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A Facile Synthesis of 3,5-Diaryl 1,2,4-Selenadiazoles from Aryl Selenoamides

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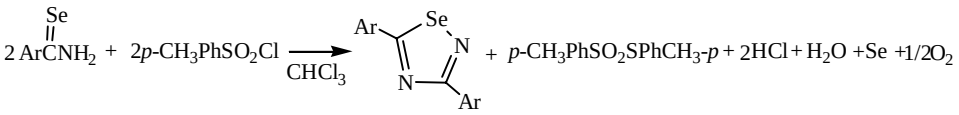
Abstract: A facile synthesis of 3,5-diaryl 1,2,4-selenadiazoles was provided by treatment of aryl selenoamides with *p*-methylphenyl sulfonyl chloride in chloroform.

Keywords: 3,5-Diaryl-1,2,4-selenadiazoles, aryl selenoamides, *p*-methylphenyl sulfonyl chloride, synthesis.

Aryl selenoamides are rather important intermediates in organic synthesis. They are not only useful for the synthesis of selenium–nitrogen heterocycles, but also can be expected to react with various organic or inorganic reagents owing to the high reactivity of their carbon-selenium double bond[1,2]. We have reported that primary selenoamides could be used as selenium transfer reagents in the synthesis of dialkyl diselenides from reactive halides[3] and diacyl selenides from acyl chlorides[4]. In order to investigate the reactivity of primary selenoamides towards sulfonyl chlorides, we added sulfonyl chlorides to the solution of primary selenoamides in chloroform at room temperature and expected that disulfonyl diselenides or disulfonyl selenides would produce by selenium transfer reaction of selenoamides. However, the expected products were not formed and 3,5-diaryl-1,2,4-selenadiazoles were afforded instead after the reaction mixture was stirred for 3 h (**Scheme 1**). Byproduct thiosulfonate **3** was also separated. The results are shown in **Table 1** and all the products were characterized by IR and ^{1}H NMR[5].

Scheme 1



1 **2** **3**

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In literature 3,5-diaryl 1,2,4-selenadiazoles have been reported to be synthesized by oxidation of primary selenoamides with I_2[6], treatment of selenoamides with N-bromosuccinimide[7], or reaction of selenoamides with -haloketones[8]. However, these methods have the disadvantages of being in low yields or at very low reaction temperature.

In summary, we provide a new and facile synthesis of 3,5-diaryl 1,2,4-selenadiazoles which has the advantage of easily available starting materials and mild reaction condition .

Typical Procedure: A solution of *p*-methylphenylsulfonyl chloride (1.2 mmol) in 10 mL of dry chloroform was added dropwise to a solution of selenobenzamide **1a** (1mmol) in 20 mL of dry chloroform at room temperature under nitrogen atomosphere, and stirred for 3 h (the end of the reaction was monitored by TLC). The reaction mixture was added 30 mL of water and continued stirring for 30 min. Then the organic layer was separated and washed successively with saturated solution of sodium carbonate (20 mL $\times$2) and water (20 mL), then dried over sodium sulfate and concentrated. The residue was purified by preparative TLC on silica gel (dichloromethane:cyclohexane=1:1 as an eluent) to give 3,5-diphenyl-1,2,4-selenadiazole **2a** in 62% yield.

Table 1 Synthesis of 3,5-diaryl 1,2,4-selenadiazoles

Entry	Ar	React time (h)	Isolated yield (%)	m. p. (°C)	Lit. m. p. (°C)
2a	C_6H_5	3	62	84-85	85[6]
2b	*p*-$CH_3C_6H_4$	3	67	121-122	122[6]
2c	*p*-ClC_6H_4	3	56	166-167	168[6]
2d	*p*-$CH_3OC_6H_4$	3	70	137-138	139[6]
2e	*p*-BrC_6H_4	3	51	161-162	162[6]

References and Notes

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5. ^{1}H NMR ($CDCl_3$,δ_H ppm) of producds: **2a**: 8.36-8.45 (m, 2H), 7.94-8.05 (m, 2H), 7.44-7.53 (m, 6H). **2b**: 8.27 (d, 2H, *J*=8.2Hz), 7.88 (d, 2H, *J*=8.0Hz), 7.26 (m, 4H), 2.42 (s, 6H). **2c**: 8.27 (d, 2H, *J*=8.2Hz), 7.87 (d, 2H, *J*=8.1Hz), 7.25 (d, 4H, *J*=8.1Hz). **2d**: 8.35 (d, 2H, *J*=8.2Hz,), 7.87 (d, 2H, *J*=8.1Hz), 6.98 (d, 2H, *J*=8.1Hz), 6.91(d, 2H, *J*=8.1Hz), 3.84 (s, 6H). **2e:** 8.29 (d, 2H, *J*=8.1Hz), 7.89 (d, 2H, *J*=8.0Hz), 7.59 (m, 4H).
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