73. Found: C, 68.11; H, 4.72; N, 2.63%.

3-Oxo-3-(phenoxathiin-2-yl)-1-phenylpropylphenyldithiocarbamate (3f): ^1H NMR (CDCl_3), δ ppm = 3.35–3.38 (d, 2H, CH_2), 4.65–4.69 (m, 1H, CH), 7.01–7.98 (m, 17H, ArH), 10.22 (s, 1H, NH, exchangeable); IR, 1684 (CO), 2930, 2845 (CH_2), 1252 (CS); Anal. Calcd for $\text{C}_{28}\text{H}_{21}\text{NO}_2\text{S}_3$ (499.67): C, 67.30; H, 4.24; N, 2.80. Found: C, 67.13; H, 4.15; N, 2.72%.

3-Oxo-3-(phenoxathiin-2-yl)-1-phenylpropylp-tolyldithiocarbamate (3g): ^1H NMR ($\text{DMSO}-d_6$), δ ppm = 2.25 (s, 3H, CH_3), 4.12 (d, 2H, CH_2), 4.85–4.89 (m, 1H, CH), 6.92–7.50 (m, 16H, ArH), 10.98 (s, 1H, NH, exchangeable); ^{13}C NMR 23.24 (CH_3), 48.31 (CHS), 51.23 ($\text{CH}_2\text{C}=\text{O}$), 115.20, 118.24, 118.66, 120.12, 121.36, 121.42, 123.56, 125.68, 128.41, 128.54, 129.14, 131.26, 133.53, 133.76, 135.64, 144.58, 152.38, 154.82 (phenoxathiin and phenyl carbons), 192.46 (CO), 196.28 (CS); IR, 1690 (CO), 3364 (NH), 2930, 2825 (CH_2), 1282 (CS) cm^{-1} ; MS: m/z : 513 (M^+); Anal. Calcd for $\text{C}_{29}\text{H}_{23}\text{NO}_2\text{S}_3$ (513.68): C, 67.81; H, 4.51; N, 2.73. Found: C, 68.05; H, 4.75; N, 2.67%.

1-(4-Chlorophenyl)-3-oxo-3-(phenoxathiin-2-yl)propyl propyldithiocarbamate (3h): ^1H NMR ($\text{DMSO}-d_6$), δ ppm = 1.12–1.15 (t, 3H, CH_3), 2.02–2.10 (m, 2H, CH_2), 3.25–3.28 (t, 2H, CH_2N), 3.41–3.45 (d, 2H, CH_2CO), 5.12–5.18 (dd, 1H, CH), 7.07–7.98 (m, 11H, ArH), 11.35 (s, 1H, NH, exchangeable); IR, 1695 (CO), 3325 (NH), 2933, 2840 (CH_2), 1310 (CS) cm^{-1} ; Anal. Calcd for $\text{C}_{25}\text{H}_{22}\text{NO}_2\text{S}_3\text{Cl}$ (500.10): C, 60.04; H, 4.43; N, 2.80. Found: C, 59.82; H, 4.30; N, 2.86%.

1-(4-Chlorophenyl)-3-oxo-3-(phenoxathiin-2-yl)propyl cyclohexyldithiocarbamate (3i): ^1H NMR ($\text{DMSO}-d_6$), δ ppm = 1.65–1.95 (m, 6H, 3CH_2), 2.40–2.48 (m, 4H, 2CH_2), 3.07–3.09 (d, 2H, CH_2CO), 3.76–3.79 (m, 1H, CHN), 5.38–5.48 (dd, 1H, CHS), 7.08–8.01 (m, 11H, ArH), 11.55 (s, 1H, NH, exchangeable); ^{13}C NMR 21.95, 29.24, 31.78 (5CH_2), 39.92 (CH-S), 44.16 ($\text{CH}_2\text{C}=\text{O}$), 50.17 (CHN), 118.21, 118.54, 123.76, 125.62, 126.57, 127.16, 127.65, 128.77, 130.48, 131.63, 132.76, 142.83, 152.15, 157.94 (phenoxathiin and phenyl carbons), 188.67 (CO), 193.84 (CS); IR, 1680 (CO), 3400 (NH), 2920, 2850 (CH_2), 1250 (CS) cm^{-1} ; Anal. Calcd for $\text{C}_{28}\text{H}_{26}\text{NO}_2\text{S}_3\text{Cl}$ (540.16): C, 62.26; H, 4.85; N, 2.59. Found: C, 62.45; H, 4.97; N, 2.66%.

1-(4-Chlorophenyl)-3-oxo-3-(phenoxathiin-2-yl)propyldiethyldithiocarbamate (3j): ^1H NMR ($\text{DMSO}-d_6$), δ ppm = 1.09–1.14 (2t, 6H, 2CH_3), 3.45–3.49 (d, 2H, $\text{CH}_2\text{C}=\text{O}$), 4.22–4.29 (2q, 4H, 2CH_2), 4.91 (m, 1H, CHS), 6.94–8.01 (m, 11H, ArH), 9.58 (s, 1H, NH, exchangeable); IR, 1687 (CO), 2930, 2835 (CH_2), 1245 (CS) cm^{-1} ; Anal. Calcd for $\text{C}_{26}\text{H}_{24}\text{NO}_2\text{S}_3\text{Cl}$ (514.12): C, 60.74; H, 4.71; N, 2.72. Found: C, 60.94; H, 4.88; N, 2.78%.

1-(4-Chlorophenyl)-3-oxo-3-(phenoxathiin-2-yl)propyl (2,2-diethoxyethyl)dithiocarbamate (3k): ^1H NMR (CDCl_3), δ ppm = 1.15–1.28 (2t, 6H, 2CH_3), 2.18–2.22 (d, 2H, CH_2N), 3.84–3.88 (d, 2H, CH_2CO),

4.12 (t, 1H, CHS), 4.14–4.27 (2q, 4H, 2CH_2), 5.11–5.16 (m, 1H, OCH), 6.92–8.06 (m, 11H, ArH), 9.84 (s, 1H, NH, exchangeable); IR, 1692 (CO), 3270 (NH), 2936, 2845 (CH_2), 1260 (CS) cm^{-1} ; Anal. Calcd for $\text{C}_{28}\text{H}_{28}\text{ClNO}_4\text{S}_3$ (574.18): C, 58.57; H, 4.92; N, 2.44. Found: C, 58.42; H, 4.80; N, 2.48%.

1-(4-Chlorophenyl)-3-oxo-3-(phenoxathiin-2-yl)propylbenzylidithiocarbamate (3l): ^1H NMR ($\text{DMSO}-d_6$), δ ppm = 3.46 (s, 2H, CH_2), 3.85–3.89 (d, 2H, CH_2CO), 5.30–5.35 (m, 1H, CH), 6.77–7.92 (m, 16H, ArH), 9.85 (s, 1H, NH, exchangeable); IR, 1700 (CO), 2938, 2840 (CH_2), 1270 (CS) cm^{-1} ; Anal. Calcd for $\text{C}_{29}\text{H}_{22}\text{NO}_2\text{S}_3\text{Cl}$ (548.14): C, 63.55; H, 4.05; N, 2.56. Found: C, 63.72; H, 4.13; N, 2.63%.

1-(4-Chlorophenyl)-3-oxo-3-(phenoxathiin-2-yl)propylphenyldithiocarbamate (3m): ^1H NMR ($\text{DMSO}-d_6$), δ ppm = 3.23–3.27 (d, 2H, CH_2), 4.82–4.87 (m, 1H, CH), 6.97–8.01 (m, 16H, ArH), 9.23 (s, 1H, NH, exchangeable); IR, 1680 (CO), 2945, 2825 (CH_2), 1312 (CS); Anal. Calcd for $\text{C}_{28}\text{H}_{20}\text{NO}_2\text{S}_3\text{Cl}$ (534.11): C, 62.96; H, 3.77; N, 2.62. Found: C, 63.11; H, 3.85; N, 2.68%.

1-(4-chlorophenyl)-3-oxo-3-(phenoxathiin-2-yl)propyl-p-tolyldithiocarbamate (3n): ^1H NMR ($\text{DMSO}-d_6$), δ ppm = 2.38 (s, 3H, CH_3), 4.32–4.36 (d, 2H, CH_2), 5.13–5.518 (m, 1H, CH), 6.79–7.95 (m, 15H, ArH), 9.87 (s, 1H, NH, exchangeable); IR, 1705 (CO), 3385 (NH), 2925, 2838 (CH_2), 1310 (CS) cm^{-1} ; Anal. Calcd for $\text{C}_{29}\text{H}_{22}\text{NO}_2\text{S}_3\text{Cl}$ (548.14): C, 63.54; H, 4.05; N, 2.56. Found: C, 63.68; H, 4.12; N, 2.62%.

All strains were kindly supplied from Botany Department, Faculty of Science, Benha University, Egypt.

Received 2 August 2009; accepted 24 November 2009

Paper 09/0719 doi: 10.3184/030823409X12594282884215

Published online: 8 December 2009

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