# Palladium-Catalyzed Coupling of Oxazol-2-yl- and 2-Oxazolin-2-yltrimethylstannanes with Aromatic Halides. A New Entry to 2-Aryl and 2-Heteroaryl Oxazoles and Oxazolines

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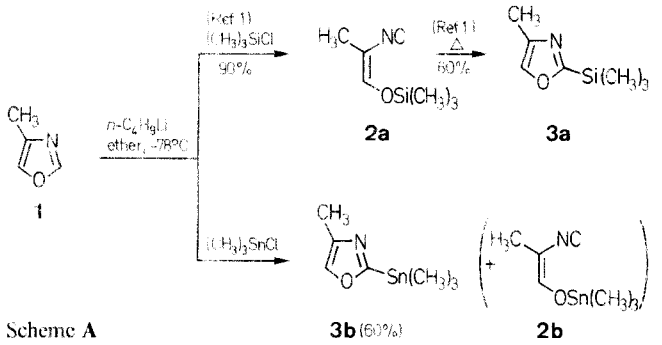
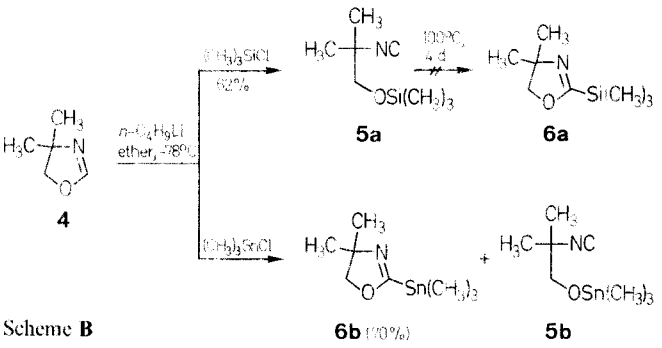
4-Methyloxazole and 4,4-dimethyl-2-oxazoline were treated with *n*-butyllithium and trimethyltin chloride to give the corresponding 2-trimethylstannyl derivatives which in the presence of tetrakis(triphenylphosphine)palladium(0) as a catalyst undergo cross-coupling reactions with various aryl and heteroaryl halides to give 2-aryl and 2-heteroaryl oxazoles and oxazolines in high yields.

We have reported[1] earlier the synthesis of various 2-trimethylsilyloxazoles, including the 4-methyl derivative **3a**, by isomerization of α-isocyano silyl enol ethers (Scheme **A**) and demonstrated their synthetic utility as stable 2-oxazolyl anion equivalents towards carbon and sulfur electrophiles. In particular, 2-silyloxalzoles proved to be quite useful as precursors to 2-acycloxazoles *via* reaction with acyl chlorides, thus overcoming the unsuccessful acylation of 2-lithio oxazoles due to the equilibration with the open-chain tautomers lithio α-isocyano enolates.[2] Unfortunately the same strategy appears unfeasible with 2*H* 2-oxazolines[3,4] because of the failure to isomerize[1] the silyloxyalkyl isocyanide **5a** into the silyloxazoline **6a** (Scheme **B**). Yet, in view of the extensive use of 2-oxazolines in synthesis,[5-7] the preparation of 2-substituted derivatives by different methods than those involving heterocyclic ring forming processes between carboxylic acids or nitriles and aminoalcohols,[3,5] are highly desirable. Following our studies on the metalation of azoles[1,8] and their use as auxiliaries in carbohydrate synthesis,[9] we would like to report here the preparation of a 2-stannyloxazole and a 2-stannyloxazoline[10] and their ready conversion into 2-aryl and 2-heteroaryl derivatives by palladium-catalyzed cross-coupling with various aromatic halides. The synthesis of a few 2-aryloxazoles and oxazolines by a transition-metal-catalyzed cross-coupling of Grignards with a 2-methylthiooxazole and oxazoline has been reported.[11]



Scheme **A**



Scheme **B**

Sequential lithiation with *n*-butyllithium of 4-methyloxazole (**1**) (Scheme **A**) and 4,4-dimethyl-2-oxazoline (**4**) (Scheme **B**) and quenching the resulting mixtures of open-chain and cyclic lithium salts[2,3] with trimethyltin chloride gave the corresponding 2-trimethylstannyloxazole **3b** and oxazoline **6b** which were isolated by distillation in 60–70% yield. The open-chain tautomer **5b** was detected through the IR absorption of the isonitrile group at 2120 $cm^{-1}$ in the crude reaction mixture whereas **2b** was not detected in a similar way. This indicates that in both cases trimethyltin chloride unlike the silyl counterpart reacts essentially with the heterocyclic carbanion rather than the open-chain oxy anion, a result which is in line with the lower affinity of tin than silicon for oxygen.[12] The stannyloxazole **3b** and the stannyloxazoline **6b** appeared to be moisture and air sensitive and therefore required handling and storage under Argon atmosphere.

The palladium-catalyzed cross-coupling between organotin reagents and organic halides is a versatile and well established method for carbon-carbon bond formation[13] which has been recently employed for the arylation of heterocycles.[14] Thus, treatment of aromatic and heterocyclic halides **7** with the stannyloxazole **3b** or the stannyloxazoline **6b** in benzene in the

presence of a catalytic amount of tetrakis(tetraphenylphosphine)palladium(0) gave the corresponding cross-coupling products namely 2-aryloxazoles **8** (Table 1) and 2-aryloxazolines **9** (Table 2) in very high yields with one exception only. In both cases successful reactions were obtained with various aromatic halides differing in the nature of the aromatic ring and their substituent, as well as the halogen. Hence the scope of these direct cross-coupling reactions appear, quite large and their synthetic utility can be foreseen as novel entries to 2-aryl oxazoles and oxazolines *directly* from aryl halides. This should be useful in case of difficult conversion of the latter into Grignards reagents, one of the partners in the Pridgen procedure,[11] and should become the method of choice for those heterocyclic systems such as pyridines, quinolines which fail to

**Table 1.** Reactions of 4-Methyl-2-trimethylstannyloxazole (**3b**) with Aryl Halides **7a, d, l, p** to give 2-Aryl-4-methyloxazole **8a, d, l, p.**

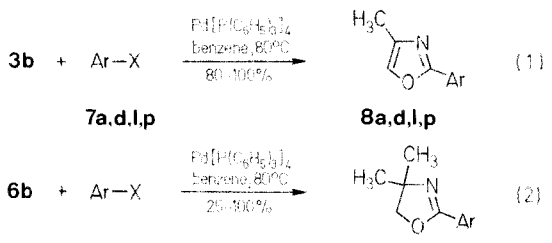
| Aryl Halide | X | Reaction Time (h) | Product | Yield (%) | m.p. (°C)[a] | Molecular Formula[b] or Lit. Data | $^1$H-NMR ($CDCl_3$/TMS)[c] $\delta$, $J$(Hz) | MS (70 eV)[d] $m/e$ ($M^+$) |
|---|---|---|---|---|---|---|---|---|
| **7a** | I | 24 | **8a** | 80 | oil | hydrobromide[17] | 2.22 (d, 3H, $J$ = 1.2); 7.33 (m, 4H); 7.9 (m, 2H) | 159 |
| **7d** | Br | 12 | **8d** | 100 | 66–68 | $C_{12}H_{11}NO_2$ (201.2) | 2.25 (d, 3H, $J$ = 1.2); 2.62 (s, 3H) 7.41 (q, 1H, $J$ = 1.2); 8.0 (m, 4H) | 201 |
| **7l** | Br | 12 | **8l** | 100 | 47–49 | $C_9H_8N_2O$ (160.2) | 2.25 (d, 3H, $J$ = 2.2); 7.3 (m, 1H); 7.41 (q, 1H, $J$ = 1.2); 8.2 (m, 1H); 9.2 (m, 1H) | 160 |
| **7p** | Br | 24 | **8p** | 92 | 57–59 | $C_{14}H_{11}NO$ (209.2) | 2.25 (d, 3H, $J$ = 1.2); 7.25–8.1 (m, 7H); 8.4 (br, 1H) | 209 |

[a] Uncorrected.
[b] Satisfactory microanalyses obtained: C ±0.23, H ±0.11, N ±0.29.
[c] Recorded on a Bruker WP-80 spectrometer.
[d] Recorded on a Varian Mat CH7 spectrometer.

**Table 2.** Reactions of 4,4-Dimethyl-2-trimethylstannyloxazoline (**6b**) with Aryl Halides **7a–s** to give 2-Aryl-4,4-dimethyloxazoline **9a–s**

| Aryl Halide | X | Reaction Time (h) | Product | Yield (%) | m.p. (°C) or[a] b.p. (°C)/mbar | Molecular Formula[b] or Lit. Data | $^1$H-NMR ($CDCl_3$/TMS)[c] $\delta$, $J$(Hz) | MS (70 eV)[d] $m/e$ ($M^+$) |
|---|---|---|---|---|---|---|---|---|
| **7a** | I | 12 | **9a** | 100 | b.p. 79–80/ 0.4 | b.p. 80–81/ 0.5[18] | 1.37 (s, 6H); 4.05 (s, 2H); 7.32 (m, 3H); 7.82 (m, 2H) | 175 |
| **7b** | Br | 24 | **9b** | 100 | b.p. 82–84/ 0.7 | b.p. 83–84/ 0.7[19] | 1.38 (s, 6H); 4.05 (s, 2H); 7.02 (m, 2H); 7.82 (m, 2H) | 193 |
| **7c** | Br | 12 | **9c** | 70 | b.p. 142–144/ 1.3 | b.p. 144–145/ 1.3[18] | 1.35 (s, 6H); 3.78 (s, 3H); 4.02 (s, 2H); 6.8 (m, 2H); 7.78 (m, 2H) | 205 |
| **7d** | Br | 12 | **9d** | 90 | m.p. 86–88 | $C_{13}H_{15}NO_2$ (217.3) | 1.38 (s, 6H); 2.6 (s, 3H); 4.11 (s, 2H); 7.9 (s, 4H) | 217 |
| **7e** | Br | 15 | **9e** | 100 | m.p. 68–70 | $C_{12}H_{15}NOS$ (221.3) | 1.35 (s, 6H); 2.47 (s, 3H); 4.02 (s, 2H); 7.12 (m, 2H); 7.75 (m, 2H) | 221 |
| **7f** | Br | 20 | **9f** | 98 | m.p. 46–48 | $C_{12}H_{12}N_2O$ (200.2) | 1.42 (s, 6H); 4.17 (s, 2H); 7.45–8.12 (m, 4H) | 200 |
| **7g** | Br | 12 | **9g** | 70 | b.p. 70–72/ 0.13 | b.p. 71–73/ 0.13[20] | 1.36 (s, 6H); 4.06 (s, 2H); 7.02 (m, 1H); 7.32 (m, 1H); 7.52 (m, 1H) | 181 |
| **7h** | Br | 12 | **9h** | 100 | m.p. 55–57 | m.p. 56–58[21] | 1.25 (m, 6H); 3.92 (s, 2H); 7.12 (m, 1H); 7.32 (m, 1H); 7.67 (m, 1H) | – |
| **7i** | Br | 24 | **9i** | 85 | oil | $C_{10}H_{12}N_2O$ (176.2) | 1.41 (s, 6H); 4.15 (s, 2H); 7.2–8.0 (m, 3H); 8.57 (m, 1H) | 176 |
| **7l** | Br | 24 | **9l** | 82 | b.p. 88–90/ 0.8 | b.p. 89–91/ 0.9[22] | 1.27 (s, 6H); 4.0 (s, 2H); 7.21 (m, 1H); 8.02 (m, 1H); 8.48 (m, 1H); 8.9 (m, 1H) | 176 |
| **7m** | Br | 24 | **9m** | 85 | oil | $C_8H_{10}N_2OS$ (182.2) | 1.42 (s, 6H); 4.17 (s, 2H); 7.47 (d, 1H, $J$ = 3.0); 7.9 (d, 1H, $J$ = 3.0) | 182 |
| **7n** | Br | 18 | **9n** | 80 | oil | $C_9H_{11}NO_2$ (165.2) | 1.35 (s, 6H); 4.0 (s, 2H); 6.68 (m, 1H); 7.32 (m, 1H); 7.77 (m, 1H) | 165 |
| **7o** | Br | 48 | **9o** | 25 | oil | $C_{15}H_{15}NO$ (225.3) | 1.45 (s, 6H); 4.11 (s, 2H); 7.15–8.27 (m, 6H); 9.0 (m, 1H) | 225 |
| **7p** | Br | 12 | **9p** | 90 | m.p. 153–155 | $C_{15}H_{15}NO$ (225.3) | 1.3 (s, 6H); 4.0 (s, 2H); 6.9–8.3 (m, 7H) | 225 |
| **7q** | Cl | 12 | **9q** | 75 | oil | $C_{14}H_{14}N_2O$ (226.3) | 1.45 (s, 6H); 4.25 (s, 2H); 7.47–8.32 (m, 6H) | 226 |
| **7r** | Br | 48 | **9r** | 92 | m.p. 76–78 | $C_{14}H_{14}N_2O$ (226.3) | 1.41 (s, 6H); 4.13 (s, 2H); 7.5–8.2 (m, 3H); 8.6 (m, 1H); 9.32 (d, 1H, $J$ = 2.4) | 226 |
| **7s** | Br | 12 | **9s** | 92 | m.p. 132–134 | $C_{14}H_{14}N_2O$ (226.3) | 1.46 (s, 6H); 4.13 (s, 2H); 7.45–8.0 (m, 3H); 8.9–9.22 (m, 3H) | 226 |

[a] Uncorrected.
[b] Satisfactory microanalysis obtained: C ±0.21, H ±0.10, N ±0.32.
[c] Recorded on a Bruker WP-80 spectrometer.
[d] Recorded on a Varian Mat CH7 spectrometer.




| 7–9 | Ar | 7–9 | Ar |
|---|---|---|---|
| a | $C_6H_5$ | l | 3-pyridyl |
| b | 4-$FC_6H_4$ | m | 2-thiazolyl |
| c | 4-$CH_3C_6H_4$ | n | 3-furyl |
| d | 4-$CH_3COC_6H_4$ | o | 1-naphtyl |
| e | 4-$CH_3SC_6H_4$ | p | 2-naphtyl |
| f | 2-$CNC_6H_4$ | q | 2-quinolyl |
| g | 2-thienyl | r | 3-quinolyl |
| h | 3-thienyl | s | 3-isoquinolyl |
| i | 2-pyridyl | | |

give the oxazolines from their 2-carboxylic acid derivatives. Finally, since the oxazole[15] and oxazoline[5,16] rings are latent carboxylic acid and carbonyl group equivalents, the reactions can be looked at as a route to a masked multifunctionalization of aromatic and heterocyclic systems. Extension of this approach to the synthesis of chiral aryl- and heteroaryloxazolines is in progress.

*Caution!* Due to the toxicity of trimethyltin chloride, all the experiments with this reagent have to be carried out under an efficient ventilated hood.

**4-Methyl-2-trimethylstannyloxazole (3b):**
A 1.5 molar solution of BuLi in *n*-hexane (42 mL, 63 mmol) is added dropwise to a cooled (−78 °C) and stirred solution of 4-methyloxazole (**1**; 4.75 g, 57 mmol) in ether (100 mL). After 30 min, the mixture is quenched with a solution of trimethyltin chloride (11.3 g, 57 mmol) in ether (50 mL) and allowed to stand at −78 °C for 30 min. The temperature is allowed to rise to 25 °C and the mixture is filtered over Celite and the solvent removed under vacuum. $^1$H-NMR and IR spectra of the crude reaction show only the presence of the stannyloxazole **3b**. Distillation gives 4-methyl-2-trimethylstannyloxazole (**3**); yield: 8.4 g (60%); b.p. 92–95 °C/27 mbar.

IR (Film): $\nu$ = 2920, 1610 cm$^{-1}$.

$^1$H-NMR ($CDCl_3$/TMS): $\delta$ = 0.4 (s, 9H, $SnMe_3$); 2.19 (d, 3H, $=CMe_3$, $J$ = 1 Hz); 7.5 (q, 1H, =CH, $J$ = 1 Hz).

| | | | | |
|---|---|---|---|---|
| $C_7H_{13}NOSn$ | calc. | C 34.19 | H 5.33 | N 5.70 |
| (245.9) | found | 34.21 | 5.32 | 5.71 |

**4,4-Dimethyl-2-trimethylstannyl-2-oxazoline (6b):**
The reaction is carried out as described above for the oxazole **1** starting from BuLi (19 mmol), 4,4-dimethyloxazoline (**4**;[2] 1.73 g, 17 mmol) in ether (100 mL) and trimethyltin chloride (3.47 g, 17 mmol) in ether (50 mL). The $^1$H-NMR spectrum of the crude mixture shows the presence of **6b** exclusively wheras the IR spectrum exhibits a peak at 2120 cm$^{-1}$ (N=C) due to traces of the open-chain tautomer **5b**. Distillation gives **6b**; yield: 3.2 g (70%); b.p. 78–80 °C/21 mbar.

IR (Film): $\nu$ = 2980, 1635 cm$^{-1}$.

$^1$H-NMR ($CDCl_3$/TMS): $\delta$ = 0.35 (s, 9H, $SnMe_3$); 1.26 (s, 6H, $CMe_2$); 3.68 (s, 2H, $CH_2$).

| | | | | |
|---|---|---|---|---|
| $C_8H_{17}NOSn$ | calc. | C 36.68 | H 6.54 | N 5.85 |
| (261.9) | found | 36.66 | 6.55 | 5.83 |

***O*-Trimethylsilyl-2-isocyano-2-methylpropan-1-ol (5a):**
To a cooled and stirred solution of 4,4-dimethyloxazoline (**4**;[2] 5 g, 5.05 mmol) in ether (100 mL) is added dropwise 1.5 molar solution of BuLi in hexane (37 mL, 55.5 mmol). After 30 min stirring, a solution of $ClSiMe_3$ (6.4 mL, 50.5 mmol) in ether (50 mL) is added and the reaction is allowed to stand at −78 °C for 30 min. After the temperature has risen to 25 °C, the mixture is filtered over Celite and the solvent removed under vacuum. Distillation gives the isocyanide **5a**; yield: 4.6 g (62%); b.p. 70–73 °C/20 mbar.

IR (Film): $\nu$ = 2120 (N=C), 1625 cm$^{-1}$.

$^1$H-NMR ($CDCl_3$/TMS): $\delta$ = 1.23 (m, 6H); 3.37 (m, 2H).

| | | | | |
|---|---|---|---|---|
| $C_8H_{17}NOSi$ | calc. | C 56.10 | H 10.00 | N 8.18 |
| (171.3) | found | 56.14 | 9.98 | 8.16 |

Attempts to isomerize **5a** to 4,4-dimethyl-2-trimethylsilyoxazoline (**6a**) are carried out by prolonged heating (4 days, 100 °C) in the presence of KOH. $Be(OH)_2$ or $LiCO_3$ and distillation. In each case part of the isonitrile is recovered unaltered together with considerable amount of tar.

**Cross-Coupling Reactions of 4-Methyl-2-trimethylstannyloxazole (3b) with Aryl Halides 7; Typical Procedure:**
A solution of oxazole **3b** (0.9 g, 3.6 mmol), aromatic halide **7** (3.6 mmol) and tetrakis(triphenylphosphine)palladium(0) (0.2 g, 0.18 mmol) in anhydrous benzene (10 mL) is heated at 80 °C for an appropriate time (see Table 1). The solvent is removed under vacuum and the residue chromatographed (silica gel, ether/*n*-hexane, 1:1) to give 2-aryloxazole **8**.

**Cross-Coupling Reactions of 4,4-Dimethyl-2-trimethylstannyl-2-oxazoline (6b) with Aryl Halides 7; Typical Procedure:**
A solution of oxazoline **6b** (0.93 g, 3.6 mmol), aromatic aryl halide **7** (3.6 mmol) and tetrakis(triphenylphosphine)palladium(0) (0.2 g, 0.18 mmol) in anhydrous benzene (10 mL) is heated for an appropriate time (see Table 2). After usual work-up of the reaction mixture, the product **9** is isolated by chromatography (silica, ether/*n*-hexane 1:1).

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**Note Added in Proof: 4,4-Dimethyl-2-tributylstannyl-2-oxazoline** has been prepared in our laboratory as described for **6b** from the lithio oxazoline and tributylstannyl chloride; yield: 72%; b.p. = 127–130 °C/4 mbar.

$^1$H-NMR ($CDCl_3$/TMS): $\delta$ = 0.9 (t, 9H); 1.25 (s, 6H); 1.32 (m, 18H); 3.70 (s, 2H).

| | | | | |
|---|---|---|---|---|
| $C_{17}H_{35}NOSn$ | calc. | C 52.60 | H 9.09 | N 3.61 |
| (388.2) | found | 52.55 | 9.13 | 3.58 |

The tributylstannyloxazoline undergoes cross-coupling reactions with various aryl and heteroaryl halides **7** to give the corresponding products **9** (yields 80–90%). Hence, 4,4-dimethyl-2-tributylstannyl-2-oxazoline should be conveniently employed in place of **6b**, in order to avoid the use of the highly toxic trimethylstannyl chloride.

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