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One-Step Synthesis and Regioselective Alkylation of Substituted 1*H*-Pyrazolo[4,3-*e*][1,2,4]triazine*

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One-pot reaction between 5-acyl-3-(methylsulfanyl)-1,2,4-triazine **(1)** or its oxime **(8)** with hydrazine hydrochloride afforded 3-methyl-5-(methylsulfanyl)-1*H*-pyrazolo[4,3-*e*][1,2,4]triazine **(4)** in good yield. Treatment of the latter with alkyl halides in the presence of base gave 1- or 2-alkyl derivatives of **4** depending on the reaction conditions.

Key words: nucleophilic substitution of hydrogen, 1*H*-pyrazolo[4,3-*e*][1,2,4]triazine, alkylation

O- and *N*-alkyl derivatives of pyrazolo[4,3-*e*][1,2,4]triazine isolated from the cultural fluids of *Pseudomonas fluorescens* and *Nostoc spongiaeforme* have received considerable attention, because they display antimicrobial and antitumor activity [2,3]. Although some methods for the preparation of substituted pyrazolo[4,3-*e*][1,2,4]triazines have been developed [4–6], there are no useful synthetic routes for the synthesis of *N*-alkyl derivatives of this heterocyclic ring system. Recently, we have reported a new approach to pyrazolo[4,3-*e*][1,2,4]triazine **(3)** (R^1 = Ph) by one-pot reaction between 5-acyl-1,2,4-triazines **(1)** and arylhydrazines **(2)** [7] (Scheme 1). The reaction most probably proceeds *via* the phenylhydrazone intermediate **(A)**, followed by acid-promoted ring closure involving nucleophilic attack of the phenylhydrazone nitrogen onto the C-6 carbon of 1,2,4-triazine ring to give the bicyclic intermediate **(B)**, that *via* an air oxidation gives compound **3** [8]. Interestingly, under the acidic conditions employed no competitive formation of the indole ring from hydrazone was observed. The formation of the pyrazolo[4,3-*e*][1,2,4]triazine seems to be favored due to the presence of the 1,2,4-triazine ring, which can be easily N-protonated becoming more reactive toward nucleophilic reagents [9].

This efficient one-step access to the pyrazolo[4,3-*e*][1,2,4]triazine **3** has prompted us to generalize the methodology to the synthesis of 1-unsubstituted derivatives of **3** (R^1 = H). The latter could be used as starting materials for the preparation of the corresponding *N*-alkyl derivatives. Thus we examined the synthesis of 3-methyl-5-(methylsulfanyl)-1*H*-pyrazolo[4,3-*e*][1,2,4]triazine **(4)**, and its reactions with several

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* see [1] in References.
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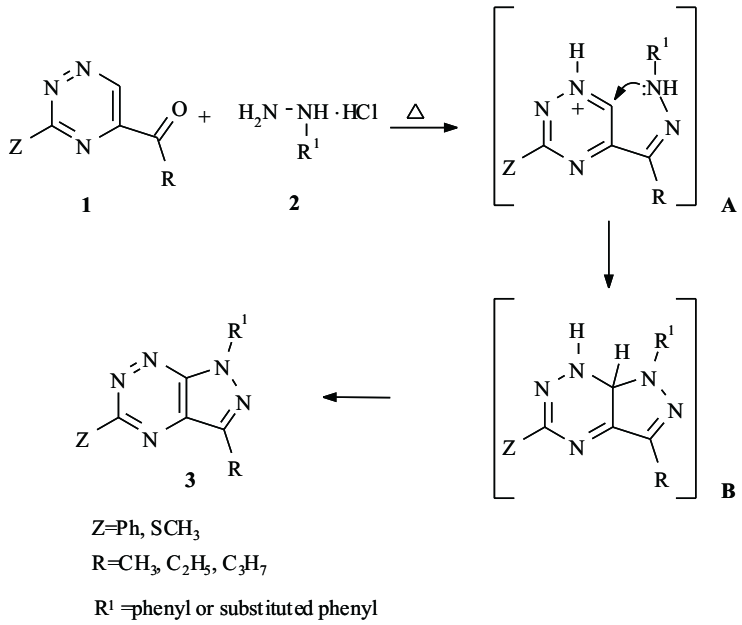
Scheme 1



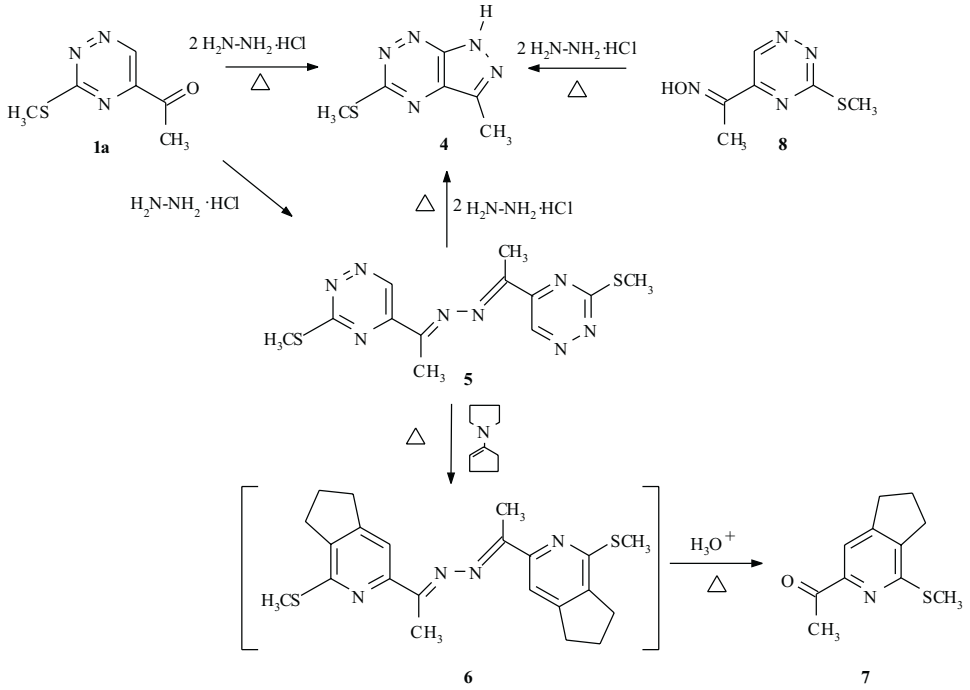
alkyl halides under basic conditions. Since methylsulfanyl substituent is good leaving group in 1,2,4-triazine and can be easily replaced by nucleophiles [10], its preparation would considerably broaden the scope of the pyrazolo[4,3-*e*][1,2,4]triazine synthesis.

RESULTS AND DISCUSSION

Compound **4** was conveniently prepared using three different synthetic procedures (Scheme 2). Treatment of 5-acetyl-3-(methylsulfanyl)-1,2,4-triazine **(1a)** with two equivalents of hydrazine hydrochloride in boiling ethanol for 1 h afforded compound **4** in 76% isolated yield. The structure assignment of **4** was based on its elemental analysis and exact mass measurements. Its IR spectrum showed the presence of the NH group and in ^{1}H-NMR spectrum no signal corresponding to H-6 proton in 1,2,4-triazine ring was observed.

To study the preparative scope of the reaction, the amount of hydrazine component was varied systematically. The reaction took a different course when one equivalent of hydrazine hydrochloride was used and the reaction was carried out at room temperature. In this case instead of **4**, azine **5** was formed as the only isolated product. The structure of **5** was confirmed by spectroscopic methods and by its chemical behaviour. Compound **5** could be easily converted into desired pyrazolo[4,3-*e*][1,2,4]triazine **(4)** by heating with an excess of hydrazine hydrochloride in ethanol. Treatment of **5** with 1-pyrrolidino-1-cyclopentene at 150°C afforded compound **6**, which was immediately converted into cyclopenta[c]pyridine **(7)**. This compound was identical with a com-

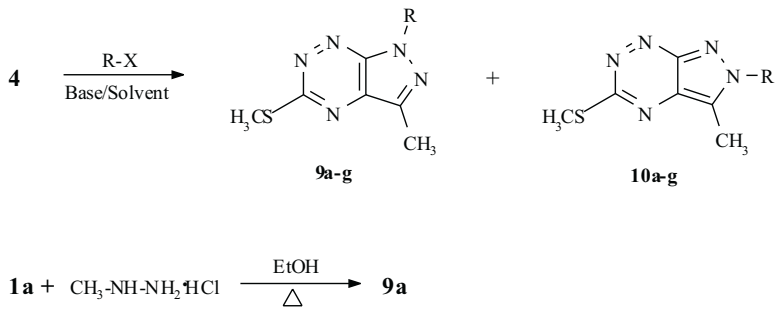
Scheme 2



pound prepared by Diels-Alder reaction between **1a** and 1-pyrrolidino-1-cyclopentene [11]. Compound **4** can be also formed from easily accessible 5-acetyl-3-(methylsulfanyl)-1,2,4-triazine oxime **8** [12] by heating with hydrazine hydrochloride.

To establish optimal conditions for alkylation, the reaction of compound **4** with methyl iodide was (Scheme 3) tested in various base-solvent systems (Table 1). In the most cases the methylation of **4** afforded predominantly 1,3-dimethyl derivative **9a** (R = CH_3), which was accompanied by varying amounts of 2,3-dimethyl isomer **10a** (R = CH_3). The highest yields of **9a** were obtained when alkylation was carried out in the presence of sodium hydride or potassium tert-butoxide in DMA at room tempera-

Scheme 3



ture (Table 1, entries 4 and 5). Under these conditions more than 70% of **9a** was formed. However, the reaction of **4** with methyl iodide in the presence of potassium carbonate in DMA or in diluted ethanol (*cf*. Table 1, entries 6 and 7) produced **10a** in 57% yield. With other alkylating agents: ethyl iodide, propyl- and benzyl chloride, butyl bromide, butenyl bromide and methyl chloroacetate the mixtures of 1- and 2-alkylated products were formed (Table 1, entries 8–13).

Table 1.

Entry	Compounds **9** and **10**	R-X	Base/Solvent	Reaction Time (h)	Yield **9+10** (%)	Ratio **9:10**
1	**a**	CH_3-I	NaH/THF	12	63	2:1
2	**a**	CH_3-I	NaH/DMF	12	96	2.1:1
3	**a**	CH_3-I	NaH/DMSO	12	85	3:1
4	**a**	CH_3-I	NaH/DMA	12	90	3.5:1
5	**a**	CH_3-I	t-BuOK/DMA	12	90	3.5:1
6	**a**	CH_3-I	K_2CO_3/DMA	12	95	1:1
7	**a**	CH_3-I	K_2CO_3/EtOH-H_2O (4:1)	8	96	1:1.3
8	**b**	C_2H_5-I	t-BuOK/DMA	12	90	2:1
9	**c**	n-C_3H_7-Cl	t-BuOK/DMA	48	44	3:1
10	**d**	n-C_4H_9-Br	t-BuOK/DMA	24	78	4:1
11	**e**	CH_2=$CHCH_2CH_2$-Br	t-BuOK/DMA	24	70	4:1
12	**f**	Ph-CH_2-Cl	t-BuOK/DMA	12	97	3:1
13	**g**	Cl-$CH_2CO_2CH_3$	t-BuOK/DMA	12	82	3:1

The structure of **9a** was confirmed by comparison of its spectroscopic data with those of the product independently prepared in reaction of methylhydrazine hydrochloride with **1a** in boiling ethanol (Scheme 3).

In summary, we report a new and efficient one-pot synthesis of 1*H*-pyrazolo[4,3-*e*][1,2,4]triazine derivative and its reaction with alkyl halides.

EXPERIMENTAL

All melting points are uncorrected. The ^{1}H NMR spectra were recorded in deuteriochloroform ($CDCl_3$) on a Varian Gemini 200 MHz spectrometer using tetramethylsilane (TMS) as the internal standard. IR spectra were measured with a Magna IR–760 spectrophotometer. Mass spectra were measured on AMD 604 spectrometer [electron impact (EI)] and API 350 [electrospray ionization (ESI)]. Elemental analyses were recorded on Perkin-Elmer 2400-CHN analyzer and the results for the indicated elements were within 0.3% of the calculated values. Merck 60F_{254} plates were used for analytical (TLC) chromatography and column chromatography was performed on silica gel (230–400 mesh, 60 Merck).

Synthesis of 3-methyl-5-(methylsulfanyl)-1*H*-pyrazolo[4,3-*e*][1,2,4]triazine (4). ***Method A***: To a solution of the ketone **1a** (1.69 g, 10 mmol) and hydrazine hydrochloride (1.36 g, 20 mmol) in 80 mL of ethanol was added 1 mL 10% aqueous hydrochloric acid. The mixture was heated at reflux for 1 hour and then the solvent was removed *in vacuo*. The solid was collected by filtration, washed with water and recrystallized from ethanol/water (1:1), to give compound **4** in 76% yield. M.p. 165°C; ^{1}H NMR ($CDCl_3$) δ: 2.67 (s, 3H), 2.74 (s, 3H), 11.05 (s, 1H); IR (KBr) cm^{-1}: 3100, 1400, 1330, 1170, 1150, 1080, 950, 760, 660; MS (EI, *m/z*, %): 181 (36) [M^+], 153 (98), 152 (49), 126 (41), 112 (46), 97 (41), 85 (58), 70 (100). Anal. Calcd. for $C_6H_7N_5S$: C, 39.77; H, 3.86; N, 38.67. Found: C, 39.93; H, 3.75; N, 38.45.

Method B: To a solution of the oxime **8** (184 mg, 1 mmol) and hydrazine hydrochloride (136 mg, 2 mmol) in 8 mL of ethanol was added 0.5 mL 37% hydrochloric acid. The mixture was heated at reflux for 1 hour and then the solvent was evaporated *in vacuo*. The solid was collected by filtration, washed with water and recrystallized from ethanol/water mixture (1:1) to give **4** in 28% yield.

Method C: To a solution of **5** (1.81 g, 10 mmol) and hydrazine hydrochloride (1.36 g, 20 mmol) in 80 mL of ethanol was added 1 mL 10% aqueous hydrochloric acid. The mixture was heated at reflux for 1 hour and then the solvent was evaporated under reduced presure. The solid was collected by filtration, washed with water and recrystallized from ethanol/water (1:1) to give compound **4** in 70% yield.

N,N-Bis-[1-(3-methylsulfanyl-5-[1,2,4]triazin-5-yl)-ethylidene]-hydrazine (5). To a solution of the ketone **1a** (1.69 g, 10 mmol) and hydrazine hydrochloride (0.68 g, 10 mmol) in 80 mL of ethanol was added 1 mL 10% aqueous hydrochloric acid. The mixture was stirred for 10 minutes and then the solvent was evaporated *in vacuo*. The solid was filtered, washed with water and recrystallized from ethanol/water (1:1) to give **5** in 78% yield. M.p. 175°C; ^{1}H NMR ($CDCl_3$) δ: 2.29 (s, 6H), 2.73 (s, 6H), 9.65 (s, 2H); IR (KBr) cm^{-1}: 1530, 1500, 1430, 1380, 1260, 1130, 1060, 980, 880, 730; MS (EI, *m/z*, %): 334 (1) [M^+], 291 (100), 190 (40). Anal. Calcd. for $C_{12}H_{14}N_8S_2$: C, 43.11; H, 4.19; N, 33.53. Found: C, 43.11; H, 4.18; N, 33.27.

3-Acetyl-5,6-dihydro-1-(methylsulfanyl)-7*H*-cyclopenta[c]pyridine (7). The mixture of **5** (0.334 g, 1 mmol) and 1-pyrrolidino-1-cyclopentene (1 mL) was heated at 150°C for 1 hour. The crude product was purified by column chromatography (silica gel, chloroform) to give 0.036 g (9%) of **7** as a white solid. M.p. 65–67°C; ^{1}H NMR ($CDCl_3$) δ: 2.15 (q, 2H, J = 7.5 Hz), 2.63 (s, 3H), 2.70 (s, 3H), 2.79 (t, 2H, J = 7.6 Hz), 2.94 (t, 2H, J = 7.6 Hz). HR-MS (ESI) *m/z* 230.0621 (M^+Na), calcd. for $C_{11}H_{13}NONaS$ (M^+Na) 230.0610.

1,3-Dimethyl-5-(methylsulfanyl)-1*H*-pyrazolo[4,3-*e*][1,2,4]triazine (9a). To a solution of 1a (1.69 g, 10 mmol) and methylhydrazine hydrochloride (1.23 g, 15 mmol) in 80 mL of ethanol was added 1 mL 10% aqueous hydrochloric acid. The mixture was heated at reflux for 1 hour and then the solvent was evaporated *in vacuo*. The solid was collected by filtration, washed with water and recrystallized from ethanol/water (1:1), to give compound **9a** in 72% yield.

General procedure for the alkylation of 4. A mixture of the corresponding base (20 mmol) and 3-methyl-5-(methylsulfanyl)-1*H*-pyrazolo[4,3-*e*][1,2,4]triazine **(4)** (1.81 g, 10 mmol) in an appropriate solvent (40 mL) was stirred for 10–15 min. at room temperature. Then, a solution of an alkyl halide (20 mmol) in this solvent (5 mL) was added dropwise and stirring was continued until the starting material disappeared (8–48 h, TLC control). An excess of base was then destroyed by addition of saturated solution of ammonium chloride and the solvent was removed *in vacuo*. The crude product was submitted to column chromatography on silica gel using chloroform/acetone (50:1) as eluent to give **9** as the first product. Further elution with chloroform/acetone (25:1) gives pure **10**. The following compounds were obtained.

1,3-Dimethyl-5-(methylsulfanyl)-1*H*-pyrazolo[4,3-*e*][1,2,4]triazine (9a). Yield 70%, yellow crystals. M.p. 123°C; ^{1}H NMR ($CDCl_3$) δ: 2.62 (s, 3H), 2.73 (s, 3H), 4.24 (s, 3H), MS (EI, *m/z*, %): 195 (20) [M^+], 152 (30), 70 (100). Anal. Calcd. for $C_7H_9N_5S$: C, 43.07; H, 4.61; N, 35.89. Found: C, 43.06; H, 4.62; N, 35.95.

1,2-Dimethyl-5-(methylsulfanyl)-1*H*-pyrazolo[4,3-*e*][1,2,4]triazine (10a). Yield 20%, yellow crystals. M.p. 173°C; ^{1}H NMR ($CDCl_3$) δ: 2.67 (s, 6H), 4.24 (s, 3H), MS (EI, *m/z*, %): 195 (4) [M^+], 167 (10), 134 (18), 56 (100). Anal. Calcd. for $C_7H_9N_5S$: C, 43.07; H, 4.61; N, 35.89. Found: C, 43.20; H, 4.52; N, 36.13.

1-Ethyl-3-methyl-5-(methylsulfanyl)-1*H*-pyrazolo[4,3-*e*][1,2,4]triazine (9b). Yield 60%, yellow crystals. M.p. 75°C; ^{1}H NMR ($CDCl_3$) δ: 1.59 (t, 3H, J = 7.2 Hz), 2.63 (s, 3H), 2.73 (s, 3H), 4.68 (q, 2H, J = 7.2 Hz); MS (EI, *m/z*, %): 209 (21) [M^+], 181 (80), 153 (84), 97 (56), 70 (100). Anal. Calcd. for $C_8H_{11}N_5S$: C, 45.93; H, 5.26; N, 33.49. Found: C, 46.09; H, 5.15; N, 33.23.

2-Ethyl-3-methyl-5-(methylsulfanyl)-1*H*-pyrazolo[4,3-*e*][1,2,4]triazine (10b). Yield 30%, yellow crystals. M.p. 168°C; ^{1}H NMR ($CDCl_3$) δ: 1.64 (t, 3H, J = 7.3 Hz), 2.60 (s, 3H), 2.69 (s, 3H), 4.55 (q, 2H, J = 7.3 Hz); MS (EI, *m/z*, %): 209 (20) [M^+], 181 (78), 166 (26), 148 (56), 120 (88), 108 (40), 70 (100). Anal. Calcd. for $C_8H_{11}N_5S$: C, 45.93; H, 5.26; N, 33.49. Found: C, 46.00; H, 5.25; N, 33.37.

3-Methyl-5-(methylsulfanyl)-1-propyl-1*H*-pyrazolo[4,3-*e*][1,2,4]triazine (9c). Yield 33%, yellow crystals. M.p. 53°C; ^{1}H NMR ($CDCl_3$) δ: 0.94 (t, 3H, J = 7.4 Hz), 1.94–2.12 (m, 2H), 2.63 (s, 3H), 2.73 (s, 3H), 4.57 (t, 2H, J = 7.1 Hz); MS (EI, *m/z*, %): 223 (28) [M^+], 180 (48), 70 (100); HRMS (ESI): *m/z* 224.0963, calcd. for $C_9H_{14}N_5S$ (M^+H) 224.0964.

3-Methyl-5-(methylsulfanyl)-2-propyl-1*H*-pyrazolo[4,3-*e*][1,2,4]triazine (10c). Yield 11%, yellow crystals. M.p. 137°C; ^{1}H NMR ($CDCl_3$) δ: 1.00 (t, 3H, J = 7.4 Hz), 1.99–2.17 (m, 2H), 2.68 (s, 6H), 4.43 (t, 2H, J = 7.1 Hz); MS (EI, *m/z*, %): 223 (4) [M^+], 195 (40), 43 (100). Anal. Calcd. for $C_9H_{13}N_5S$: C, 48.43; H, 5.82; N, 31.39. Found: C, 48.30; H, 5.69; N, 31.20.

1-Butyl-3-methyl-5-(methylsulfanyl)-1*H*-pyrazolo[4,3-*e*][1,2,4]triazine (9d). Yield 62%, yellow crystals. M.p. 42°C; ^{1}H NMR ($CDCl_3$) δ: 0.95 (t, 3H, J = 7.2 Hz), 1.25–1.44 (m, 2H), 1.91–2.05 (m, 2H), 2.63 (s, 3H), 2.73 (s, 3H), 4.60 (t, 2H, J = 7.1 Hz); MS (EI, *m/z*, %): 237 (32) [M^+], 194 (100), 152 (98), 70 (30), 55 (46). Anal. Calcd. for $C_{10}H_{15}N_5S$: C, 50.63; H, 6.32; N, 29.53. Found: C, 50.86; H, 6.29; N, 29.56.

2-Butyl-3-methyl-5-(methylsulfanyl)-1*H*-pyrazolo[4,3-*e*][1,2,4]triazine (10d). Yield 16%, yellow crystals. M.p. 76°C; ^{1}H NMR ($CDCl_3$) δ: 0.99 (t, 3H, J = 7.3 Hz), 1.32–1.50 (m, 2H), 1.95–2.10 (m, 2H), 2.68 (s, 6H), 4.47 (t, 2H, J = 7.3 Hz); MS (EI, *m/z*, %): 237 (4) [M^+], 209 (32), 180 (30), 120 (100), 57 (60). Anal. Calcd. for $C_{10}H_{15}N_5S$: C, 50.63; H, 6.32; N, 29.53. Found: C, 50.80; H, 6.35; N, 29.37.

1-(3-Buten)-3-methyl-5-(methylsulfanyl)-1*H*-pyrazolo[4,3-*e*][1,2,4]triazine (9e). Yield 56%, yellow crystals. M.p. 41°C; ^{1}H NMR ($CDCl_3$) δ: 2.63 (s, 3H), 2.73 (s, 3H), 2.71–2.82 (m, 2H), 4.67 (t, 2H, J = 7.0 Hz), 4.95–5.09 (m, 2H), 5.70–5.90 (m, 1H); MS (EI, *m/z*, %): 235 (42) [M^+], 192 (100), 160 (44), 70 (48), 55 (88); HRMS (ESI): *m/z* 236.0965, calcd. for $C_{10}H_{14}N_5S$ (M+H) 236.0964.

2-(3-Buten)-3-methyl-5-(methylsulfanyl)-1*H*-pyrazolo[4,3-*e*][1,2,4]triazine (10e). Yield 14%, yellow crystals. M.p. 85°C; ^{1}H NMR ($CDCl_3$) δ: 2.68 (s, 3H), 2.69 (s, 3H), 2.76–2.86 (m, 2H), 4.53 (t, 2H, J = 7.0 Hz), 5.02–5.12 (m, 2H), 5.68–5.88 (m, 1H); MS (EI, *m/z*, %): 235 (4) [M^+], 207 (24), 120 (56), 55 (100). Anal. Calcd. for $C_{10}H_{13}N_5S$: C, 51.06; H, 5.53; N, 29.78. Found: C, 51.08; H, 5.50; N, 29.70.

1-Benzyl-3-methyl-5-(methylsulfanyl)-1*H*-pyrazolo[4,3-*e*][1,2,4]triazine (9f). Yield 72%, yellow crystals. M.p. 115°C; ^{1}H NMR ($CDCl_3$) δ: 2.62 (s, 3H), 2.73 (s, 3H), 5.75 (s, 2H), 7.29–7.45 (m, 5H); MS (EI, *m/z*, %): 271 (12) [M^+], 242 (68), 91 (100). Anal. Calcd. for $C_{13}H_{13}N_5S$: C, 57.56; H, 4.79; N, 25.83. Found: C, 57.51; H, 4.71; N, 25.79.

2-Benzyl-3-methyl-5-(methylsulfanyl)-1*H*-pyrazolo[4,3-*e*][1,2,4]triazine (10f). Yield 25%, yellow crystals. M.p. 177°C; ^{1}H NMR ($CDCl_3$) δ: 2.60 (s, 3H), 2.66 (s, 3H), 5.69 (s, 2H), 7.23–7.37 (m, 5H); MS (EI, *m/z*, %): 271 (2) [M^+], 91 (100), 65 (52). Anal. Calcd. for $C_{13}H_{13}N_5S$: C, 57.56; H, 4.79; N, 25.83. Found: C, 57.53; H, 4.80; N, 25.79.

1-(Methoxycarbonylmethyl)-3-methyl-5-(methylsulfanyl)-1*H*-pyrazolo[4,3-*e*][1,2,4]-triazine (9g). Yield 62%, yellow crystals. M.p. 109°C; ^{1}H NMR ($CDCl_3$) δ: 2.65 (s, 3H), 2.73 (s, 3H), 3.77 (s, 3H), 5.37 (s, 2H); MS (EI, *m/z*, %): 253 (24) [M^+], 225 (46), 152 (92), 125 (64), 70 (90), 45 (100). Anal. Calcd. for $C_9H_{11}N_5SO_2$: C, 42.68; H, 4.34; N, 27.66. Found: C, 42.78; H, 4.23; N, 27.69.

2-(Methoxycarbonylmethyl)-3-methyl-5-(methylsulfanyl)-1*H*-pyrazolo[4,3-*e*][1,2,4]-triazine (10g). Yield 20%, yellow crystals. M.p. 149°C; ^{1}H NMR ($CDCl_3$) δ: 2.66 (s, 3H), 2.68 (s, 3H), 3.84 (s, 3H), 5.29 (s, 2H); MS (EI, *m/z*, %): 253 (2) [M^+], 225 (22), 121 (20), 45 (100). Anal. Calcd. for $C_9H_{11}N_5SO_2 \cdot 0.25H_2O$: C, 41.94; H, 4.27; N, 27.18. Found: C, 41.82; H, 4.12; N, 27.18.

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