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Synthesis and Fungicidal Activity of Hydrazone Derivatives Containing 2(3*H*)-Benzothiazolone Moiety

JIAN-QUAN WENG*, HUA HUANG, ZHI-QIN YU, HONG-MIN ZHANG, CHENG-XIA TAN and XING-HAI LIU

College of Chemical Engineering and Materials Science, Zhejiang University of Technology, Hangzhou 310014, Zhejiang, P.R. China

*Corresponding author: Tel: +571 88320147; E-mail: jqweng@zjut.edu.cn

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Fifteen novel hydrazone derivatives containing 2(3*H*)-benzothiazolone moiety were synthesized by condensing 2-oxobenzothiazolin-3-ylacetohydrazide with substituted ketones. Their structures were characterized by ^{1}H NMR, FTIR, MS and elemental analysis. The biological activities of the hydrazone derivatives were investigated. The bioassay results indicated that some of these compounds exhibit excellent fungicidal activities.

Key Words: Hydrazone, 2(3*H*)-Benzothiazolone, Synthesis, Fungicidal activity.

INTRODUCTION

Sulfur and nitrogen linked heterocyclic compounds have recently attracted considerable attention owing to their prominent biological activity[1-4]. As one of the potent heterocyclic compounds, the benzothiazole derivatives played a good role in the field of novel agrochemicals because of their wide biological activity, such as insecticidal[5], fungicidal[6], antiviral[7], herbicidal[8] and plant-growth-regulating[9] activities.

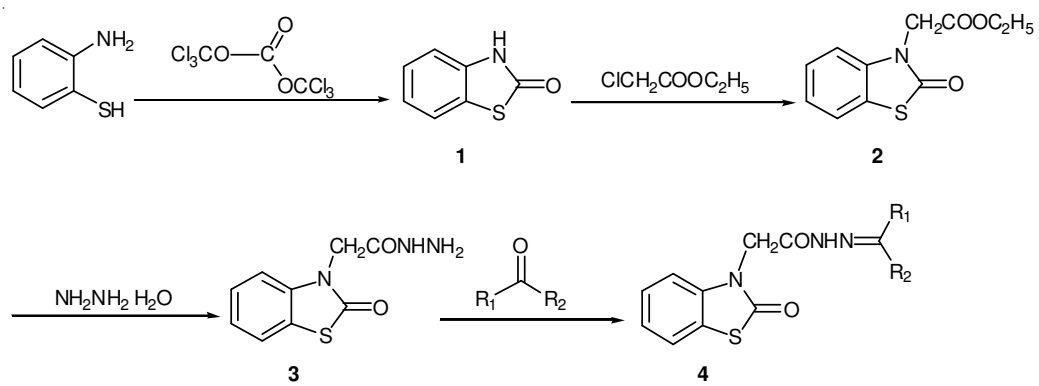
Meanwhile, it is reported that the hydrazones possess a diverse range of bioactivities in agrochemical field, such as insecticidal[10], fungicidal[11], herbicidal[12] and acaricidal[13] activity. In addition, hydrazones are very useful starting materials in many reactions for the synthesis of various bioactive molecules[14].

In view of these facts and also as a part of our work on the development of bioactive heterocyclic compounds, herein we report the synthesis, characterization, and biological study of a series of hydrazone derivatives containing 2(3*H*)-benzothiazolone moiety.

EXPERIMENTAL

Melting points were conducted on a X-4 melting-point apparatus and are uncorrected. The ^{1}H NMR spectra were recorded on Bruker ADVANCE III instrument (500 MHz) using TMS as an internal standard and $CDCl_3$ or DMSO-d_6 as solvents. FTIR spectra were recorded on a NICOLET 6700 instrument. Mass spectra (EI, 70 eV) were recorded on a Thermo Scientific ITQ 1100 TM instrument. Elemental analyses were performed on a Vario EL elemental analyzer.

Synthesis of compounds: All the hydrazone derivatives were synthesized according to the route as shown in **Scheme-I** and theyields were not optimized.



Scheme-I Synthetic route for compounds 4a-o

2(3*H*)-Benzothiazolone (1): *o*-Aminothiophenol (12.6 g, 0.10 mol) and Et_3N (33.8 mL, 0.24 mol) were dissolved in 30 mL of $CHCl_3$, cooled to 0 ºC and a solution of triphosgene (10.5 g, 0.035 mol) in 15 mL of $CHCl_3$ was added dropwise. The resulting mixture was refluxed for 7 h and then cooled to room temperature. Washed with H_2O (3 × 50 mL), the organic layer was dried over anhydrous $MgSO_4$ and evaporated *in vacuo*. The residual solid was recrystallized from ethyl acetate to give pure 2(3*H*)-benzothiazolone (**1**). Yield: 80.4 %; m.p. 140-142 ºC (Lit[15]. m.p. 140-141 ºC). ^{1}H NMR($CDCl_3$) δ: 7.14-7.42 (m, -Ph, 4H), 9.75 (s, NH, 1H).

Ethyl 2(3*H*)-benzothiazolinon-3-ylacetate (2): White solid, 94.5 % yield according to the literature[16], m.p. 92-93 ºC (Lit[16]. m.p. 90-91 ºC). ^{1}H NMR ($CDCl_3$) δ: 1.27 (t, *J* = 7.0 Hz, -CH_2CH_3, 3H), 4.24 (q, *J* = 7.0 Hz, -CH_2CH_3, 2H), 4.68 (s, -CH_2, 2H), 6.89-7.46 (m, -Ph, 4H).

[2(3*H*)-Benzothiazolinon-3-yl]acetohydrazide (3): White solid, 78.0 % yield according to the literature[16], m.p. 213-215 ºC (Lit[16]. m.p. 213-214 ºC). ^{1}H NMR ($CDCl_3$) δ: 4.34 (s, -$NHNH_2$, 2H), 4.68 (s, -CH_2, 2H), 7.16-7.67 (m, -Ph, 4H), 9.44 (s, -$NHNH_2$, 1H).

General procedure for the synthesis of 4a-o: [2(3*H*)-benzothiazolinon-3-yl]acetohydrazide (**3**) (0.018 mol) was dissolved in 30 mL of ethanol and 0.019 mol of an appropriate substituted ketones added, then the final mixture was refluxed for 1.5 h. The precipitate formed was filtered and washed with ethanol, dried and recrystallized from EtOH to give the title compounds **4a-o**.

***N'*-(propan-2-ylidene)-2-(2(3*H*)-benzothiazolinon-3-yl)acetohydrazide(4a):** White solid, 89.4 %. m.p. 243-244 ºC; IR (KBr, ν_{max}, cm^{-1}): 3453 (N-H), 1693 (ring carbonyl), 1679 (hydrazide carbonyl), 1597 (C=N); ^{1}H NMR ($CDCl_3$) δ: 1.83 (s, =CCH_3CH_3, 3H), 2.04 (s, -CCH_3CH_3, 3H), 5.06 (s, N-CH_2, 2H), 6.91-7.44 (m, ArH, 4H), 8.83 (s, 1H, NH); MS m/z: 263 (M^+, 20), 192 (7), 165 (100), 136 (90), 109 (34), 99 (25), 69 (5), 65 (10); Elemental anal. (%), calcd. for $C_{12}H_{13}N_3O_2S$: C, 54.74; H, 4.98; N, 15.96; found: C, 54.59; H, 4.91; N, 15.84.

N'-(butan-2-ylidene)-2-(2(3*H*)-benzothiazolinon-3-yl)acetohydrazide(4b): White solid, 81.7 %. m.p. 169-170 ºC; IR(KBr, ν_{max}, cm^{-1}): 3454 (N-H), 1695 (ring carbonyl), 1670 (hydrazide carbonyl), 1594 (C=N); ^{1}H NMR ($CDCl_3$) δ: 1.14 (t, *J* = 7.5 Hz, -CH_2CH_3, 3H), 1.83 (s, =C-CH_3, 3H), 2.34 (q, *J* = 7.5 Hz, -CH_2CH_3, 2H), 5.08 (s, N-CH_2, 2H), 6.92-7.47 (m, ArH, 4H), 8.52 (s, NH, 1H); MS m/z: 277 (M^+, 20), 248 (7), 192 (7), 165 (90), 136 (100), 113 (55), 109 (40), 65 (15); Elemental anal. (%), calcd. for $C_{13}H_{15}N_3O_2S$: C, 56.30; H, 5.45; N, 15.15; found: C, 56.19; H, 5.39; N, 15.24.

***N'*-(1-phenylethylidene)-2-(2(3*H*)-benzothiazolinon-3-yl)acetohydrazide(4c):** White solid, 84.0 %. m.p. 244-245 ºC; IR(KBr, ν_{max}, cm^{-1}): 3451(N-H), 1694 (ring carbonyl), 1663 (hydrazide carbonyl), 1594 (C=N); ^{1}H NMR ($CDCl_3$) δ: 2.20 (s, =CCH_3, 3H), 5.23 (s, 2H, N-CH_2), 6.94-7.77 (m, ArH, 9H), 9.12 (s, NH, 1H); MS m/z: 325 (M^+, 4), 192 (5), 165 (12), 161 (100), 136 (25), 109 (10), 77 (7), 65 (5); Elemental anal. (%), calcd. for $C_{17}H_{15}N_3O_2S$: C, 62.75; H, 4.65; N, 12.91; found: C, 62.66; H, 4.57; N, 13.02.

***N'*-(1-(2,6-difluorophenyl)ethylidene)-2-(2(3*H*)-benzothiazolinon-3-yl)acetohydrazide(4d):** White solid, 79.2 %. m.p. 247-248 ºC; IR (KBr, ν_{max}, cm^{-1}): 3449 (N-H), 1698 (ring carbonyl), 1665 (hydrazide carbonyl), 1590 (C=N); ^{1}H NMR (DMSO-d_6) δ: 2.26(s, =CCH_3, 3H), 5.00 (s, N-CH_2, 2H), 7.18-7.69 (m, ArH, 7H), 11.27 (s, NH, 1H); MS m/z: 361 (M^+,17), 197 (100), 191 (20), 165 (70), 154 (10), 136 (65), 127 (15), 109 (26); Elemental anal. (%), calcd. for $C_{17}H_{13}N_3O_2SF_2$: C, 56.50; H, 3.63; N, 11.63; found: C, 56.38; H, 3.61; N, 11.80.

***N'*-(1-(4-chlorophenyl)ethylidene)-2-(2(3*H*)-benzothiazolinon-3-yl)acetohydrazide(4e):** White solid, 86.8 %. m.p. 241-242 ºC; IR (KBr, ν_{max}, cm^{-1}): 3455 (N-H), 1704 (ring carbonyl), 1656 (hydrazide carbonyl), 1593 (C=N); ^{1}H NMR (DMSO-d_6) δ: 2.29(s, =CCH_3, 3H), 5.21 (s, N-CH_2, 2H), 7.19-7.91 (m, ArH, 8H), 11.10 (s, NH, 1H); MS m/z: 359 (M^+, 3) 195 (100), 192 (7), 165 (37), 152 (7), 136 (45), 109 (20), 77 (6); Elemental anal. (%), calcd. for $C_{17}H_{14}N_3O_2SCl$: C, 56.74; H, 3.92; N, 11.68; found: C, 56.55; H, 3.83; N, 11.84.

***N'*-(1-(p-tolyl)ethylidene)-2-(2(3*H*)-benzothiazolinon-3-yl)acetohydrazide(4f):** White solid, 88.5 %. m.p. 246-247 ºC; IR (KBr, ν_{max}, cm^{-1}): 3447 (N-H), 1698 (ring carbonyl), 1679 (hydrazide carbonyl), 1596 (C=N); ^{1}H NMR ($CDCl_3$) δ: 2.20 (s, =CCH_3, 3H), 2.39 (s, -PhCH_3, 3H), 5.22 (s, N-CH_2, 2H), 6.94-7.67 (m, ArH, 8H), 9.15 (s, NH, 1H); MS m/z: 339 (M^+, 3), 175 (100), 164 (8), 136 (17), 132 (10), 109 (7), 91 (6), 65 (5); Elemental anal. (%), calcd. for $C_{18}H_{17}N_3O_2S$: C, 63.70; H, 5.05; N, 12.38; found: C, 63.52; H, 4.96; N, 12.54.

***N'*-(1-(4-nitrophenyl)ethylidene)-2-(2(3*H*)-benzothiazolinon-3-yl)acetohydrazide(4g):** Yellow solid, 80.7 %. m.p. 302-303 ºC; IR (KBr, ν_{max}, cm^{-1}): 3456 (N-H), 1708 (ring carbonyl), 1652 (hydrazide carbonyl), 1589 (C=N); ^{1}H NMR (DMSO-d_6) δ: 2.36 (s, =CCH_3, 3H), 5.26 (s, N-CH_2, 2H), 7.20-8.27 (m, ArH, 8H), 11.32 (s, NH, 1H); MS m/z: 370 (M^+,11), 206 (55), 192 (30), 165 (100), 136 (85), 109 (32), 77 (7), 65 (10); Elemental anal. (%), calcd. for $C_{17}H_{14}N_4O_4S$: C, 55.13; H, 3.81; N, 15.13; found: C, 54.96; H, 3.73; N, 15.34.

***N'*-(1-(4-fluorophenyl)ethylidene)-2-(2(3*H*)-benzothiazolinon-3-yl)acetohydrazide(4h):** White solid, 74.9 %. m.p. 247-248 º; IR (KBr, ν_{max}, cm_{-1}): 3455 (N-H), 1696 (ring carbonyl), 1679 (hydrazide carbonyl), 1596 (C=N); ^{1}H NMR ($CDCl_3$) δ: 2.22 (s, =CCH_3, 3H), 5.21 (s, N-CH_2, 2H), 6.94-7.78 (m, ArH, 8H), 9.08 (s, NH, 1H); MS m/z: 343 (M^+, 5) 192 (7), 179 (100), 165 (30), 136 (42), 109 (20), 95 (5), 83 (7); Elemental anal. (%), calcd. for $C_{17}H_{14}N_3O_2SF$: C, 59.46; H, 4.11; N, 12.24; found: C, 59.29; H, 4.03; N, 12.41.

***N'*-(1-(3-chlorophenyl)ethylidene)-2-(2(3*H*)-benzothiazolinon-3-yl)acetohydrazide(4i):** White solid, 80.6 %. m.p. 226-227 ºC; IR (KBr, ν_{max}, cm^{-1}): 3453 (N-H), 1690 (ring carbonyl), 1673 (hydrazide carbonyl), 1589 (C=N); ^{1}H NMR (DMSO-d_6) δ: 2.30 (s, =CCH_3, 3H), 5.23 (s, N-CH_2, 2H), 7.20-7.91 (m, ArH, 8H), 11.41 (s, NH, 1H); MS m/z: 359 (M^+, 3), 197 (35), 195 (100), 165 (35), 136 (48), 109 (20), 77 (6), 65 (6); Elemental anal. (%), calcd. for $C_{17}H_{14}N_3O_2SCl$: C, 56.74; H, 3.92; N, 11.68; found: C, 56.58; H, 3.84; N, 11.84.

***N'*-(1-(2-methoxyphenyl)ethylidene)-2-(2(3*H*)-benzothiazolinon-3-yl)acetohydrazide(4j):** White solid, 73.3 %. m.p. 217-218 ºC; IR (KBr, ν_{max}, cm^{-1}): 3453 (N-H), 1702 (ring carbonyl), 1652 (hydrazide carbonyl), 1594 (C=N); ^{1}H NMR ($CDCl_3$) δ: 2.21 (s, =CCH_3, 3H), 3.88 (s, -OCH_3, 3H), 5.14 (s, N-CH_2, 2H), 6.92-7.44 (m, ArH, 8H), 8.76 (s, NH, 1H); MS m/z: 355 (M^+, 3), 324 (7), 191 (100), 164 (10), 136 (22), 133

TABLE-1
THE FUNGICIDAL ACTIVITIES OF TITLE COMPOUNDS *IN VITRO* AT 500 mg/L

No.	*Sclerotonia sclerotiorum*	*Botrytis cinerea*	*Alternaria alternata*	*Ustilaginoidea oryzae*	*Rhizoctonia solani*
4a	40.6	0.0	43.7	0.0	70.0
4b	0.0	0.0	47.2	0.0	60.0
4c	9.4	0.0	12.7	0.0	0.0
4d	28.1	0.0	17.0	0.0	95.0
4e	56.3	0.0	21.5	0.0	70.0
4f	0.0	0.0	32.6	0.0	95.0
4g	0.0	0.0	39.2	0.0	90.0
4h	0.0	0.0	19.6	0.0	0.0
4i	0.0	0.0	28.7	0.0	95.0
4j	0.0	0.0	31.3	0.0	0.0
4k	37.5	0.0	20.5	0.0	90.0
4l	43.8	0.0	24.8	0.0	20.0
4m	37.5	0.0	37.6	0.0	90.0
4n	71.9	0.0	42.5	0.0	0.0
4o	78.1	0.0	37.9	0.0	0.0
propiconazole	87.5	0.0	0.0	0.0	90.0

(15), 105 (12), 91 (7); Elemental anal. (%), calcd. for $C_{18}H_{17}N_3O_3S$: C, 60.83; H, 4.82; N, 11.82; found: C, 60.62; H, 4.68; N, 11.97.

***N'*-(1-(4-methoxyphenyl)ethylidene)-2-(2(3*H*)-benzothiazolinon-3-yl)acetohydrazide(4k):** White solid, 85.2%. m.p. 218-219 ºC; IR (KBr, ν_{max}, cm^{-1}): 3461 (N-H), 1693 (ring carbonyl), 1656 (hydrazide carbonyl), 1600 (C=N); ^{1}H NMR ($CDCl_3$) δ: 2.16 (s, $=CCH_3$, 3H), 3.86 (s, $-OCH_3$, 3H), 5.21(s, $N-CH_2$, 2H), 6.92-7.73 (m, ArH, 8H), 9.07 (s, NH, 1H); MS m/z: 355 (M^+, 5), 195 (4), 191 (100), 164 (10), 136 (17), 109 (8), 77 (4), 65 (4); Elemental anal. (%), calcd. for $C_{18}H_{17}N_3O_3S$: C, 60.83; H, 4.82; N, 11.82; found: C, 60.69; H, 4.71; N, 12.01.

N'-(1-(4-bromophenyl)ethylidene)-2-(2(3*H*)-benzothiazolinon-3-yl)acetohydrazide(4l): White solid, 92.4 %. m.p. 266-267 ºC; IR (KBr, ν_{max}, cm^{-1}): 3455 (N-H), 1706 (ring carbonyl), 1656 (hydrazide carbonyl), 1600 (C=N); ^{1}H NMR (DMSO-d_6) δ: 2.29 (s, $=CCH_3$, 3H), 5.21 (s, $N-CH_2$, 2H), 7.19-7.84 (m, ArH, 8H), 11.13 (s, NH, 1H); MS m/z: 404 (M^+,3), 239 (100), 192 (25), 165 (82), 160 (14), 136 (96), 109 (40), 77 (14); Elemental anal. (%), calcd. for $C_{17}H_{14}BrN_3O_2S$: C, 50.50; H, 3.49; N, 10.39; found: C, 50.32; H, 3.41; N, 10.64.

***N'*-(3-methylbutan-2-ylidene)-2-(2(3*H*)-benzothiazolinon-3-yl)acetohydrazide(4m):** White solid, 83.9 %. m.p. 179-180 ºC; IR (KBr, ν_{max}, cm^{-1}): 3454 (N-H), 1692 (ring carbonyl), 1654 (hydrazide carbonyl), 1588 (C=N); ^{1}H NMR ($CDCl_3$) δ: 1.13 (d, J = 7.0 Hz, $-CH(CH_3)_2$, 6H), 1.77 (s, $=CCH_3$, 3H), 2.50-2.56 (m, -CH, 1H), 5.07 (s, $N-CH_2$, 2H), 6.92-7.45 (m, ArH, 4H); 8.70 (s, NH, 1H); MS m/z: 291 (M^+,12), 248 (45), 165 (50), 136 (100), 127 (80), 109 (37), 85 (7), 65 (17); Elemental anal. (%), calcd. for $C_{14}H_{17}N_3O_2S$: C, 57.71; H, 5.88; N, 14.42; found: C, 57.52; H, 5.71; N, 14.64.

***N'*-cyclopentylidene-2-(2(3*H*)-benzothiazolinon-3-yl)acetohydrazide(4n):** White solid, 73.8 %. m.p. 188-189 ºC; IR (KBr, ν_{max}, cm^{-1}): 3455 (N-H), 1696 (ring carbonyl), 1671 (hydrazide carbonyl), 1594 (C=N); ^{1}H NMR ($CDCl_3$) δ: 1.75-1.90 (m, $-CH_2(CH_2)_2CH_2-$, 4H), 2.19 (t, J = 7.5 Hz, $-CH_2(CH_2)_2CH_2-$, 2H), 2.45 (t, J = 7.5 Hz, $-CH_2(CH_2)_2CH_2-$, 2H), 5.05 (s, $N-CH_2$, 2H), 6.92-7.44 (m, ArH, 4H); 8.34 (s, NH, 1H); MS m/z: 289 (M^+,15), 192 (12), 165 (100), 136 (92), 125 (60), 109 (40), 82 (14), 65 (17); Elemental anal. (%), calcd. for $C_{14}H_{15}N_3O_2S$: C, 58.11; H, 5.23; N, 14.52; found: C, 57.90; H, 5.01; N, 14.74.

***N'*-cyclohexylidene-2-(2(3*H*)-benzothiazolinon-3-yl)acetohydrazide(4o):** White solid, 78.1%. m.p. 165-166 ºC; IR (KBr, ν_{max}, cm^{-1}): 3453 (N-H), 1695 (ring carbonyl), 1673 (hydrazide carbonyl), 1593 (C=N); ^{1}H NMR ($CDCl_3$) δ: 1.56-1.75 (m, $-CH_2(CH_2)_3CH_2-$, 6H), 2.22-2.36 (m, $-CH_2(CH_2)_3CH_2-$, 4H), 5.08 (s, $N-CH_2$, 2H), 6.93-7.47 (m, ArH, 4H); 8.70 (s, NH, 1H); MS m/z: 303 (M^+, 10), 191 (7), 165 (60), 139 (100), 136 (60), 109 (26), 96 (15), 65 (11); Elemental anal. (%), calcd. for $C_{15}H_{17}N_3O_2S$: C, 59.38; H, 5.65; N, 13.85; found: C, 59.17; H, 5.54; N, 14.01.

Bioassay of fungicidal activities: Fungicidal activities of the title compounds against *Rhizoctonia solani, Botrytis cinerea, Sclerotonia sclerotiorum, Alternaria alternata, Ustilaginoidea oryzae* were evaluated using pot culture test according to reference.[17] The culture plates were cultivated at (24 ± 1) ºC. The relative inhibition rate of the circle mycelium compared to blank assay was calculated *via* the following equation:

$$\text{Relative ratio } \% = (N_S - N_C)/N_C \times 100\ \%$$

where, N_S is the extended diameter of the circle mycelium during the blank assay; and N_C, is the extended diameter of the circle mycelium during testing.

RESULTS AND DISCUSSION

Fifteen novel hydrazone derivatives containing 2(3*H*)-benzothiazolone moiety were synthesized by condensing [2(3*H*)-benzothiazolinon-3-yl]acetohydrazide with substituted ketones. Reaction mixtures were maintained at refluxing temperature, leading to the desired compounds in 73.3-92.4 % yields. All the compounds were identified and characterized by ^{1}H NMR, FTIR, MS and elemental analysis. The IR spectra indicated the presence of two carbonyl bands at 1690-1708 and 1679-1652 cm^{-1} (for the ring carbonyl and hydrazide carbonyl, respectively). Meanwhile, all of the title compounds exhibited M^+ peak in the EI-MS results.

Fungicidal activities: The results of fungicidal activity testing are listed in Table-1. At the dose of 500 mg/L, some of title compounds exhibited excellent fungicidal activity against *Rhizoctonia solani*, which is compared with the commercial fungicide propiconazole. For example, the compounds **4d, 4f, 4g, 4i, 4k** and **4m** can inhibit *Rhizoctonia solani* above 90.0 %. And some of them exhibited moderate fungicidal activity

against *Sclerotonia sclerotiorum*. For example, the fungicidal activity of compounds **4n** and **4o** against *Sclerotonia sclerotiorum* reach 71.9 and 78.1 % respectively. All compounds displayed weak fungicidal activity against *Alternaria alternate* and had no fungicidal activity against *Botrytis cinerea* and *Ustilaginoidea oryzae*.

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