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## AN EFFICIENTLY SONOCHEMICAL SYNTHESIS OF 2-(*N*-ARYLSULFONYLINDOL-3-YL)-3-*N*-ACYL-5-PHENYL-1,3,4-OXADIAZOLINES

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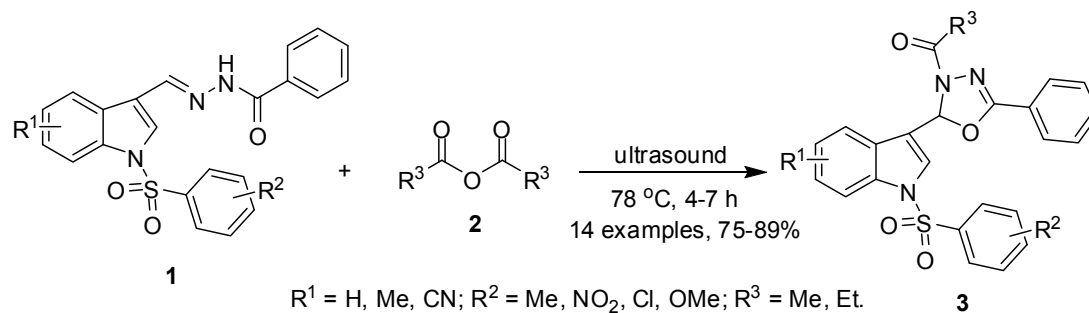
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Dedicated to Professor Dr. Albert Eschenmoser on the occasion of his 85<sup>th</sup> birthday

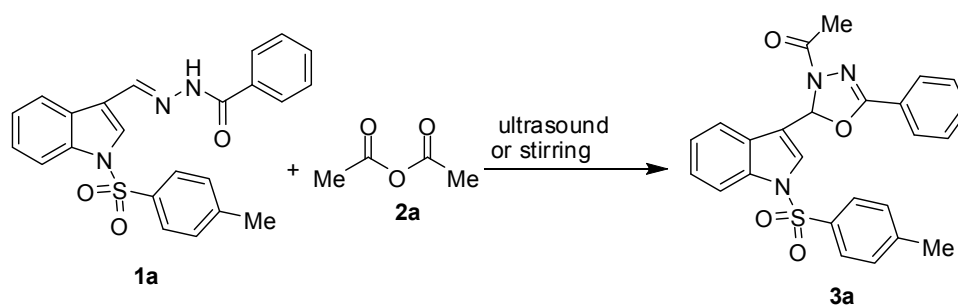
**Abstract** – An efficient and rapid synthesis of 2-(*N*-arylsulfonylindol-3-yl)-3-*N*-acyl-5-phenyl-1,3,4-oxadiazolines from *N*-arylsulfonyl-3-formylindole benzoyl hydrazones and anhydrides under ultrasonic irradiation in good yields is described.

The *N*-arylsulfonylindole subunits have gained widespread interest due to their key roles in medically important species, such as those displaying serotonin receptor affinity,<sup>1</sup> potent antagonists against the peptidoleukotrienes,<sup>2</sup> and anti-HIV-1 activity.<sup>3,4</sup> On the other hand, the 1,3,4-oxadiazoline ones exhibit antibacterial activity,<sup>5</sup> inhibiting activity against chitin synthesis,<sup>6</sup> and antiviral activity.<sup>7</sup> In continuation of our program aimed at the discovery and development of compounds with superior biological activities, therefore, we want to prepare some 2-(*N*-arylsulfonylindol-3-yl)-3-*N*-acyl-5-phenyl-1,3,4-oxadiazoline analogs by combining the *N*-arylsulfonylindole units with the 1,3,4-oxadiazolines together.

Ultrasound has increasingly been used in organic synthesis in recent years. Compared with the traditional methods with stirring, many organic reactions could be carried out in higher yields, shorter reaction time and milder reaction conditions under ultrasonic irradiation.<sup>8</sup> However, to the best of our knowledge, the ultrasound-assisted synthesis of 2-(*N*-arylsulfonylindol-3-yl)-3-*N*-acyl-5-phenyl-1,3,4-oxadiazolines from hydrazones and anhydrides has not yet been studied. Herein we report the synthesis of 2-(*N*-arylsulfonylindol-3-yl)-3-*N*-acyl-5-phenyl-1,3,4-oxadiazolines (**3a-n**) from *N*-arylsulfonyl-3-formylindole benzoyl hydrazones (**1a-l**) and anhydrides (**2a-b**) by ultrasonic irradiation (Scheme 1).



Scheme 1

Table 1. Optimization studies<sup>a</sup>

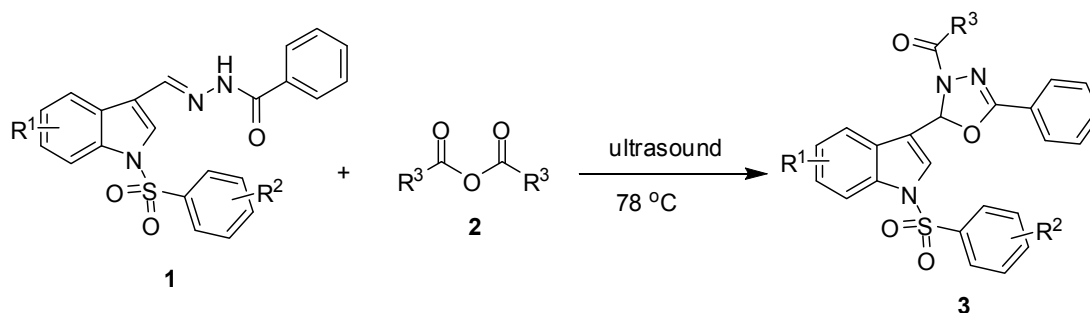
Entry	Reaction conditions	Temperature (°C)	Time (h)	Yield of <b>3a</b> (%) <sup>b</sup>
1	stirring	25	48	< 2
2	stirring	40	24	< 7
3	stirring	40	48	22
4	stirring	60	24	31
5	stirring	60	48	57
6	stirring	80	12	54
7	stirring	80	24	80
8	ultrasound	58	6	47
9	ultrasound	58	10	68
10	ultrasound	68	10	79
11	ultrasound	78	4	89

<sup>a</sup> 0.5 mmol **1a** reacted with 5 mL **2a**; <sup>b</sup> Isolated yield.

We firstly investigated the reaction of *N*-toluenesulfonyl-3-formylindole benzoyl hydrazone (**1a**) with acetic anhydride (**2a**) under different reaction conditions, and the results were summarized in Table 1. When the reaction mixture of **1a** and **2a** was stirred under the traditional conditions at 25 °C, 40 °C, or 60

°C for 48 h, the corresponding yields of 2-(*N*-toluenesulfonyl indol-3-yl)-3-*N*-acetyl-5-phenyl-1,3,4-oxadiazoline (**3a**) were < 2%, 22%, and 57%, respectively (Table 1, entries 1, 3 and 5). Even if the reaction temperature was raised to 80 °C for 12 h, the yield of **3a** was only 54% (Table 1, entry 6), and when the reaction time was prolonged to 24 h, **3a** was obtained in the 80% yield. On the contrary, once **1a** reacted with **2a** under ultrasonic irradiation, the yields were improved and the reaction time was shortened (Table 1, entries 8-11). For example, when the mixture was reacted at 58 °C for 10 h under ultrasonic irradiation, the yield of **3a** was 68% (Table 1, entries 4 and 5 vs. 9). Consequently, the ultrasound could accelerate the synthesis of **3a**. Especially, when the reaction temperature was raised from 68 °C to 78 °C, the yield of **3a** was improved from 79% to 89%, while the reaction time was reduced from 10 h to 4 h (Table 1, entries 10 and 11). Obviously, the ultrasonic irradiation and the reaction temperature were two very important factors to the above reaction. The optimized reaction condition for the synthesis of **3a** was the reaction of **1a** with **2a** at 78 °C under ultrasonic irradiation.

**Table 2.** Synthesis of 2-(*N*-arylsulfonylindol-3-yl)-3-*N*-acyl-5-phenyl-1,3,4-oxadiazolines (**3a-n**) by ultrasonic irradiation

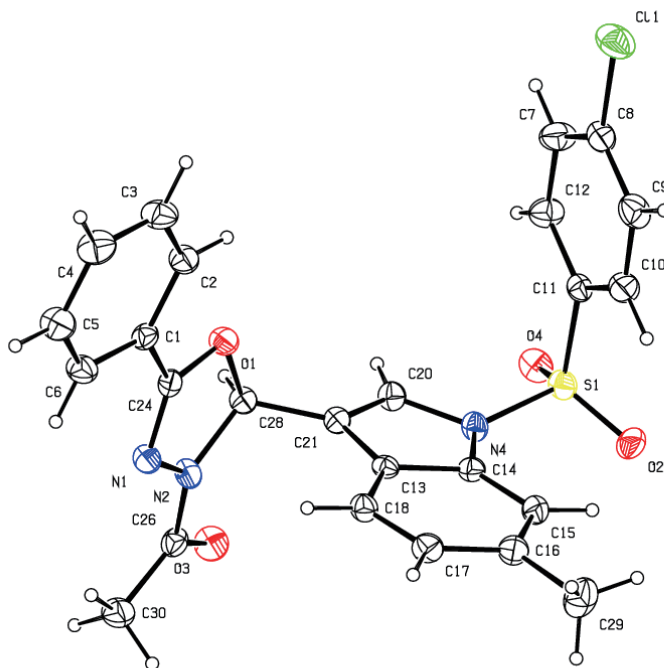


Compound	R <sup>1</sup>	R <sup>2</sup>	R <sup>3</sup>	Time (h)	Yield (%) <sup>a</sup>
<b>3a</b>	H	4-Me	Me	4	89
<b>3b</b>	H	3-NO <sub>2</sub>	Me	5	78
<b>3c</b>	6-Me	4-Me	Me	5	76
<b>3d</b>	H	3-NO <sub>2</sub> , 4-Cl	Me	6.5	75
<b>3e</b>	6-Me	3-NO <sub>2</sub>	Me	6.5	75
<b>3f</b>	6-Me	3-NO <sub>2</sub> , 4-Cl	Me	6.5	75
<b>3g</b>	H	4-Cl	Me	6.5	77
<b>3h</b>	6-Me	4-Cl	Me	6	76
<b>3i</b>	H	4-OMe	Me	7	87
<b>3j</b>	6-Me	4-OMe	Me	7	83
<b>3k</b>	5-CN	4-Me	Me	7	88
<b>3l</b>	5-CN	3-NO <sub>2</sub>	Me	7	89
<b>3m</b>	H	4-Me	Et	5.5	88
<b>3n</b>	5-CN	4-Me	Et	6.5	87

<sup>a</sup> Isolated yield.

Based upon the above findings, we further studied the reaction of different *N*-arylsulfonyl-3-formylindole benzoyl hydrazones (**1a-l**) with anhydrides (**2a-b**) at 78 °C under ultrasonic irradiation. As shown in Table 2, a wide range of **1** ( $R^1 = \text{H, Me, CN}$ ; and  $R^2 = \text{Me, Cl, NO}_2, \text{OMe}$ ), including electron-withdrawing and electron-donating substituents, efficiently reacted with **2** ( $R^3 = \text{Me, Et}$ ) under the optimum reaction conditions. 2-(*N*-Arylsulfonylindol-3-yl)-3-*N*-acyl-5-phenyl-1,3,4-oxadiazolines (**3a-n**) were obtained in 75-89% yields for 4-7 h. For example, when *N*-toluenesulfonyl-3-formyl-6-methylindole benzoyl hydrazone (**1c**) reacted with acetic anhydride (**2a**) at 78 °C for 5 h under ultrasonic irradiation, 2-(*N*-toluenesulfonyl-6-methylindol-3-yl)-3-*N*-acetyl-5-phenyl-1,3,4-oxadiazoline (**3c**) was obtained in a 76% yield (Table 2, entry 3); when *N*-toluenesulfonyl-3-formyl-5-cyanoindole benzoyl hydrazone (**1k**) reacted with acetic anhydride (**2a**) at 78 °C for 7 h under ultrasonic irradiation, the yield of 2-(*N*-toluenesulfonyl-5-cyanoindol-3-yl)-3-*N*-acetyl-5-phenyl-1,3,4-oxadiazoline (**3k**) was 88% (Table 2, entry 11). On the other hand, when the propionic anhydride (**2b**) reacted with **1a** or **1k** at 78 °C under ultrasonic irradiation, the corresponding yields of **3m** and **3n** were 88% for 5.5 h, and 87% for 6.5 h, respectively (Table 2, entries 13 and 14).

Meanwhile, to obtain the precise three-dimensional structural information of **3a-n**, the structure of 2-(*N*-4-chlorophenylsulfonyl-6-methylindol-3-yl)-3-*N*-acetyl-5-phenyl-1,3,4-oxadiazoline (**3h**) was confirmed by X-ray crystal analysis (Figure 1).<sup>9</sup>



**Figure 1.** The X-ray crystallography of 2-(*N*-4-chlorophenylsulfonyl-6-methylindol-3-yl)-3-*N*-acetyl-5-phenyl-1,3,4-oxadiazoline (**3h**).

The above results might be due to the “cavitation” from ultrasonic waves propagating in a liquid medium. During the rarefaction cycle of the wave, the molecules of the liquid are separated, generating bubbles that subsequently collapse in the compression cycle. These rapid and violent implosions generate short-lived regions with high temperatures and pressures, therefore, the highly reactive species are locally produced, and the energy of sound is transformed into a useful chemical form.<sup>10</sup> Accordingly, sonication could probably provide more efficient influence on the reactions than the traditional conditions with stirring, and reduce the reaction time sharply.

In summary, we have described an efficient and rapid method for the synthesis of 2-(*N*-arylsulfonylindol-3-yl)-3-*N*-acyl-5-phenyl-1,3,4-oxadiazolines from *N*-arylsulfonyl-3-formylindole benzoyl hydrazones with acetic or propionic anhydride in good yields under ultrasonic irradiation. Compared to the traditional conditions using stirring, the main advantage of the present procedure is milder conditions and shorter reaction time.

## EXPERIMENTAL

The materials were used as purchased. Analytical thin-layer chromatography (TLC) and preparative thin-layer chromatography (PTLC) were performed with silica gel plates using silica gel 60 GF<sub>254</sub> (Qingdao Haiyang Chemical Co., Ltd.). Melting points were uncorrected. <sup>1</sup>H NMR spectra were recorded on a Bruker Avance DMX 300 or 400 MHz instrument using TMS as an internal standard and CDCl<sub>3</sub> as a solvent. EI-MS and ESI-TRAP-MS were carried out with the HP 5988, and the Bruker ESI-TRAP Esquire 3000 plus mass spectrometry instruments, respectively. HRMS were carried out with APEX II Bruker 4.7T AS instrument. Ultrasonic irradiation was performed in Ningbo SB-5200DT ultrasonic cleaner with the size of the interior trough of 300 × 240 × 150 mm, the frequency of 40 kHz, and an output power of 200 W. The temperature of the water bath was controlled by addition or removal of water.

### General procedure for the preparation of 2-(*N*-arylsulfonylindol-3-yl)-3-*N*-acyl-5-phenyl-1,3,4-oxadiazolines (3a-n) by ultrasonic irradiation

The 50 mL rockered flask which was filled with a mixture of **1** (0.5 mmol) and **2** (5 mL) was immersed into the zone of maximum cavitation of water bath of ultrasonic cleaner, and the surface of the reaction mixture was kept at a slightly lower level than the level of the water in the bath. Subsequently, the mixture was irradiated by ultrasound at 78 °C until complete consumption of the starting material checked by TLC, and the reaction time was indicated in Table 2. Then the reaction mixture was poured into ice water and stirred until the precipitate was produced, which was filtered, washed with water, and dissolved in CH<sub>2</sub>Cl<sub>2</sub> (30 mL). Finally, the organic solution was washed with saturated aqueous NaHCO<sub>3</sub> (30 mL × 2), brine (20 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered, concentrated *in vacuo* and purified by

preparative TLC to give the pure 2-(*N*-arylsulfonylindol-3-yl)-3-*N*-acyl-5-phenyl-1,3,4-oxadiazolines (**3a-n**). All compounds were characterized by <sup>1</sup>H-NMR (300 or 400 MHz), EI-MS and mp. The yields of **3a-n** were listed in Table 2.

**Compound 3a**: White solid, mp 54-55 °C; <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>) δ: 7.78-7.94 (m, 6H), 7.44-7.52 (m, 4H), 7.22-7.33 (m, 5H), 2.35 (s, 6H); EI-MS *m/z*: 459 (M<sup>+</sup>, 30); HRMS: Calcd. for C<sub>25</sub>H<sub>21</sub>N<sub>3</sub>O<sub>4</sub>NaS (M+Na<sup>+</sup>): 482.1145. Found: 482.1148.

**Compound 3b**: Yellow solid, mp 99-100 °C; <sup>1</sup>H-NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ: 8.71 (s, 1H), 8.54 (d, *J* = 6.3 Hz, 2H), 8.42 (d, *J* = 6.3 Hz, 1H), 7.82-8.06 (m, 4H), 7.30-7.57 (m, 7H), 2.24 (s, 3H); EI-MS *m/z*: 490 (M<sup>+</sup>, 5); HRMS: Calcd. for C<sub>24</sub>H<sub>18</sub>N<sub>4</sub>O<sub>6</sub>NaS (M+Na<sup>+</sup>): 513.0839. Found: 513.0837.

**Compound 3c**: White solid, mp 145-146 °C; <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>) δ: 7.89 (d, *J* = 7.2 Hz, 2H), 7.73-7.79 (m, 4H), 7.24-7.50 (m, 7H), 7.03 (d, *J* = 7.8 Hz, 1H), 2.43 (s, 3H), 2.34 (s, 6H); EI-MS *m/z*: 473 (M<sup>+</sup>, 10); HRMS: Calcd. for C<sub>26</sub>H<sub>23</sub>N<sub>3</sub>O<sub>4</sub>NaS (M+Na<sup>+</sup>): 496.1301. Found: 496.1308.

**Compound 3d**: Yellow solid, mp 54-55 °C; <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>) δ: 8.41 (s, 1H), 7.88-7.95 (m, 4H), 7.80 (s, 1H), 7.65 (d, *J* = 8.1 Hz, 1H), 7.30-7.52 (m, 7H), 2.33 (s, 3H); EI-MS *m/z*: 524 (M<sup>+</sup>, 3); HRMS: Calcd. for C<sub>24</sub>H<sub>17</sub>N<sub>4</sub>O<sub>6</sub>NaSCl (M+Na<sup>+</sup>): 547.0450. Found: 547.0457.

**Compound 3e**: Yellow solid, mp 81-82 °C; <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>) δ: 8.77 (s, 1H), 8.40 (d, *J* = 8.1 Hz, 1H), 8.15 (d, *J* = 7.8 Hz, 1H), 7.88 (d, *J* = 7.5 Hz, 2H), 7.78 (d, *J* = 9.9 Hz, 2H), 7.66-7.71 (m, 1H), 7.37-7.54 (m, 4H), 7.29 (s, 1H), 7.08 (d, *J* = 7.8 Hz, 1H), 2.46 (s, 3H), 2.33 (s, 3H); EI-MS *m/z*: 504 (M<sup>+</sup>, 28); HRMS: Calcd. for C<sub>25</sub>H<sub>20</sub>N<sub>4</sub>O<sub>6</sub>NaS (M+Na<sup>+</sup>): 527.0996. Found: 527.0994.

**Compound 3f**: Yellow solid, mp 84-85 °C; <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>) δ: 8.41 (s, 1H), 7.87-7.90 (m, 3H), 7.73 (s, 2H), 7.62-7.65 (m, 1H), 7.27-7.52 (m, 5H), 7.09 (d, *J* = 7.5 Hz, 1H), 2.45 (s, 3H), 2.32 (s, 3H); EI-MS *m/z*: 538 (M<sup>+</sup>, 85); HRMS: Calcd. for C<sub>25</sub>H<sub>19</sub>N<sub>4</sub>O<sub>6</sub>NaSCl (M+Na<sup>+</sup>): 561.0606. Found: 561.0613.

**Compound 3g**: White solid, mp 148-149 °C; <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>) δ: 7.81-7.93 (m, 6H), 7.19-7.52 (m, 9H), 2.35 (s, 3H); EI-MS *m/z*: 479 (M<sup>+</sup>, 70); HRMS: Calcd. for C<sub>24</sub>H<sub>18</sub>N<sub>3</sub>O<sub>4</sub>NaSCl (M+Na<sup>+</sup>): 502.0599. Found: 502.0592.

**Compound 3h**: White solid, mp 221-222 °C; <sup>1</sup>H-NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ: 8.16 (s, 1H), 8.10 (d, *J* = 5.7 Hz, 2H), 7.78-7.82 (m, 3H), 7.73 (d, *J* = 6.9 Hz, 2H), 7.52-7.59 (m, 3H), 7.43 (s, 1H), 7.09-7.23 (m, 2H), 2.41 (s, 3H), 2.23 (s, 3H); MS (ESI-TRAP) *m/z*: 494 ((M+H)<sup>+</sup>, 100); HRMS: Calcd. for C<sub>25</sub>H<sub>20</sub>N<sub>3</sub>O<sub>4</sub>NaSCl (M+Na<sup>+</sup>): 516.0755. Found: 516.0759.

**Compound 3i**: White solid, mp 63-64 °C; <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) δ: 7.83-7.93 (m, 6H), 7.49-7.52 (m, 2H), 7.45 (t, *J* = 7.6 Hz, 2H), 7.28-7.33 (m, 2H), 7.21 (t, *J* = 7.6 Hz, 1H), 6.91 (d, *J* = 9.2 Hz, 2H), 3.82 (s, 3H), 2.35 (s, 3H); EI-MS *m/z*: 475 (M<sup>+</sup>, 24); HRMS: Calcd. for C<sub>25</sub>H<sub>21</sub>N<sub>3</sub>O<sub>5</sub>NaS (M+Na<sup>+</sup>): 498.1094. Found: 498.1089.

**Compound 3j:** White solid, mp 78-79 °C; <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) δ: 7.83-7.88 (m, 4H), 7.76 (s, 1H), 7.73 (s, 1H), 7.52 (t, *J* = 7.6 Hz, 1H), 7.45 (t, *J* = 7.2 Hz, 2H), 7.38 (d, *J* = 8.8 Hz, 1H), 7.31 (s, 1H), 7.02 (d, *J* = 8.4 Hz, 1H), 6.91 (d, *J* = 8.8 Hz, 2H), 3.79 (s, 3H), 2.43 (s, 3H), 2.34 (s, 3H); EI-MS *m/z*: 489 (M<sup>+</sup>, 56); HRMS: Calcd. for C<sub>26</sub>H<sub>23</sub>N<sub>3</sub>O<sub>5</sub>NaS (M+Na<sup>+</sup>): 512.1251. Found: 512.1260.

**Compound 3k:** White solid, mp 231-232 °C; <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) δ: 8.04 (d, *J* = 8.4 Hz, 1H), 7.93 (s, 1H), 7.87-7.89 (m, 3H), 7.81 (d, *J* = 8.0 Hz, 2H), 7.52-7.58 (m, 2H), 7.48 (t, *J* = 7.2 Hz, 2H), 7.31 (d, *J* = 9.6 Hz, 3H), 2.37 (s, 6H); MS (ESI-TRAP) *m/z*: 485 ((M+H)<sup>+</sup>, 65); HRMS: Calcd. for C<sub>26</sub>H<sub>20</sub>N<sub>4</sub>O<sub>4</sub>NaS (M+Na<sup>+</sup>): 507.1097. Found: 507.1094.

**Compound 3l:** Yellow solid, mp 227-228 °C; <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>) δ: 8.78 (s, 1H), 8.48 (d, *J* = 7.8 Hz, 1H), 8.21 (d, *J* = 7.5 Hz, 1H), 8.10 (d, *J* = 8.4 Hz, 1H), 7.86-7.94 (m, 4H), 7.79 (t, *J* = 7.8 Hz, 1H), 7.66 (d, *J* = 8.7 Hz, 1H), 7.44-7.54 (m, 3H), 7.30 (s, 1H), 2.36 (s, 3H); MS (ESI-TRAP) *m/z*: 516 ((M+H)<sup>+</sup>, 100); HRMS: Calcd. for C<sub>25</sub>H<sub>17</sub>N<sub>5</sub>O<sub>6</sub>NaS (M+Na<sup>+</sup>): 538.0792. Found: 538.0797.

**Compound 3m:** White solid, mp 119-120 °C; <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>) δ: 7.78-7.94 (m, 6H), 7.18-7.51 (m, 9H), 2.69-2.79 (m, 2H), 2.34 (s, 3H), 1.17 (t, *J* = 7.5 Hz, 3H); EI-MS *m/z*: 473 (M<sup>+</sup>, 8); HRMS: Calcd. for C<sub>26</sub>H<sub>23</sub>N<sub>3</sub>O<sub>4</sub>NaS (M+Na<sup>+</sup>): 496.1301. Found: 496.1306.

**Compound 3n:** White solid, mp 174-175 °C; <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>) δ: 7.79-8.04 (m, 7H), 7.45-7.58 (m, 4H), 7.28-7.36 (m, 3H), 2.71-2.78 (m, 2H), 2.37 (s, 3H), 1.18 (t, *J* = 7.5 Hz, 3H); EI-MS *m/z*: 498 (M<sup>+</sup>, 7); HRMS: Calcd. for C<sub>27</sub>H<sub>22</sub>N<sub>4</sub>O<sub>4</sub>NaS (M+Na<sup>+</sup>): 521.1254. Found: 521.1255.

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9. Crystallographic data (excluding structure factors) for the structure of 2-(*N*-4-chlorophenylsulfonyl-6-methylindol-3-yl)-3-*N*-acetyl-5-phenyl-1,3,4-oxadiazoline (**3h**) in this paper has been deposited with the Cambridge Crystallographic Data Centre as supplementary publication number CCDC 757955. Copies of the data can be obtained, free of charge, on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [fax: +44 (0)1223 336033 or e-mail: deposit@ccdc.cam.ac.uk].
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