

CONDENSED HETEROAROMATIC RING SYSTEMS. XIX.¹ SYNTHESIS AND REACTIONS OF 5-(TRIBUTYLSTANNYL)ISOXAZOLES²

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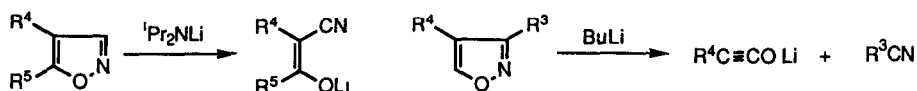
3-Substituted 5-(tributylstannyl)isoxazoles were newly synthesized by the 1,3-dipolar cycloaddition of nitrile oxides to tributylethynylstannane. The iodination and the palladium-catalyzed benzoylation of 5-(tributylstannyl)-3-methylisoxazole gave 5-iodo-3-methylisoxazole and 3-methyl-5-isoxazolyl phenyl ketone in satisfactory yields, respectively. The palladium-catalyzed cross-coupling reaction of the stannylisoxazole with 2-bromonitrobenzene followed by the catalytic hydrogenation over Raney nickel resulted in the formation of 2-methyl-4(1*H*)-quinolinone in 57% overall yield

Trialkylstannylarenes and heteroarenes are useful for the synthesis of the corresponding acyl or halogeno derivatives.³ But, the general synthetic methods for the stannyl compounds are limited to the reaction of aryllithiums⁴ or aryl Grignard reagents⁵ with trialkylstannyl halides, the reaction of aryl halides with trialkylstannylolithiums⁶ or sodiums,⁷ and the palladium-catalyzed reaction of aryl halides or triflates with hexaalkyldistannanes.⁸ Furthermore, there is few papers for the synthesis by cycloaddition reaction of trialkylethynylstannanes with 1,3-dipolar compounds.⁹

In this paper, we describe the synthesis of 5-tributylstannylisoxazoles by the 1,3-dipolar cycloaddition reaction of tributylethynylstannane with nitrile oxides, the palladium-catalyzed cross-coupling reaction of the tributylstannylisoxazoles with aryl halides, and the synthesis of some bicyclic heteroaromatics.

Synthesis of Tributylstannylisoxazoles

It is well known that an attempted lithiation of isoxazole at the 3-position or the 5-position with butyllithium resulted in the ring-cleavage as shown below.^{10,11} These facts suggest unsatisfactory results for the synthesis of 3- and 5-(trialkylstannyl)isoxazoles using the substitution of metallated isoxazoles with trialkylstannyl halides.

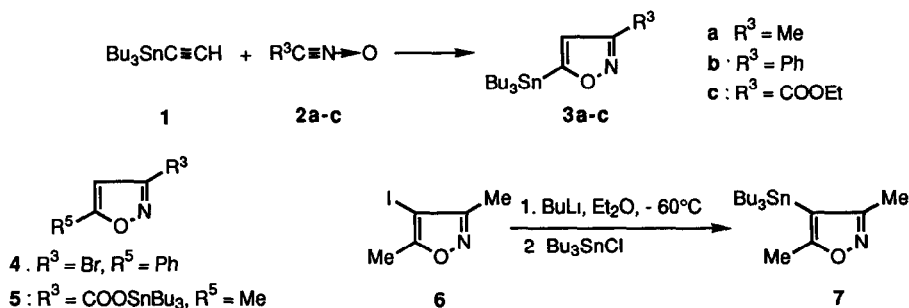


Scheme 1

Thus, we investigated the 1,3-dipolar cycloaddition of nitrile oxides (2a-c) generated from appropriate precursors with tributylethynylstannane (1) as an alternative method for the synthesis of 5-(tributylstannyl)isoxazoles (3a-c). When 1 was treated with acetonitrile oxide (2a) generated *in situ* from nitroethane and phenyl isocyanate,¹² 3a was obtained in nearly quantitative yield. Similarly, 3b was synthesized by the reaction of 1 with benzonitrile oxide (2b) from phenylnitromethane. In addition to the above, the reaction of 1 with ethyl chlorooximido-

acetate¹³ (a precursor of **2c**) in the presence of triethylamine, smoothly gave ethyl 5-(tributylstannyl)isoxazole-3-carboxylate (**3c**).

In connection with the above, the synthesis of 3- and 4-(tributylstannyl)isoxazoles were examined. The condensation of 3-bromo-5-phenylisoxazole (**4**) with tributylstannyllithium¹⁴ and the decarboxylation of tributylstannyl 3-methylisoxazole-3-carboxylate (**5**)¹⁵ failed to give the desired 3-(tributylstannyl)isoxazole derivatives. On the other hand, 4-(tributylstannyl)-3,5-dimethylisoxazole (**7**) was isolated through the lithiation of 4-iodo-3,5-dimethylisoxazole (**6**), and the subsequent reaction of the lithiated isoxazole with tributylstannyl chloride,¹⁶ although the yield of **7** was unsatisfactory.

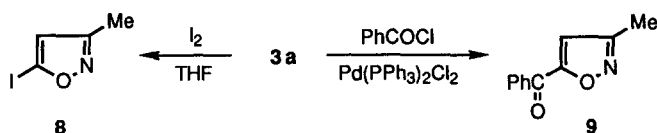


Scheme 2

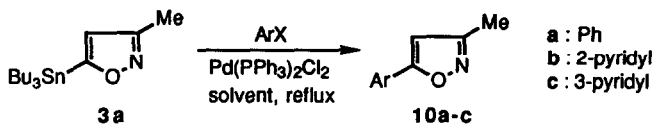
Reactions of 5-(Tributylstannyl)-3-methylisoxazole (**3**)

It has been known that, in principle, the 5-position of isoxazole ring is inactive towards electrophilic substitutions, but **3a** smoothly reacted with iodine in tetrahydrofuran to give 5-iodo-3-methylisoxazole (**8**). Further, when **3a** was allowed to react with benzoyl chloride in the presence of dichlorobis(triphenylphosphine)palladium as a catalyst, the reaction in dioxane proceeded under reflux to give 3-methyl-5-isoxazolyl phenyl ketone (**9**).

As well as the benzoylation of **3a**, the cross-coupling reaction of **3a** with aryl halides was also catalyzed by the palladium complex. The results of the cross-coupling reaction are listed in Table I, in which advantage of dioxane as a solvent is clearly demonstrated.



Scheme 3

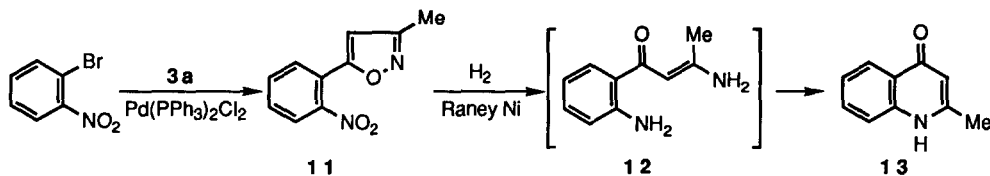
Table I. The Palladium-Catalyzed Cross-Coupling Reaction of **3 a** with Aryl Halides


a : Ph
b : 2-pyridyl
c : 3-pyridyl

ArX	Solvent	Reaction time (h)	Yield of 10 (%)
PhI	THF	7	82
PhBr	THF	5.5	18
PhBr	dioxane	6	59
PhBr	dioxane	25	72
2-Bromopyridine	THF	14.5	17
2-Bromopyridine	dioxane	6	64
3-Bromopyridine	THF	15	15
3-Bromopyridine	dioxane	4	60

Syntheses of Bicyclic Heteroaromatic Compounds

In order to confirm synthetic utility of the isoxazole moiety introduced into benzene ring, the synthesis of some heteroaromatic compounds with bicyclic structure were tested using the cross-coupling reaction of **3 a** and **7** with 2-substituted bromobenzenes. As shown in Table II, in the reaction of 2-bromonitrobenzene with **3 a**, a satisfactory result was obtained by using 1 mole% of the palladium catalyst. When 3-methyl-5-(2-nitrophenyl)isoxazole (**11**) thus obtained was hydrogenated over Raney nickel, 2-methyl-4(1*H*)-quinolinone (**13**) was isolated as a sole product. The cross-coupling reaction of **3 a** with 2-(methoxymethoxy)bromobenzene gave the corresponding 5-[2-(methoxymethoxy)phenyl]-3-methylisoxazole (**14**) which was also readily converted into 2-methylchromone (**16**) by the catalytic hydrogenation over Raney nickel followed by treatment with concentrated hydrochloric acid in acetic acid. These results clearly exhibited the formation of enaminketone intermediate (**12** and **15**) through the ring-cleavage of the isoxazole moiety, as expected.



Schme 4

Table II. The Palladium-Catalyzed Cross-Coupling Reaction of 2-Nitrobromobenzene with **3 a**

Mole% of Pd catalyst	Reaction time (h)	Yield (%)
10%	20	27
5%	15	32
5%	20	54
1%	20	90

The whole mixture was stirred at 50°C for 16 h, diluted with water and then filtered through a Celite pad. The filtrate was extracted with benzene. The benzene extract was dried over MgSO_4 and concentrated under reduced pressure. The residue was chromatographed on a silica gel column with benzene as an eluent to give a yellow viscous liquid (2.16 g, 100%). $^1\text{H-NMR}$ (CDCl_3 , ppm): 0.6 - 1.8 (27H, m), 6.70 (1H, s), 7.2 - 7.6 (3H, m), 7.8 - 8.0 (2H, m). $\text{MS}(m/z)$: 378 ($\text{M}^+ - \text{C}_4\text{H}_9$). High Resolution MS Calcd for $\text{C}_{17}\text{H}_{24}\text{NOSn}$ ($\text{M}^+ - \text{C}_4\text{H}_9$): 378.0880. Found: 378.0880.

Ethyl 5-(tributylstannyl)isoxazole-3-carboxylate (3c)

An ethereal (4 ml) solution of Et_3N (0.5 ml) was added to an ethereal (6 ml) solution of ethyl chlorooximidoacetate (0.46 g, 3 mmol) and **1** (1.04 g, 3.3 mmol) at room temperature. The mixture was stirred for 5 h, diluted with water, and extracted with ether. The ethereal extract was dried over MgSO_4 and concentrated under reduced pressure. The residue was chromatographed on a silica gel column with benzene as an eluent to give colorless viscous liquid (1.10 g, 85%). $^1\text{H-NMR}$ (CDCl_3 , ppm): 0.7-1.9 (30H, m), 4.47 (2H, q, $J = 7$ Hz), 6.82 (1H, s). IR (CHCl_3), cm^{-1} : 1735. $\text{MS}(m/z)$: 374 ($\text{M}^+ - \text{C}_4\text{H}_9$). Resolution MS Calcd for $\text{C}_{14}\text{H}_{24}\text{NO}_3\text{Sn}$ ($\text{M}^+ - \text{C}_4\text{H}_9$): 374.0778. Found: 374.0752

3,5-Dimethyl-4-(tributylstannyl)isoxazole (7)

1.56 M Butyllithium in hexane (4.30 ml, 10 mmol) was added dropwise to a solution of 4-iodo-3,5-dimethylisoxazole (**6**) (2.24 g, 10 mmol) in ether (60 ml) with stirring under nitrogen atmosphere at -60°C to -55°C. The resulting suspension was stirred at the same temperature for 2.5 h, to which tributylstannyl chloride (3.58 g, 11 mmol) in ether (50 ml) was added at such rate as to keep the reaction temperature below -50°C. The mixture was stirred at -55°C for 1 h, allowed to warm to room temperature, and set aside overnight. After addition of water, the ethereal solution was separated, and the aqueous phase was extracted with ether. The residue obtained from the ethereal extracts was distilled under reduced pressure to give a colorless liquid (1.96 g, 51%), bp 140-150°C / 0.6 mmHg. $^1\text{H-NMR}$ (CDCl_3 , ppm): 0.7 - 1.9 (27H, m), 2.23 (3H, s), 2.36 (3H, s). *Anal.* Calcd for $\text{C}_{17}\text{H}_{33}\text{NOSn}$. C, 52.88, H, 8.61; N, 3.63. Found: C, 52.76; H, 8.50; N, 3.49. $\text{MS}(m/z)$: 330 ($\text{M}^+ - \text{C}_4\text{H}_9$). High Resolution MS Calcd for $\text{C}_{13}\text{H}_{24}\text{NOSn}$ ($\text{M}^+ - \text{C}_4\text{H}_9$): 330.0880. Found: 330.0871.

5-Iodo-3-methylisoxazole (8)

Iodine (0.51 g, 2 mmol) in THF (30 ml) was added dropwise to a stirred solution of **3a** (0.75 g, 2 mmol) in THF (20 ml) at room temperature, and the mixture was stirred for additional 1 h. After addition of aqueous NaHCO_3 , the mixture was extracted with ether. The ethereal extract was washed with $\text{Na}_2\text{S}_2\text{O}_3$, dried over MgSO_4 , and concentrated under reduced pressure. The residue was chromatographed on a silica gel column with benzene as an eluent. The product obtained from the benzene eluate was distilled under reduced pressure to give a colorless liquid (0.24 g, 57%), bp 70°C / 3 mmHg. $^1\text{H-NMR}$ (CDCl_3 , ppm): 2.25 (3H, s), 6.23 (1H, s). *Anal.* Calcd for $\text{C}_4\text{H}_4\text{INO}$: C, 22.99, H, 1.93; N, 6.70. Found: C, 23.23; H, 2.09; N, 6.67.

3-Methyl-5-Isoxazolyl phenyl ketone (9)

A mixture of **3a** (1.12 g, 3 mmol), benzoyl chloride (0.42 g, 3 mmol), and $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$ (0.06 g) in dioxane (20 ml) was refluxed for 3 h. After cooling, the mixture was diluted with water and extracted with CHCl_3 . The CHCl_3 extract was dried over MgSO_4 and concentrated under reduced pressure. The residue was chromatographed on a silica gel column with hexane-AcOEt (7:1) as an eluent to give a colorless liquid (0.45 g, 80%), bp 150°C / 3 mmHg. The liquid crystallized on cooling and was recrystallized from hexane to give colorless scales, mp 67 - 69°C (lit.¹⁷ mp 68°C). $^1\text{H-NMR}$ (CDCl_3 , ppm).

2.42 (3H, s), 6.84 (1H, s), 7.3 - 7.7 (3H, m), 8.0 - 8.3 (2H, m). IR (CHCl₃, cm⁻¹): 1665

General Procedure for the Reaction of **3a** with Aryl Halides

A mixture of **3a**, an aryl halide, and Pd(PPh₃)₂Cl₂ in dioxane was refluxed until the substrate was disappeared (monitored by TLC). After removal of the dioxane under reduced pressure, the residue was diluted with H₂O and extracted with CHCl₃. The CHCl₃ extract was dried over MgSO₄ and concentrated under reduced pressure. The residue was chromatographed on a silica gel column with an appropriate solvent as an eluent. Distillation under reduced pressure or recrystallization from an appropriate solvent gave the pure product.

3-Methyl-5-phenylisoxazole (**10a**)

1) A mixture of bromobenzene (0.47 g, 3 mmol), **3a** (1.67 g, 4.5 mmol), and Pd(PPh₃)₂Cl₂ (0.06 g) in dioxane (20 ml) was refluxed for 25 h. After treatment as described above, the residue was chromatographed on a silica gel column with benzene as an eluent to give a colorless liquid (0.34 g, 72%), bp 130°C / 3 mmHg. The liquid crystallized on cooling was recrystallized from aqueous MeOH, mp 62 - 64°C (lit.¹⁸ mp 65°C). ¹H-NMR (CDCl₃, ppm): 2.33 (3H, s), 6.34 (1H, s), 7.2 - 7.9 (5H, m)

2) A mixture of bromobenzene (0.47 g, 3 mmol), **3a** (1.67 g, 4.5 mmol), and Pd(PPh₃)₂Cl₂ (0.06 g) in THF (20 ml) was refluxed for 55 h. Treatment as described above gave **10a** (0.08 g, 18%).

3) A mixture of iodobenzene (0.61 g, 3 mmol), **3a** (1.67 g, 4.5 mmol), and Pd(PPh₃)₂Cl₂ (0.06 g) in THF (20 ml) was refluxed for 7 h. Treatment as described above gave **10a** (0.39 g, 82%).

3-Methyl-5-(2-pyridyl)isoxazole (**10b**)

1) A mixture of 2-bromopyridine (0.48 g, 3 mmol), **3a** (1.67 g, 4.5 mmol), and Pd(PPh₃)₂Cl₂ (0.06 g) in dioxane (20 ml) was refluxed for 6 h. After treatment as described above, the residue was chromatographed on a silica gel column with hexane-AcOEt (2:1) as an eluent to give colorless prisms (0.31 g, 64%), mp 55 - 57°C, which was recrystallized from hexane. ¹H-NMR (CDCl₃, ppm): 2.36 (3H, s), 6.73 (1H, s), 7.1 - 7.5 (1H, m), 7.7 - 8.0 (2H, m), 8.6 - 8.8 (1H, m). *Anal. Calcd* for C₉H₈N₂O: C, 67.49; H, 5.03; N, 17.49. Found: C, 67.62; H, 5.04, N, 17.58

2) A mixture of 2-bromopyridine (0.25 g, 1.6 mmol), **3a** (0.90 g, 2.4 mmol), and Pd(PPh₃)₂Cl₂ (0.03 g) in THF (11 ml) was refluxed for 14.5 h. Treatment as described above gave **10b** (0.04 g, 17%).

3-Methyl-5-(3-pyridyl)isoxazole (**10c**)

1) A mixture of 3-bromopyridine (0.48 g, 3 mmol), **3a** (1.67 g, 4.5 mmol), and Pd(PPh₃)₂Cl₂ (0.06 g) in dioxane (20 ml) was refluxed for 4 h. After treatment described above, the residue was chromatographed on a silica gel column with hexane-AcOEt (2:1) as an eluent to give colorless prisms (0.29 g, 60%), mp 62 - 63°C, which were recrystallized from hexane. ¹H-NMR (CDCl₃, ppm): 2.38 (3H, s), 6.48 (1H, s), 7.3 - 7.6 (1H, m), 8.0 - 8.2 (1H, m), 8.6 - 8.8 (1H, m), 9.0 - 9.1 (1H, m). *Anal. Calcd* for C₉H₈N₂O: C, 67.49, H, 5.03, N, 17.49. Found: C, 67.35, H, 4.83, N, 17.54

2) A mixture of 3-bromopyridine (0.48 g, 3 mmol), **3a** (1.67 g, 4.5 mmol), and Pd(PPh₃)₂Cl₂ (0.06 g) in dioxane (20 ml) was refluxed for 15 h. Treatment described above gave **10c** (0.06 g, 15%).

3-Methyl-5-(2-nitrophenyl)isoxazole (**11**)

1) 2-Bromonitrobenzene (0.40 g, 2 mmol), **3a** (0.89 g, 2.4 mmol), Pd(PPh₃)₂Cl₂ (0.02 g) in dioxane (10 ml) was refluxed for 20 h. After treatment described above (extracted with ether), the residue was chromatographed on a silica gel column with hexane-AcOEt (5:1) as an eluent to give a yellow liquid (0.36 g, 90%), bp 180 - 190°C / 5 mmHg. ¹H-NMR

(CDCl₃, ppm): 2.35 (3H, s), 6.36 (1H, s), 7.3 - 8.0 (4H, m). *Anal.* Calcd for C₁₀H₈N₂O₃: C, 58.82; H, 3.95; N, 13.72. Found: C, 58.92; H, 3.91; N, 13.64.

5-[2-(Methoxymethoxy)phenyl]-3-methylisoxazole (14)

A mixture of 2-(methoxymethoxy)iodobenzene (0.53 g, 2 mmol), **3a** (0.89 g, 2.4 mmol), and Pd(PPh₃)₂Cl₂ (0.02 g) in dioxane (10 ml) was refluxed for 4 h. After treatment described above (extracted with ether), the residue was chromatographed on a silica gel column with hexane-AcOEt (10:1) as an eluent to give a colorless liquid (0.30 g, 68%), bp 150 - 160°C / 6 mmHg. ¹H-NMR (CDCl₃, ppm) 2.33 (3H, s), 3.48 (3H, s), 5.30 (2H, s), 6.62 (1H, s), 6.9 - 7.5 (3H, m), 7.8 - 8.1 (1H, m). MS (*m/z*): 219 (M⁺). High Resolution MS Calcd for C₁₂H₁₃NO₃ (M⁺): 219.0895. Found: 219.0900.

3,5-Dimethyl-4-(2-nitrophenyl)isoxazole (17)

A mixture of 2-bromonitrobenzene (0.40 g, 2 mmol), **7** (0.93 g, 2.4 mmol), and Pd(PPh₃)₂Cl₂ (0.02 g) in dioxane (10 ml) was refluxed for 20 h. After treated with as described above (extracted with ether), the residue was chromatographed on a silica gel column with hexane-AcOEt (5:1) as an eluent to give a yellow liquid (0.25 g, 57%), bp 180-190°C / 5 mmHg). ¹H-NMR (CDCl₃, ppm): 2.21 (3H, s), 2.27 (3H, s), 7.2 - 8.2(4H, m). *Anal.* Calcd for C₁₁H₁₀N₂O₃: C, 60.55; H, 4.62; N, 12.84. Found: C, 60.60; H, 4.60; N, 12.85.

2-Methyl-4(1*H*)-quinolone (13)

An MeOH (10 ml) solution of 3-methyl-5-(2-nitrophenyl)isoxazole (**11**) (1.02 g, 5 mmol) was hydrogenated at 5.2 atm. pressure over Raney nickel W-1 prepared from Ni-Al alloy (2.50 g) for 3.5 h. The catalyst was filtered off and washed with MeOH, and the MeOH solution was concentrated under reduced pressure. The residue was recrystallized from MeOH-AcOEt to give colorless needles (0.50 g, 63%), mp 233 - 234°C (lit.¹⁹ mp 234 - 235.5°C). ¹H-NMR (CDCl₃-DMSO-d₆, ppm): 2.41 (3H, s), 6.08 (1H, m), 7.1 - 7.7 (4H, m), 8.1 - 8.4 (1H, m). IR (CHCl₃, cm⁻¹): 1605.

2-Methylchromone (16)

An MeOH (10 ml) solution of 5-[(2-methoxymethoxy)phenyl]-3-methylisoxazole (**14**) (0.22 g, 1 mmol) was hydrogenated at 5.6 atm. pressure over Raney nickel W-1 prepared from Ni-Al alloy (2.50 g) for 2 h. The catalyst was filtered off and washed with MeOH, and the MeOH solution was concentrated under reduced pressure. The residue was dissolved in a mixture of concentrated HCl (10 ml) and AcOH (10 ml), and the mixture was stirred at 80°C for 20 min. After neutralization with 3N NaOH, the mixture was extracted with CHCl₃. The CHCl₃ extract was dried over MgSO₄ and evaporated under reduced pressure. The residue was recrystallized from hexane-CHCl₃ to give colorless prisms (0.09 mg, 56%), mp 72°C (lit.²⁰ mp 70 - 71°C). ¹H-NMR (CDCl₃, ppm). 2.26 (3H, s), 6.20 (1H, s), 7.2 - 7.9 (3H, m), 8.1 - 8.4 (1H, m)

Methyl 2-methyl-3-Indolyl ketone (18)

An MeOH (10 ml) solution of 3,5-dimethyl-4-(2-nitrophenyl)isoxazole (**17**) (0.87 g, 4 mmol) was hydrogenated at 5.2 atm. pressure over Raney nickel W-1 prepared from Ni-Al alloy (2.50 g) for 3.5 h. The catalyst was filtered off and washed with MeOH, and the MeOH solution was concentrated under reduced pressure. The soluble residue in AcOEt was chromatographed on a silica gel column with hexane-AcOEt (1.1) as an eluent to give colorless needles (0.22 g, 32%), mp 199 - 202°C (lit.²¹ mp 195 - 196°C), which was recrystallized from EtOH. ¹H-NMR (DMSO-d₆, ppm): 2.63 (3H, s), 2.74 (3H, s), 7.1 - 7.5 (3H, m), 7.9 - 8.2 (1H, m), 10.5 - 10.9 (1H, br).

References and Notes

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