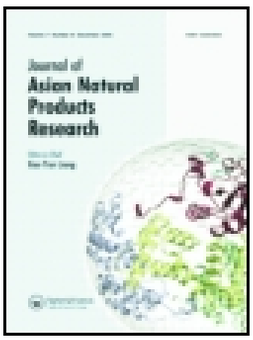

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Controlled synthesis of *N*, *N*-dimethylarylsulfonamide derivatives as nematicidal agents

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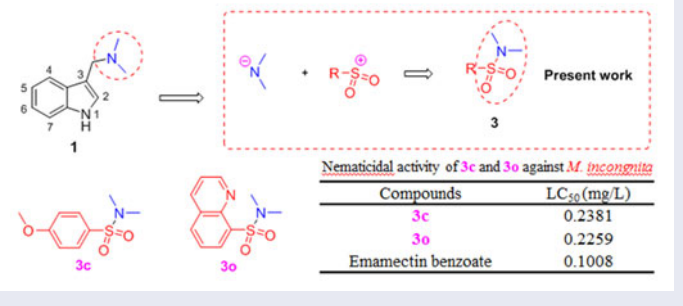
ABSTRACT

Gramine can be intelligently and efficiently supplied with *N*, *N*-dimethylamino group and then reacted with the corresponding sulfonyl chlorides to synthesize *N*, *N*-dimethylarylsulfonamides. We herein designed and controlled synthesis of *N*, *N*-dimethylarylsulfonamide derivatives, and first reported the results of the nematicidal activity of 15 title compounds **3a-o** against *Meloidogyne incongnita in vitro*, respectively. Among all of the title derivatives, compounds **3a**, **3c**, **3k**, and **3o** exhibited potent nematicidal activity with median lethal concentration (LC_{50}) values ranging from 0.22 to 0.26 mg/L. Most noteworthy, *N, N*-dimethyl-4-methoxyphenylsulfonamide (**3c**) and *N, N*-dimethyl-8-quinolinesulfonamide (**3o**) showed the best promising and pronounced nematicidal activity, with LC_{50} values of 0.2381 and 0.2259 mg/L, respectively.



Nematicidal activity of 3c and 3o against *M. incongnita*

Compounds	LC_{50} (mg/L)
3c	0.2381
3o	0.2259
Emamectin benzoate	0.1008

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Gramine; N; N-dimethylarylsulfonamides; nematicidal activity; *Meloidogyne incongnita*

1. Introduction

Plant-parasitic nematode is one of the most important plant pathogen, which is responsible for serious losses every year. Globally, the annual crops losses caused by nematodes were as high as 14%, resulting in economic losses of $80–100 billion [1]. Southern root-knot nematode (*Meloidogyne incongnita* (Kofold & White) Chitwood), a typical parasitic pest, is widely distributed in the world [2]. Moreover, at present,

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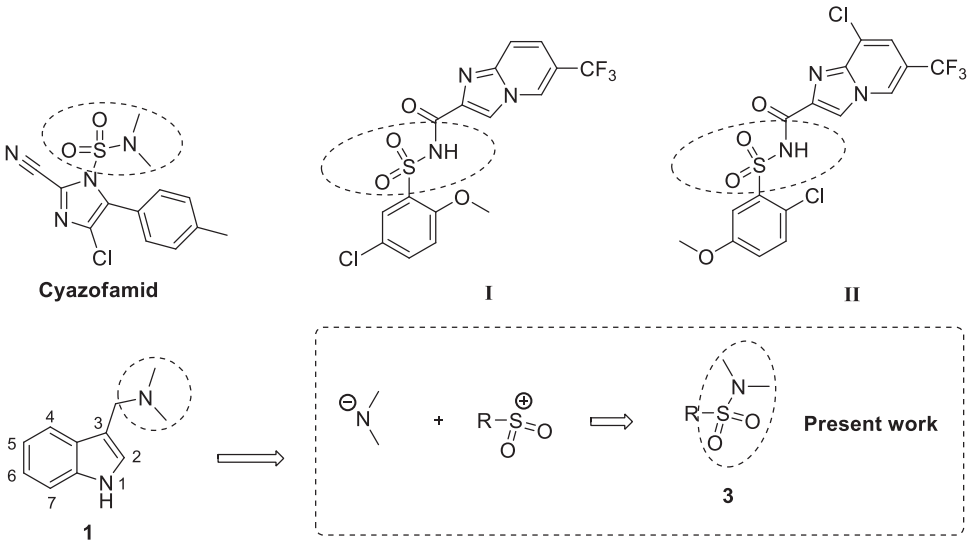


Figure 1. Structures of cyazofamid, compounds **I** and **II**, gramine (**1**), and design of *N*, *N*-dimethylarylsulfonamide derivatives (**3**).

there are only a few commercial nematicides left in use, and their repeated applications over the years have led to the development of resistance [3–6]. Therefore, to prevent southern root-knot nematode and overcome the problems of resistance development and environmental pollution, the research and development of efficacious nematicidal agents has received much attention internationally in recent years [7–11].

To the best of our knowledge, sulfonamides (SAs) show a variety of biological activities including nematicidal activity [12,13], anti-oomycete activity [1], anticonvulsant activity [14], antibacterial activity [15], antifungal activity [16], cardiac myosin activator [17], and 5-HT_6 receptor antagonists [18]. In addition, SAs are widely used as pharmaceuticals in pesticide. For example, cyazofamid, whose structural formula is shown in Figure 1, containing *N*, *N*-dimethylsulfonamide group, is an imidazole fungicide for use on food crops. As shown in Figure 1, Dupont patents protect the nematicidal activity of compounds **I** and **II** in 2010 and 2012, respectively [12,13]. Furthermore, compound **I** displayed the promising nematicidal activity at a concentration of 250 mg/L *in vivo*. Especially, compound **II** exhibited the best promising nematicidal activity, the mortality rate was higher than 50% against the southern root-knot nematode, *M. incognita*, *in vivo* at a concentration of 50 mg/L.

Generally, there are two ways to synthesize *N*, *N*-dimethylarylsulfonamides. The first kind is usually prepare the intermediate of SAs, and then the SAs reacts with iodomethane to form *N*, *N*-dimethylarylsulfonamides [17]. The second is the direct reaction of sulfonyl chlorides with dimethylamine to prepare *N*, *N*-dimethylarylsulfonamides. Of course, this process can also be assisted by ultrasonic irradiation [18,19]. It is well-known that the traditional procedure for the synthesis of *N*, *N*-dimethylarylsulfonamides often requires rigorous reaction conditions, and generally gives lower to moderate yields. Interestingly, gramine (**1**, 3-(dimethylaminomethyl)indole; Figure 1), a commercial natural metabolite, can be intelligently and efficiently supplied with *N*, *N*-dimethylamino

group, and then reacted with the corresponding sulfonyl chlorides to synthesize *N*, *N*-dimethylarylsulfonamides.

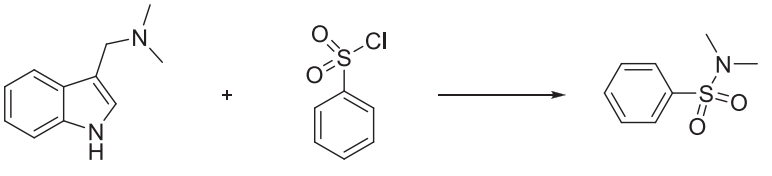
Inspired by the above-mentioned interesting results, and the aim in our continuing endeavor to find more active nematicidal hits [7,8], we herein designed and controlled synthesis of *N*, *N*-dimethylarylsulfonamide derivatives (**3,** Figure 1), and first report the results of the nematicidal activity of 15 *N*, *N*-dimethylarylsulfonamide derivatives against *M. incongnita in vitro*, respectively. Additionally, their structure–activity relationship (SAR) studies were also described.

2. Results and discussion

2.1. Chemistry

To find out the most compatible reaction conditions for synthesizing *N*, *N*-dimethylarylsulfonamide derivatives, a wide range of reaction parameters were tested by altering the base as well as the temperature and the solvent in a test reaction of gramine (**1**) with benzenesulfonyl chloride (**2a**) (Scheme 1).

As outlined in Table 1, the reaction catalyzed by benzyltriethylaminium·chloride (TEBA) was found to be sluggish (entries 1–3). When 1.0 mmol of **1** and 1.2 mmol of **2a** reacted at 25 °C in the presence of 1.8 mmol of sodium hydroxide (NaOH) in a dry dichloromethane (CH_2Cl_2) solution, *N*, *N*-dimethylphenylsulfonamide (**3a**) was not obtained (entry 1). When the temperature was lowered to 0 °C or −15 °C, **3a** was obtained in a trace or 29% yields even if the reaction time was prolonged to 24 h, respectively (entries 1 and 2). To our delight, this process proves that the reaction we originally designed can be achieved. Subsequently, the effect of different bases, temperatures, and solvents to the yield were also investigated. When the molar ratio between **2a** and **1** was 2, the reaction was stirred at 25 °C for 24 h in the presence of 3.0 mmol of potassium carbonate (K_2CO_3) in different solvents, the corresponding yields of **3a** were 0%, trace, and 63%, respectively (entries 4–6). It was preliminarily proved that the different solvents had a greater influence on the yield of **3a**, and the solvent acetonitrile (CH_3CN) (entry 6) was found to greatly enhance the reaction rate. Meanwhile, when 1.0 mmol of **1** and 2.0 mmol of **2a** reacted at reflux for 24 h in the presence of 4.5 mmol of triethylamine (Et_3N) in different solvents, the corresponding yields of **3a** were 32%, 51%, and 78%, respectively (entries 7–9). It was further proved that the base and the solvent were the two main factors affecting the yield of **3a**. Subsequently, the effect of molar ratio between **1**, **2a**, and base to the yield of **3a** was also investigated (entries 10–12). When 1.0 mmol of **1** and 1.2 mmol



1 **2a** **3a**

Scheme 1. Model reaction.

Table 1. Optimization of the reaction conditions.

	Amount of (mmol)						
Entry	1	2a	Base (mmol)	*T* (°C)	*t* (h)	Solvent	Yield of **3a** (%)
1[a]	1.0	1.2	NaOH (1.8)	25	24	CH_2Cl_2	0
2[a]	1.0	1.2	NaOH (1.8)	0	24	CH_2Cl_2	Trace
3[a]	1.0	1.2	NaOH (1.8)	−15	24	CH_2Cl_2	29
4	1.0	2.0	K_2CO_3 (3.0)	25	24	CH_3COCH_3	0
5	1.0	2.0	K_2CO_3 (3.0)	25	24	CH_2Cl_2	Trace
6	1.0	2.0	K_2CO_3 (3.0)	25	24	CH_3CN	63
7	1.0	2.0	Et_3N (4.5)	56	24	CH_3COCH_3	32
8	1.0	2.0	Et_3N (4.5)	40	24	CH_2Cl_2	51
9	1.0	2.0	Et_3N (4.5)	80	24	CH_3CN	78
10	1.0	1.2	Et_3N (3.0)	25	24	CH_3CN	75
11	1.0	1.2	Et_3N (1.5)	25	24	CH_3CN	73
12	1.0	1.5	Et_3N (1.5)	25	24	CH_3CN	79

[a]Catalyzed by TEBA (0.1 mmol).

of **2a** reacted at 25 °C for 24 h in the presence of 3.0 mmol of Et_3N in a dry CH_3CN solution, **3a** was obtained in a 75% yield (entry 10). When the amount of Et_3N was reduced by half, **3a** was obtained in a 73% yield (entry 11). When 1.0 mmol of **1** and 1.5 mmol of **2a** reacted at 25 °C for 24 h in the presence of 1.5 mmol of Et_3N in a dry CH_3CN solution, **3a** was obtained in a 79% yield (entry 12). Based on the amount of **2a**, base, temperatures, and solvents screened, evidently, the optimized reaction condition was the reaction of 1.0 mmol of **1** with 1.5 mmol of **2a** at 25 °C for 24 h in the presence of 1.5 mmol of Et_3N in a dry CH_3CN solution.

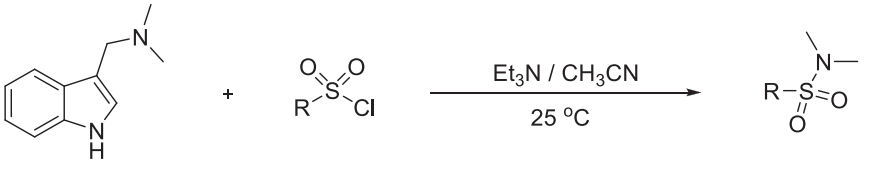
Based upon the above findings, subsequently, a wide range of arylsulfonyl chlorides (**2**, *R* = (*p*-Me)Ph, (*p*-OMe)Ph, (*p*-tert-butyl)Ph, (2,4,6-trimethyl)Ph, (2,4,6-triisopropyl)Ph, (*p*-F)Ph, (*p*-Br)Ph, (*o*-NO_2)Ph, (*m*-NO_2)Ph, (*p*-NO_2)Ph, (*p*-Cl, *m*-NO_2)Ph, 2-thienyl, 1-naphthyl, or 8-quinolyl), and gramine (**1**) were investigated to explore the scope of the reaction. As outlined in Table 2, *N, N*-dimethylarylsulfonamide derivatives (**3b-o**) were prepared in 76%–98% yields for 24 h. The steric and electronic effects of substituents of **2a** to the reaction were not very obvious. Compared with the traditional methods, the main advantage of the present procedure is milder conditions and better yields.

2.2. Biological activities

Fifteen *N*, *N*-dimethylarylsulfonamide derivatives **3a-o** and emamectin benzoate (used as a positive control) were screened *in vitro* for their nematicidal activities against *M. incongnita*. As shown in Table 3, among all of the title derivatives, compounds **3a**, **3c**, **3k**, and **3o** exhibited potent nematicidal activity with median lethal concentration (LC_{50}) values ranging from 0.22 to 0.26 mg/L. Most noteworthy, *N, N*-dimethyl-4-methoxyphenylsulfonamide (**3c**) and *N, N*-dimethyl-8-quinolinesulfonamide (**3o**) showed the best promising and pronounced nematicidal activity, with LC_{50} values of 0.2381 and 0.2259 mg/L, respectively.

Meanwhile, some interesting results of the SARs of **3a-o** were also observed. (1) Among *N*, *N*-dimethylarylsulfonamide derivatives (**3a-d**), compounds **3a** and **3c** exhibited the most potent nematicidal activity, but when the 4-methyl or the 4-tert-butyl group was introduced on the phenyl ring of **3a**, the nematicidal activity of the

Table 2. Synthesis of *N*, *N*-dimethylarylsulfonamide derivatives (**3b-o**).



1 **2b-o** **3b-o**

Entry	Gramine (**1**)	Arylsulfonyl chlorides (**2**)	*N*,*N*-Dimethylarylsulfonamides (**3**)	Yield (%)[a]
1	**1**	**2b**	**3b**	88
2	**1**	**2c**	**3c**	98
3	**1**	**2d**	**3d**	86
4	**1**	**2e**	**3e**	89
5	**1**	**2f**	**3f**	76
6	**1**	**2g**	**3g**	82
7	**1**	**2h**	**3h**	89
8	**1**	**2i**	**3i**	83
9	**1**	**2j**	**3j**	96
10	**1**	**2k**	**3k**	86
11	**1**	**2l**	**3l**	84
12	**1**	**2m**	**3m**	87
13	**1**	**2n**	**3n**	90
14	**1**	**2o**	**3o**	82

[a]Isolated yields.

Table 3. Nematicidal activity of *N*, *N*-dimethylarylsulfonamide derivatives **3a-o** against *M. incongnita*.

Compounds	Toxicity regression equation	Correlation coefficient	LC_{50} (mg/L)
3a	$y = 10.6256 + 9.3290x$	0.9961	0.2494
3b	$y = 7.5499 + 5.7516x$	0.9968	0.3603
3c	$y = 10.3300 + 8.5530x$	0.9861	0.2381
3d	$y = 7.3212 + 4.5467x$	0.9805	0.3087
3e	$y = 7.2335 + 4.8446x$	0.9824	0.3459
3f	$y = 7.2476 + 4.7251x$	0.9959	0.3344
3g	$y = 7.5218 + 4.7958x$	0.9970	0.2980
3h	$y = 7.8832 + 5.2788x$	0.9967	0.2843
3i	$y = 7.4962 + 4.8661x$	0.9813	0.3069
3j	$y = 8.9788 + 7.0711x$	0.9827	0.2737
3k	$y = 10.1810 + 8.8113x$	0.9915	0.2582
3l	$y = 6.5731 + 3.4350x$	0.9777	0.3484
3m	$y = 7.6271 + 4.7300x$	0.9946	0.2783
3n	$y = 7.6170 + 4.8484x$	0.9841	0.2886
3o	$y = 9.9566 + 7.6712x$	0.9861	0.2259
Emamectin benzoate	$y = 7.0620 + 2.0687x$	0.9750	0.1008

corresponding compounds were reduced sharply (**3a** vs. **3b** and **3d**). Interestingly, when the 4-methoxy group was introduced on the phenyl ring of **3a**, the nematicidal activity of the corresponding compound was enhanced (**3a** vs. **3c**). The LC_{50} values of **3a-d** against *M. incongnita* were 0.2494, 0.3603, 0.2381, and 0.3087 mg/L, respectively. (2) When $R = 2,4,6$-trimethylphenyl or $R = 2,4,6$-triisopropylphenyl, the nematicidal activity was decreased dramatically compared with **3a** (**3a** vs. **3e** and **3f**). (3) *N*, *N*-Dimethylphenylsulfonamide (**3a**) displayed potent nematicidal activity with LC_{50} value of 0.2494 mg/L, but when the chloro or the bromo atom was introduced on the phenyl ring of **3a**, the nematicidal activity of the corresponding compounds was decreased significantly (**3a** vs. **3g** and **3 h**). (4) We found that the R = nitrophenylsulfonyl, and the nitro group at different positions of the benzene ring, could lead to derivatives with different nematicidal activity (**3k** vs. **3j** and **3i**). For example, the LC_{50} values of **3i-k** against *M. incongnita* were 0.3069, 0.2737, and 0.2582 mg/L, respectively. (5) It is interesting that the $R = 4$-chloro-3-nitrophenylsulfonyl as a two-electron-withdrawing substituent (such as NO_2 and Cl) could result in weaker compound **3l** relative to those containing phenylsulfonyl as a one-electron-withdrawing substituent (e.g. **3g-k**, the LC_{50} values ranging from 0.2582 to 0.3069 mg/L). (6) The introduction of the sulfonyl containing heterocyclic ring moieties can significantly improve the nematicidal activity. As compared to 2-thiophenesulfonyl and 1-naphthalensulyonyl derivatives, 8-quinolinesulfonyl derivative displayed the best promising and pronounced nematicidal activity (**3m** and **3n** vs. **3o**). For example, the LC_{50} values of **3m-o** against *M. incongnita* were 0.2783, 0.2886, and 0.2259 mg/L, respectively.

3. Experimental

3.1. General experimental procedures

Melting points were taken on a X-6 microscopic melting point apparatus (Beijing Tech instrument Co., Ltd., Beijing, China) and are uncorrected. Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker Avance DMX 400 MHz instrument (Bruker Daltonik, Bremen, Germany) in $CDCl_3$ (^1H at 400 MHz) using TMS

(tetramethylsilane) as the internal standard. Electrospray ion trap mass spectrometry (ESI-TRAP-MS) was carried out with a Bruker ESI-TRAP Esquire 6000 plus mass spectrometry instrument (Bruker, Germany). Gramine and arylsulfonyl chloride were purchased from Aladdin Chemistry Co., Ltd. (Shanghai, China). Ethyl acetate, CH_2Cl_2, and petroleum ether were purchased from Beichen Fangzheng Reagent Factory (Tianjin, China). Analytical thin-layer chromatography (TLC) was performed with silica gel plate using silica gel 60 GF_{254} (Qingdao Haiyang Chemical Co., Ltd., Shandong, China). Silica gel column chromatography was performed with silica gel 200–300 mesh (Qingdao Haiyang Chemical Co., Ltd., Shandong, China).

3.2. Preparation of N, N-dimethylarylsulfonamide derivatives (3a-o)

To a solution of gramine (**1**, 1.0 mmol) and arylsulfonyl chloride (**2**, 1.5 mmol) in dry CH_3CN (10 ml) at 25 °C, a solution of Et_3N (1.5 mmol) in dry CH_3CN (5 ml) was added drop wise for 10 min [20–24]. After reaction for 24 h, the reaction solution was concentrated under reduced pressure to give crude product. The crude product was dissolved in CH_2Cl_2 (15 ml) and diluted with water (15 ml) and extracted with CH_2Cl_2 (30 ml × 3). Subsequently, the combined organic phase was washed by saturated aq. brine (30 ml), dried over anhydrous Na_2SO_4, concentrated *in vacuo*, and purified by silica gel column chromatography to obtain the target compounds in 76%–98% yields. The data for **3a-o** are shown as follows.

3.2.1. Data for N, N-dimethylphenylsulfonamide (3a)

Yield = 79%, Yellow oily liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.77–7.80 (m, 2 H), 7.53–7.63 (m, 3 H), 2.71 (s, 6 H). HRESIMS: *m/z* 186.0585 $[M+H]^+$ (calcd for $C_8H_{12}NO_2S$, 186.0583).

3.2.2. Data for N, N-dimethyl-4-methylphenylsulfonamide (3 b)

Yield = 88%, Pale yellow solid, m.p. 79–80 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.67 (d, J = 8.0 Hz, 2 H), 7.34 (d, J = 8.0 Hz, 2 H), 2.69 (s, 6 H), 2.44 (s, 3 H). HRESIMS: *m/z* 200.0745 $[M+H]^+$ (calcd for $C_9H_{14}NO_2S$, 200.0740).

3.2.3. Data for N, N-dimethyl-4-methoxyphenylsulfonamide (3c)

Yield = 98%, Brown solid, m.p. 73–74 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.70–7.73 (m, 2 H), 6.99–7.03 (m, 2 H), 3.88 (s, 3 H), 2.68 (s, 6 H). HRESIMS: *m/z* 216.0690 $[M+H]^+$ (calcd for $C_9H_{14}NO_3S$, 216.0689).

3.2.4. Data for N, N-dimethyl-4-tert-butylphenylsulfonamide (3d)

Yield = 86%, White solid, m.p. 118–119 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.68–7.71 (m, 2 H), 7.52–7.56 (m, 2 H), 2.71 (s, 6 H), 1.35 (s, 9 H). HRESIMS: *m/z* 242.1211 $[M+H]^+$ (calcd for $C_{12}H_{20}NO_2S$, 242.1209).

3.2.5. Data for N, N-dimethyl-2,4,6-trimethylsulfonamide (3e)

Yield = 89%, Yellow oily liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 6.94–6.95 (m, 2 H), 2.73 (s, 6 H), 2.61 (s, 6 H), 2.30 (s, 3 H). HRESIMS: *m/z* 228.1055 $[M+H]^+$ (calcd for $C_{11}H_{18}NO_2S$, 228.1053).

3.2.6. Data for N, N-dimethyl-2,4,6-triisopropylsulfonamide (3f)

Yield = 76%, Pale yellow solid, m.p. 122–123 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.16 (s, 2 H), 4.14–4.20 (m, 2 H), 2.86–2.93 (m, 1 H), 2.75 (s, 6 H), 1.23–1.26 (m, 18 H). HRESIMS: *m/z* 312.1995 $[M+H]^+$ (calcd for $C_{17}H_{30}NO_2S$, 312.1992).

3.2.7. Data for N, N-dimethyl-4-fluorophenylsulfonamide (3 g)

Yield = 82%, Brown solid, m.p. 75–76 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.77–7.82 (m, 2 H), 7.20–7.27 (m, 2 H), 2.71 (s, 6 H). HRESIMS: *m/z* 204.0490 $[M+H]^+$ (calcd for $C_8H_{11}FNO_2S$, 204.0489).

3.2.8. Data for N, N-dimethyl-4-bromophenylsulfonamide (3 h)

Yield = 89%, White solid, m.p. 91–92 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.67–7.70 (m, 2 H), 7.62–7.65 (m, 2 H), 2.71 (s, 6 H). HRESIMS: *m/z* 263.9693 $[M+H]^+$ (calcd for $C_8H_{11}BrNO_2S$, 263.9688).

3.2.9. Data for N, N-dimethyl-2-nitrophenylsulfonamide (3i)

Yield = 83%, Yellow oily liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.94–7.99 (m, 1 H), 7.67–7.74 (m, 2 H), 7.60–7.63 (m, 1 H), 2.91 (s, 6 H). HRESIMS: *m/z* 231.0435 $[M+H]^+$ (calcd for $C_8H_{11}N_2O_4S$, 231.0434).

3.2.10. Data for N, N-dimethyl-3-nitrophenylsulfonamide (3j)

Yield = 96%, Pale yellow solid, m.p. 121–122 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.62 (t, J = 2.0 Hz, 1 H), 8.46–8.48 (m, 1 H), 8.10–8.13 (m, 1 H), 7.77–7.81 (m, 1 H), 2.79 (s, 6 H). HRESIMS: *m/z* 231.0431 $[M+H]^+$ (calcd for $C_8H_{11}N_2O_4S$, 231.0434).

3.2.11. Data for N, N-dimethyl-4-nitrophenylsulfonamide (3k)

Yield = 86%, Pale yellow solid, m.p. 175–176 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.38–8.42 (m, 2 H), 7.96–7.99 (m, 2 H), 2.78 (s, 6 H). HRESIMS: *m/z* 231.0437 $[M+H]^+$ (calcd for $C_8H_{11}N_2O_4S$, 231.0434).

3.2.12. Data for N, N-dimethyl-4-chloro-3-nitrophenylsulfonamide (3 l)

Yield = 84%, Violet solid, m.p. 102–103 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.25 (d, J = 2.0 Hz, 1 H), 7.92 (dd, J = 8.4 Hz, 2.0 Hz, 1 H), 7.77 (d, J = 8.4 Hz, 1 H), 2.80 (s, 6 H). HRESIMS: *m/z* 265.0048 $[M+H]^+$ (calcd for $C_8H_{10}ClN_2O_4S$, 265.0044).

3.2.13. Data for N, N-dimethyl-2-thiophenesulfonamide (3 m)

Yield = 87%, Brown solid, m.p. 67–68 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.63 (dd, J = 5.2 Hz, 1.2 Hz, 1 H), 7.56 (dd, J = 3.6 Hz, 1.2 Hz, 1 H), 7.17 (dd, J = 5.2 Hz, 4.0 Hz, 1 H), 2.75 (s, 6 H). HRESIMS: *m/z* 192.0148 $[M+H]^+$ (calcd for $C_6H_{10}NO_2S_2$, 192.0147).

3.2.14. Data for N, N-dimethyl-1-naphthalenesulfonamide (3n)

Yield = 90%, Yellow oily liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.76–8.79 (m, 1 H), 8.21 (dd, J = 7.2 Hz, 1.2 Hz, 1 H), 8.06–8.09 (m, 1 H), 7.92–7.94 (m, 1 H), 7.53–7.67 (m, 3 H), 2.82 (s, 6 H). HRESIMS: m/z 236.0745 $[M+H]^+$ (calcd for $C_{12}H_{14}NO_2S$, 236.0740).

3.2.15. Data for N, N-dimethyl-8-quinolinesulfonamide (3o)

Yield = 82%, Pale yellow solid, m.p. 130–131 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.08–9.10 (m, 1 H), 8.47–8.50 (m, 1 H), 8.26 (dd, J = 8.0 Hz, 1.6 Hz, 1 H), 8.05 (dd, J = 8.0 Hz, 1.2 Hz, 1 H), 7.61–7.65(m, 1 H), 7.54 (q, J = 4.4 Hz, 1 H), 2.99 (s, 6 H). HRESIMS: m/z 237.0689 $[M+H]^+$ (calcd for $C_{11}H_{13}N_2O_2S$, 237.0692).

3.3. Nematicidal activity of compounds 3a-o against M. incongnita

Fifteen *N*, *N*-dimethylarylsulfonamide derivatives (**3a-o**) were screened *in vitro* for their nematicidal activity against *M. incongnita*. The ranges of compounds **3a-o** and emamectin benzoate (used as a positive control) concentrations for the assays were defined in preliminary experiments. Dimethyl sulfoxide solution of compounds **3a-o** and emamectin benzoate, and the final concentrations of active ingredients (a.i.) in medium were the following: for compounds **3a-o** 0.20, 0.25, 0.30, 0.35, and 0.40 mg/L, for emamectin benzoate 0.050, 0.075, 0.100, 0.125, and 0.150 mg/L. Then 50 sterilized nematodes (second-stage juveniles (J_2) of *M. incongnita*) were transferred to a 60-mm-diameter watch-glass with 2 ml of the above solutions, and placed the watch-glass in Petri dishes and kept in an incubator at 25 °C for 12 h. The blank control group was prepared in the same way but lacked the tested compound. Three replicates in each trial were made and the independent experiment was repeated three times. The activities of five concentrations of the tested compounds were monitored under a microscope by recording the death rate of the tested nematodes. Nematodes that did not move when prodded with a needle were considered to be dead. The method for determining nematicidal activity was based on as described in our previous article [25]. The LC_{50} values of compounds **3a-o** and emamectin benzoate were calculated using the probit method.

Disclosure statement

No potential conflict of interest was reported by the authors.

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