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Supporting Information

Rh(III)-Catalyzed Olefination to Build Diverse Oxazole Derivatives from Functional Alkynes

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Table of Contents

| 1. General Information2 |
|--|
| 2. Material2 |
| 3. General Procedure for the Synthesis of Oxazole Derivatives |
| 4. Optimization of the Reaction Conditions |
| 5. Synthetic Applications5 |
| 6. Mechanism Study7 |
| 7. References |
| 8. X-ray Data of Compound 3aa and Sample Preparation11 |
| 9. Characterization Data and NMR Spectra of Oxazole Derivatives 19 |
| 10. ¹ H , ¹³ C and ¹⁹ F NMR spectra |

1. General Information

All reactions were carried out in high-pressure reaction tube. Column chromatography was performed with silica gel (200–300 mesh). High-resolution mass spectra (HRMS) were obtained with a Waters-Q-TOF-Premier (ESI). ¹H NMR and ¹³C NMR spectra were recorded on a Bruker 400 MHz and 100MHz instrument. Spectra were reported relative to Me₄Si (δ 0.0 ppm), CDCl₃ (δ 7.26 ppm). ¹³C NMR were reported relative to CDCl₃ (δ 77.16 ppm). Splitting patterns are designated as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. Compounds were characterized by ¹H NMR, ¹³C NMR, ¹⁹F NMR and HRMS.

2. Material

Unless otherwise noted, all reagents were obtained from commercial suppliers and used without further purification. Solvents were dried over sodium (for THF and ether) and CaH₂ (for toluene, DCM and DCE) by refluxing for overnight and freshly distilled prior to use. Methanol was dried and distilled from magnesium powder under nitrogen atmosphere. 2-aryloxazoles was prepared following literature procedures^[1]. Alkynes were purified by column chromatography (petroleum ether) before each used.

General Procedure A for the Synthesis of Alkynes^[2]



To a solution of the allylic halide (1.5 equiv), Na₂SO₃ (0.5 equiv), CuI (2 mol%), K₂CO₃ (1 equiv) and 1 drop of DBU in DMSO, alkyne derivatives (1 equiv) were added and stirred at 30 °C overnight. After acidic work up, the mixture was quenched with water and extracted with dichloromethane (3×20 mL), the combined organic layer was washed with saturated aqueous NaHCO₃ and brine, then the combined organic layers were dried (MgSO₄), filtered, and concentrated in vacuo. Purification of the residue by column chromatography (petroleum ether) afforded the product **2**.

General Procedure B for the Synthesis of Alkynes^[3]

$$R_{l}^{\square} \xrightarrow{X} + R \xrightarrow{Pd(PPh_3)_2Cl_2 (5 \text{ mol}\%)} R \xrightarrow{Pd(Ph_3)_2Cl_2 (5 \text{ mol}\%)} R \xrightarrow{P$$

A mixture of the Pd(PPh₃)₂Cl₂ (5 mol%), CuI (5 mol%), aryl halide or bromide (1.0 equiv), Degassed Et₃N and alkyne derivatives (1.1 equiv) were added and stirred at 60 °C overnight under Ar atmosphere. The mixture was quenched with aqueous saturated NH₄Cl and extracted with EtOAc (3 × 20 mL), the combined organic layer was washed with brine and then the combined organic layers were dried (MgSO4), filtered, and concentrated in vacuo. Purification of the residue by column chromatography (petroleum ether) afforded the product **2**.

General Procedure B for the Synthesis of Alkynes^[4]

A mixture of alkyne (1.0 equiv) in THF (0.25 M) was cooled to -78 °C and a solution of *n*-BuLi (1.0 equiv, 1.6 M in hexanes) was added. The reaction mixture was allowed to warm to room temperature. Then NaI (0.1 equiv.) and the corresponding bromoethylbenzene (1.2 equiv) were added. The mixture was heated to reflux. The reaction mixture was cooled to room temperature and quenched by addition of ammonium chloride solution. After completion of the reaction, extracted with EtOAc (3×20 mL), the combined organic layer was washed with brine and then the combined organic layers were dried (MgSO4), filtered, and concentrated in vacuo. Purification of the residue by column chromatography (petroleum ether) afforded the product **2**.

3. General Procedure for the Synthesis of Oxazole Derivatives



A mixture of 2-aryloxazolines 1 (0.1 mmol), acetylene derivatives 2 (2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (7 mol%), PhOCH₂COOH (1.5 equiv), and MnO₂ (0.3 equiv) were added to an oven dried high pressure tube under Ar atmosphere. Toluene (1 mL) was then added by syringe. The reaction mixture was stirred at 80 °C until raw materials disappear (monitored by TLC). After removal of the volatiles under vacuum, the crude product was purified by column chromatography on silica gel afforded the pure product **3**.

4. Optimization of the Reaction Conditions



| Table S1. The effect of catalysis on the reaction | fable S1 | le S1. The effect | t of catalysts or | 1 the reaction ^a |
|---|----------|-------------------|-------------------|-----------------------------|
|---|----------|-------------------|-------------------|-----------------------------|

| Entry | Catalyst | Acid | Yield |
|-------|---|-------------------------|-------|
| 1 | [Cp*IrCl ₂] ₂ | PhOCH ₂ COOH | ND |
| 2 | [RuCl ₂ (p-cymene)] ₂ | PhOCH ₂ COOH | ND |
| 3 | Cp*Rh(CH ₃ CN) ₃ (SbF ₆) ₂ | PhOCH ₂ COOH | 56% |
| 4 | [Cp*RhCl2]2 | PhOCH ₂ COOH | ND |
| 5^b | [Cp*RhCl ₂] ₂ | PhOCH ₂ COOH | 27% |
| 6 | Cp*Rh(OAc)2·H2O | PhOCH ₂ COOH | 28% |
| 7 | Cp*Co(CO)I2 | PhOCH ₂ COOH | ND |

^aReaction conditions: 1a (0.05 mmol), 2a (2.5 equiv), catalyst (8 mol%), PhOCH₂COOH (1.5 equiv), toluene

(0.5 mL), 100 °C, 24 h under Ar. ^bAgSbF₆ (20 mol%) was added.

| N O | + <u>catalyst, addi Ph solvent</u> | tive Ph | Ph |
|-------|---|--------------------|-------|
| 1a | 2a | 3aa | |
| Entry | Catalyst | Solvent | Yield |
| 1 | Cp*Rh(CH ₃ CN) ₃ (SbF ₆) ₂ | DCE | 42% |
| 2 | Cp*Rh(CH ₃ CN) ₃ (SbF ₆) ₂ | DCM | 50% |
| 3 | Cp*Rh(CH ₃ CN) ₃ (SbF ₆) ₂ | Toluene | 56% |
| 4 | Cp*Rh(CH ₃ CN) ₃ (SbF ₆) ₂ | CH ₃ OH | 22% |
| 5 | Cp*Rh(CH ₃ CN) ₃ (SbF ₆) ₂ | CH ₃ CN | 10% |
| 6 | Cp*Rh(CH ₃ CN) ₃ (SbF ₆) ₂ | THF | 36% |
| 7 | Cp*Rh(CH ₃ CN) ₃ (SbF ₆) ₂ | 1,4-Dioxane | 19% |
| 8 | Cp*Rh(CH3CN)3(SbF6)2 | Chlorobenzene | 54% |

Table S2. The effect of solvents on the reaction^{*a*}

^{*a*}Reaction conditions: **1a** (0.05 mmol), **2a** (2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (8 mol%), PhOCH₂COOH (1.5 equiv), solvent (0.5 mL), 100 °C, 24 h under Ar.

| | N O + Ph | catalyst, additiv solvent | | Ph |
|-------|---------------------------------|------------------------------|-------|-------|
| | 1a 2a | | 3aa | |
| Entry | Acid | Additive | Temp. | Yield |
| 1 | PhOCH ₂ COOH | - | 100 | 56% |
| 2 | HOAc | - | 100 | 18% |
| 3 | PhCOOH | - | 100 | 17% |
| 4 | PhCH ₂ COOH | - | 100 | 45% |
| 5 | ClCH ₂ COOH | - | 100 | 36% |
| 6 | Na ₂ CO ₃ | - | 100 | ND |
| 7 | PhOCH ₂ COOH | - | 80 | 61% |
| 8 | PhOCH ₂ COOH | - | 120 | 29% |
| 9 | PhOCH ₂ COOH | AgO | 80 | 65% |
| 10 | PhOCH ₂ COOH | Ag ₂ O | 80 | 71% |
| 11 | PhOCH ₂ COOH | AgOAc | 80 | 66% |
| 12 | PhOCH ₂ COOH | Cu(OAc) ₂ | 80 | 39% |
| 13 | PhOCH ₂ COOH | CuO | 80 | 65% |
| 14 | PhOCH ₂ COOH | Cu ₂ O | 80 | 52% |
| 15 | PhOCH ₂ COOH | MnO ₂ | 80 | 72% |

Table S3. The effect of acids, additives and temperature on the reaction^{*a*}

^{*a*}Reaction conditions: conditions: **1a** (0.05 mmol), **1a** (2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (8 mol%), PhOCH₂COOH (1.5 equiv), additive (0.3 equiv), toluene (0.5 mL), 100 °C, 24 h under Ar.

Table S4. The effect of equivalant on the reaction^a



| Entry | Catalyst | Acid | Additive | Yield |
|----------------|--|-----------------------------|-----------------------|-------|
| 1 | Cp*Rh(CH ₃ CN) ₃ (SbF ₆) ₂ /8 | PhOCH ₂ COOH/1 | MnO ₂ /0.3 | 69% |
| 2 | Cp*Rh(CH ₃ CN) ₃ (SbF ₆) ₂ /8 | PhOCH ₂ COOH/1.5 | MnO ₂ /0.3 | 77% |
| 3 | Cp*Rh(CH ₃ CN) ₃ (SbF ₆) ₂ /8 | PhOCH ₂ COOH/3 | MnO ₂ /0.3 | 75% |
| 4 | Cp*Rh(CH ₃ CN) ₃ (SbF ₆) ₂ /8 | PhOCH ₂ COOH/1.5 | MnO ₂ /0.5 | 71% |
| 5 | Cp*Rh(CH ₃ CN) ₃ (SbF ₆) ₂ /8 | PhOCH ₂ COOH/1.5 | MnO ₂ /1 | 72% |
| 6^b | Cp*Rh(CH ₃ CN) ₃ (SbF ₆) ₂ /8 | PhOCH ₂ COOH/1.5 | MnO ₂ /0.3 | 75% |
| 7 ^c | Cp*Rh(CH ₃ CN) ₃ (SbF ₆) ₂ /8 | PhOCH ₂ COOH/1.5 | MnO ₂ /0.3 | 75% |
| 8 | Cp*Rh(CH ₃ CN) ₃ (SbF ₆) ₂ /9 | PhOCH ₂ COOH/1.5 | MnO ₂ /0.3 | 76% |
| 9 | Cp*Rh(CH ₃ CN) ₃ (SbF ₆) ₂ /7 | PhOCH ₂ COOH/1.5 | MnO ₂ /0.3 | 80% |
| 10 | Cp*Rh(CH ₃ CN) ₃ (SbF ₆) ₂ /6 | PhOCH ₂ COOH/1.5 | MnO ₂ /0.3 | 75% |
| 11^{d} | Cp*Rh(CH ₃ CN) ₃ (SbF ₆) ₂ /8 | PhOCH ₂ COOH/1.5 | MnO ₂ /0.3 | 72% |
| 12^{e} | Cp*Rh(CH ₃ CN) ₃ (SbF ₆) ₂ /8 | PhOCH ₂ COOH/1.5 | MnO ₂ /0.3 | 51% |
| 13 | Cp*Rh(CH ₃ CN) ₃ (SbF ₆) ₂ /8 | - | - | NR |

^{*a*}Reaction conditions: conditions: **1a** (0.05 mmol), **2a** (2.5 equiv), toluene (0.5 mL), 80 °C, 34 h under Ar. ^{*b*}**2a** (3 equiv). ^{*c*} under Air. ^{*d*} 60 °C, ^{*e*} 100 °C.

Table S5. The scope of alkynes^a





5. Synthetic Applications

Synthesis of 4



To a stirred solution of **3as** (0.026 mmol, 15.0 mg, 1.0 equiv) in MeOH (1.0 mL), con HCl (0.1 mL) was added, and the mixture was stirred at room temperature. After completion of the reaction (monitored by TLC), the mixture was quenched with water and extracted with ethyl acetate ($3 \times 20 \text{ mL}$), the combined organic layer was washed with brine and then the combined organic layers were dried (MgSO₄), filtered, and concentrated in vacuo. Purification of the residue by column

chromatography (petroleum ether/ethyl acetate = 1/1, v/v) afforded the product **4** as a colorless oil (9.8 mg, 79% yield).

Synthesis of 5



To a stirred solution of **3aa** (0.05 mmol, 23.6 mg, 1.0 equiv) in EA (1.0 mL), DMSO (8.6 μ L, 1.2 mmol) and aqueous hydrobromic acid (48%, 40.4 mg, 2.4 mmol) was added at 60 °C for 0.5 h, after completion of the reaction (monitored by TLC), the mixture was concentrated in vacuo. Purification of the residue by column chromatography (petroleum ether/ethyl acetate = 10/1, v/v) afforded the product **5** as a brownish red solid (18.1 mg, 46% yield)^[5].

Synthesis of 6



A mixture of **3ra** (0.057 mmol, 25.4 mg, 1.0 equiv), phenylacetylene (7.0 mg, 1.2 equiv), $Pd(PPh_3)_2Cl_2$ (2.0 mg, 5 mol%), CuI (1.1 mg, 10 mol%), and Et₃N (0.5 mL) were added to an oven dried high pressure tube under Ar atmosphere. The reaction mixture was stirred at 60 °C until raw materials disappear (monitored by TLC). The mixture was quenched with saturated ammonium chloride and extracted with ethyl acetate (3 × 20 mL), the combined organic layer was washed with brine and then the combined organic layers were dried (MgSO4), filtered, and concentrated in vacuo. Purification of the residue by column chromatography (petroleum ether/ethyl acetate = 10/1, v/v) afforded the product 6 as a colorless oil (21.5 mg, 91% yield)^[3].

Synthesis of 7



A mixture of **3xa** (0.056 mmol, 29.0 mg, 1.0 equiv), NaOH (1M in H₂O, 0.17 mL, 3 equiv), THF (0.3 mL), MeOH (0.3 mL) were added to an oven dried high-pressure tube under Ar atmosphere. The reaction mixture was stirred at 50 °C overnight. The mixture was cooled to room temperature, and con HCl (12 M, 14 μ L, 3 equiv) was added and the mixture was concentrated in

vacuo. The mixture was quenched with water and extracted with ethyl acetate $(3 \times 20 \text{ mL})$, the combined organic layer was washed with brine and then the combined organic layers were dried (MgSO4), filtered, and concentrated in vacuo to give the product 7 as a white solid (26.1 mg, 93% yield).

Synthesis of 8



A mixture of **3ja** (0.15 mmol, 79.0 mg, 1.0 equiv), LiOH (18.0 mg, 5 equiv), 1,4-dioxane (0.5 mL), H₂O (0.5 mL) were added to an oven dried high pressure tube under Ar atmosphere. The reaction mixture was stirred at room temperature overnight. The mixture was cooled to 0 °C, and con HCl (12 M, 63 μ L, 5 equiv) was added. The mixture was quenched with water and extracted with ethyl acetate (3 × 20 mL), the combined organic layer was washed with brine and then the combined organic layers were dried (MgSO4), filtered, and concentrated in vacuo to give the crude product acid as a white solid (67.2 mg, 87% yield).

A 25 mL oven-dried tube flask with a stir bar was purged with argon and charged with the crude product acid (0.13 mmol, 67.2 mg) in anhydrous CH₂Cl₂ (1.0 mL), which was added DMF (1 drop) at 0 °C and (COCl)₂ (13.2 μ L, 1.2 equiv) was subsequently added dropwise by syringe. After 15 min, the reaction mixture was allowed to warm to room temperature overnight. After removal of the volatiles under vacuum, the crude product acid chloride was used directly without any further purification. To a solution of the propargylic amine (16.7 μ L, 2 equiv) in anhydrous CH₂Cl₂ (2.0 mL) at 0 °C, Et₃N (27.1 μ L, 1.5 equiv) and DMAP (0.3 mg, 2 mol%) were added, and the acid chloride (1.0 equiv) was added dropwise at 0 °C. After 15 min, the mixture was stirred at room temperature overnight. After the reaction was completed, the mixture was quenched with water and extracted with CH₂Cl₂ (3 × 20 mL), the combined organic layer was washed with brine and then the combined organic layers were dried (MgSO₄), filtered, and concentrated in vacuo. Purification of the residue by column chromatography (petroleum ether/ethyl acetate = 3/1, v/v) afforded the product **8** as a colorless oil (35.0 mg, 41% yield).

6. Mechanism Study

(1) Intermolecular competition experiments

The intermolecular competition reaction between Me- and Cl- substituent on the para-position of aryl ring with 2a under standard conditions reveled that the electron-poor substituent exhibited a little bit better reaction activity in this competition reaction (eq 1). Furthermore, **3ab** and **3ae** showed no obvious reactivity difference in this reaction system, and the little mixed product with alkenes that would come from the two alkynes has been obtained, which was determined by LC-MS, exact mass: 520.2407, found: 520.2. (eq 2).



the little mixed product was determined by LC-MS

(1) H/D exchange experiments

Deuterium-labeling experiments were performed to study the mechanism of this reaction. **1a** (15.9 mg, 0.1 mmol), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) were stirred in toluene (1.0 mL) and D₂O (0.1 mL) under Ar atmosphere at 80 °C for 12 h. After completion, the reaction mixture was purified by column chromatography (petroleum ether/ethyl acetate =10/1, v/v) to afford the product **1a**+[**D**₂]-**1a**. The deuterium rate (78%) was obtained from ¹H NMR. Deuterium was observed at both *ortho*-positions of phenyl ring, which indicated the possibility of the reaction pathway via *ortho* C–H activation.





(2) Kinetic isotope experiments Intermolecular kinetic isotope effect:

The kinetic isotope effect (KIE) study was conducted. Two oven-dried 25 mL tube were separately charged with **1a** (8.0 mg, 0.05 mmol), or **[D5]-1a** (8.2 mg, 0.05 mmol), and (4-methylpent-4-en-1-yn-1-yl)benzene **2a** (19.5 mg, 2.5 equiv) were stirred at 80 °C for 2 h under standard conditions. After completion, the reaction mixture was purified by flash chromatography eluting with ethyl acetate and petroleum ether (1:15) to give **3aa** and **[D]-3aa** in 21% and 10% respectively. The KIE value was determined using isolated yields to give kinetic isotopic effect (KIE) $k_{\rm H}/k_{\rm D} = 2.1$, thus indicating that the first C-H bond cleavage might be involved in the

product determining step.



Intramolecular kinetic isotope effect:

In addition, the kinetic isotope effect (KIE) study was conducted. An oven-dried 25 ml tube **1a** (8.0 mg, 0.05 mmol), [**D**₅]-**1a** (8.2 mg, 0.05 mmol), and (4-methylpent-4-en-1-yn-1-yl)benzene **2a** (39.0 mg, 2.5 equiv) were stirred at 80 °C for 5 h under standard conditions. After completion, the reaction mixture was purified by flash chromatography eluting with ethyl acetate and petroleum ether (1:15) to give **3aa** + [**D**]-**3aa**. The ratio of two products was determined by ¹H NMR integration method to give kinetic isotopic effect (KIE) $k_{\rm H}/k_{\rm D}$ =2.0, thus indicating that the first C-H bond cleavage might be involved in the product determining step.





7. References

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8. X-ray Data of Compound 3ya and Sample Preparation

8.1 Sample preparation and crystal measurement

Single crystals suitable for X-ray diffraction experiment were obtained by slow evaporation of EA/petroleum ether (1:20, V/V) solution containing the corresponding compound **3ya**. The crystal was kept at 298.0 K during data collection. Using Olex2 [1], the structure was solved with the SHELXT [2] structure solution program using Intrinsic Phasing and refined with the SHELXL [3] refinement package using Least Squares minimisation.

8.2 X-ray data of compound 3ya



Figure S1: X-ray structure of 3ya.

Table 1 Crystal data and structure refinement for 3ya.

| Identification code | 3ya |
|------------------------------|------------------------------------|
| Empirical formula | C ₃₃ H ₃₁ NO |
| Formula weight | 457.59 |
| Temperature/K | 298.0 |
| Crystal system | triclinic |
| Space group | P-1 |
| a/Å | 9.8028(4) |
| b/Å | 11.5069(5) |
| c/Å | 12.8022(6) |
| α/° | 65.998(3) |
| β/° | 84.345(3) |
| γ/° | 84.332(3) |
| Volume/Å ³ | 1310.06(10) |
| Z | 2 |
| $\rho_{calc}g/cm^3$ | 1.160 |
| μ/mm^{-1} | 0.529 |
| F(000) | 488.0 |
| Crystal size/mm ³ | $0.53 \times 0.44 \times 0.24$ |
| Radiation | CuKa ($\lambda = 1.54178$) |

| 2Θ range for data collection/ ^c | 7.574 to 134.106 |
|---|---|
| Index ranges | $\textbf{-10} \leq h \leq 11, \textbf{-13} \leq k \leq 13, \textbf{-15} \leq l \leq 15$ |
| Reflections collected | 33779 |
| Independent reflections | $4597 \; [R_{int} = 0.0718, R_{sigma} = 0.0415]$ |
| Data/restraints/parameters | 4597/0/337 |
| Goodness-of-fit on F ² | 1.112 |
| Final R indexes [I>= 2σ (I)] | $R_1 = 0.0556, \mathrm{wR}_2 = 0.1616$ |
| Final R indexes [all data] | $R_1 = 0.0659, wR_2 = 0.1707$ |
| Largest diff. peak/hole / e Å ⁻³ | 0.25/-0.21 |

Table 2 Fractional Atomic Coordinates (×10⁴) and Equivalent Isotropic Displacement Parameters (Å2×10³) for 3ya. Ueq is defined as 1/3 of of the trace of the orthogonalised UIJ tensor.

| Atom | x | у | Z | U(eq) |
|------|-------------|------------|------------|-----------|
| C1 | 6496(2) | 6880(3) | 2929(2) | 82.8(7) |
| C2 | 8926(3) | -609(3) | 3692(3) | 123.5(11) |
| C33 | 6759.9(14) | 1843.4(14) | 4041.8(12) | 41.3(4) |
| C32 | 7991.4(14) | 3482.8(14) | 4237.2(12) | 41.5(4) |
| O3 | 8330.8(18) | 3319(5) | 1640.8(17) | 64.3(9) |
| N4 | 6675(5) | 4693(4) | 1716(4) | 62.1(8) |
| C30 | 2237(2) | 1473(2) | 663(2) | 80.2(7) |
| C31 | 1733(2) | 1795(2) | 1557(2) | 75.3(6) |
| C3 | 2620.6(18) | 1966.7(17) | 2250.5(17) | 60.2(5) |
| C4 | 4035.8(16) | 1812.1(15) | 2060.9(14) | 49.4(4) |
| C5 | 4952.7(15) | 2072.5(15) | 2786.9(13) | 47.4(4) |
| C6 | 6019.4(15) | 1363.3(14) | 3340.9(12) | 43.6(4) |
| C7 | 7386.9(14) | 3003.9(14) | 3550.9(12) | 40.3(4) |
| C8 | 8756.3(15) | 4667.7(14) | 3741.4(12) | 42.2(4) |
| С9 | 9813.6(15) | 4751.7(15) | 2974.6(13) | 46.1(4) |
| C10 | 10771.9(16) | 5775.6(16) | 2421.2(12) | 46.5(4) |
| C11 | 12168.6(17) | 5443.0(19) | 2316.3(15) | 56.2(4) |
| C12 | 13120(2) | 6351(2) | 1833.2(17) | 68.3(5) |
| C13 | 12698(2) | 7613(2) | 1418.8(17) | 69.7(6) |
| C14 | 3613(3) | 1324(2) | 467(2) | 83.6(7) |
| C15 | 4517(2) | 1496(2) | 1152.9(17) | 69.9(5) |
| C16 | 11335(2) | 7964(2) | 1479.5(18) | 73.6(6) |
| C17 | 10364.8(19) | 7061.3(17) | 1968.4(16) | 58.8(5) |
| C18 | 8356.0(16) | 5637.4(16) | 4253.5(13) | 47.9(4) |
| C19 | 6926.2(18) | 6263.1(16) | 4042.0(15) | 55.4(4) |
| C21 | 7912.6(16) | 2781.1(16) | 5419.7(13) | 50.1(4) |

| Atom | x | у | z | U(eq) |
|------|------------|------------|------------|---------|
| C22 | 7325.5(17) | 1626.8(17) | 5899.7(13) | 52.6(4) |
| C23 | 6772.6(16) | 1146.0(16) | 5219.0(13) | 49.9(4) |
| C24 | 7437.5(16) | 3709.8(15) | 2294.2(12) | 45.0(4) |
| C25 | 8092(3) | 4173(3) | 545.9(16) | 84.2(7) |
| C26 | 7089(3) | 4980(2) | 593.9(17) | 84.6(7) |
| C27 | 6467.3(17) | 36.1(16) | 3418.9(16) | 54.7(4) |
| C28 | 7935(2) | -109.2(19) | 2975(2) | 71.2(6) |
| C29 | 8210(2) | 331(3) | 1738(2) | 93.8(8) |
| O3A | 6470(50) | 4760(50) | 1900(30) | 64.3(9) |
| N4A | 8450(60) | 3870(80) | 1540(40) | 62.1(8) |
| C20 | 5981(8) | 6043(17) | 4981(6) | 97(3) |
| C20A | 6323(17) | 6638(16) | 4933(8) | 73(3) |

Table 3 Anisotropic Displacement Parameters (Å $^2 \times 10^3$) for 3ya. The Anisotropicdisplacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U_{11} + 2hka^* b^* U_{12} + ...].$

| Atom | U11 | U22 | U33 | U23 | U13 | U12 |
|------|----------|-----------|----------|-----------|-----------|-----------|
| C1 | 65.2(13) | 109.2(17) | 83.8(14) | -47.6(13) | -21.9(11) | 6.3(12) |
| C2 | 70.4(16) | 149(3) | 152(3) | -65(2) | -31.6(17) | 33.9(18) |
| C33 | 32.6(7) | 44.0(8) | 42.1(8) | -12.3(6) | -2.8(6) | -1.4(6) |
| C32 | 34.0(7) | 48.8(8) | 40.1(8) | -16.1(6) | -5.0(6) | -0.5(6) |
| O3 | 73.7(10) | 75(2) | 43.0(8) | -24.9(10) | 5.0(7) | -1.7(10) |
| N4 | 79(2) | 56.5(11) | 42.6(18) | -12.3(11) | -10.6(13) | 6.2(12) |
| C30 | 81.2(15) | 79.2(14) | 82.6(15) | -26.0(12) | -35.4(12) | -14.3(11) |
| C31 | 48.1(11) | 72.2(13) | 99.0(16) | -22.3(12) | -22.6(10) | -9.1(9) |
| C3 | 49.5(10) | 57.2(10) | 70.4(11) | -20.9(9) | -10.4(8) | -2.3(8) |
| C4 | 46.5(9) | 45.7(8) | 50.0(9) | -10.8(7) | -10.0(7) | -6.5(7) |
| C5 | 44.7(9) | 46.1(8) | 49.0(9) | -15.7(7) | -5.3(7) | -4.7(7) |
| C6 | 36.9(8) | 45.7(8) | 44.0(8) | -13.3(6) | 0.7(6) | -7.2(6) |
| C7 | 34.5(7) | 46.2(8) | 37.2(7) | -13.9(6) | -2.9(6) | -1.0(6) |
| C8 | 38.0(8) | 50.5(9) | 38.1(7) | -17.0(6) | -7.1(6) | -2.5(6) |
| С9 | 44.2(8) | 54.2(9) | 44.1(8) | -23.2(7) | -3.7(6) | -5.4(7) |
| C10 | 45.0(8) | 60.1(10) | 38.4(8) | -23.3(7) | 0.6(6) | -8.9(7) |
| C11 | 47.3(9) | 67.4(11) | 57.9(10) | -29.7(8) | 2.3(7) | -6.9(8) |
| C12 | 48.9(10) | 94.6(15) | 68.2(12) | -39.3(11) | 7.3(8) | -17.6(10) |
| C13 | 66.4(12) | 83.8(15) | 62.9(11) | -31.9(10) | 17.0(9) | -32.5(10) |
| C14 | 87.5(16) | 105.9(18) | 69.6(13) | -43.9(13) | -17.9(11) | -10.2(13) |
| C15 | 56.0(11) | 96.3(15) | 60.9(11) | -33.9(11) | -6.1(8) | -7.8(10) |
| C16 | 81.9(15) | 60.1(11) | 69.9(12) | -18.1(10) | 11.4(10) | -15.2(10) |
| C17 | 51.5(10) | 60.2(11) | 58.2(10) | -17.5(8) | 1.7(8) | -6.2(8) |

Table 3 Anisotropic Displacement Parameters (Å2×103) for 3ya. The Anisotropicdisplacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+...]$.

| Atom | U11 | U ₂₂ | U ₃₃ | U ₂₃ | U ₁₃ | U ₁₂ |
|------|-----------|-----------------|-----------------|-----------------|-----------------|-----------------|
| C18 | 46.1(9) | 59.1(9) | 44.2(8) | -26.0(7) | -3.3(6) | -5.6(7) |
| C19 | 52.8(10) | 55.6(10) | 61.3(10) | -27.6(8) | -5.2(8) | 0.3(8) |
| C21 | 46.2(9) | 63.4(10) | 38.3(8) | -16.6(7) | -8.4(6) | -3.2(7) |
| C22 | 48.4(9) | 61.3(10) | 36.5(8) | -6.8(7) | -6.2(6) | -3.5(7) |
| C23 | 42.8(9) | 52.5(9) | 43.9(8) | -7.9(7) | -2.3(6) | -6.4(7) |
| C24 | 48.9(9) | 47.4(9) | 38.4(8) | -15.0(7) | -2.1(6) | -12.0(7) |
| C25 | 107.7(19) | 103.2(18) | 36.8(10) | -23.2(10) | 6.7(10) | -18.0(15) |
| C26 | 124(2) | 75.1(14) | 39.7(10) | -1.6(9) | -21.1(11) | -16.6(14) |
| C27 | 49.1(9) | 47.6(9) | 64.4(10) | -18.4(8) | -6.6(8) | -4.7(7) |
| C28 | 52.2(11) | 59.8(11) | 109.6(17) | -42.6(11) | -10.3(11) | 3.5(8) |
| C29 | 71.8(15) | 108.6(19) | 116(2) | -64.9(16) | 20.7(13) | -9.5(13) |
| O3A | 73.7(10) | 75(2) | 43.0(8) | -24.9(10) | 5.0(7) | -1.7(10) |
| N4A | 79(2) | 56.5(11) | 42.6(18) | -12.3(11) | -10.6(13) | 6.2(12) |
| C20 | 69(3) | 117(8) | 90(3) | -34(4) | 13(2) | 19(4) |
| C20A | 77(6) | 61(6) | 71(4) | -25(4) | 9(4) | 18(5) |

Table 4 Bond Lengths for 3ya.

| Atom | n Atom | Length/Å | Atom | n Atom | Length/Å |
|------|--------|----------|------|--------|-----------|
| C1 | C19 | 1.397(3) | C8 | C18 | 1.512(2) |
| C2 | C28 | 1.324(3) | C9 | C10 | 1.477(2) |
| C33 | C6 | 1.499(2) | C10 | C11 | 1.393(2) |
| C33 | C7 | 1.400(2) | C10 | C17 | 1.384(2) |
| C33 | C23 | 1.390(2) | C11 | C12 | 1.377(3) |
| C32 | C7 | 1.410(2) | C12 | C13 | 1.364(3) |
| C32 | C8 | 1.493(2) | C13 | C16 | 1.362(3) |
| C32 | C21 | 1.394(2) | C14 | C15 | 1.387(3) |
| 03 | C24 | 1.324(3) | C16 | C17 | 1.388(3) |
| 03 | C25 | 1.373(4) | C18 | C19 | 1.508(2) |
| N4 | C24 | 1.283(5) | C19 | C20 | 1.395(7) |
| N4 | C26 | 1.365(5) | C19 | C20A | 1.433(10) |
| C30 | C31 | 1.375(4) | C21 | C22 | 1.375(2) |
| C30 | C14 | 1.355(3) | C22 | C23 | 1.380(2) |
| C31 | C3 | 1.383(3) | C24 | O3A | 1.41(5) |
| C3 | C4 | 1.391(2) | C24 | N4A | 1.28(5) |
| C4 | C5 | 1.485(2) | C25 | C26 | 1.299(3) |
| C4 | C15 | 1.381(3) | C25 | N4A | 1.25(5) |
| C5 | C6 | 1.330(2) | C26 | O3A | 1.65(3) |

Table 4 Bond Lengths for 3ya.

| Aton | n Atom | Length/Å | Aton | n Atom | Length/Å |
|------|--------|----------|------|--------|----------|
| C6 | C27 | 1.512(2) | C26 | N4A | 1.89(5) |
| C7 | C24 | 1.476(2) | C27 | C28 | 1.513(3) |
| C8 | С9 | 1.338(2) | C28 | C29 | 1.459(3) |

Table 5 Bond Angles for 3ya.

| Atom Atom Atom | | n Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|----------------|-----|--------|------------|------|------|------|------------|
| C7 | C33 | C6 | 121.76(12) | C4 | C15 | C14 | 120.9(2) |
| C23 | C33 | C6 | 119.16(14) | C13 | C16 | C17 | 121.2(2) |
| C23 | C33 | C7 | 119.01(14) | C10 | C17 | C16 | 120.25(17) |
| C7 | C32 | C8 | 122.52(13) | C19 | C18 | C8 | 115.53(12) |
| C21 | C32 | C7 | 117.91(14) | C1 | C19 | C18 | 120.64(17) |
| C21 | C32 | C8 | 119.51(13) | C1 | C19 | C20A | 120.4(4) |
| C24 | 03 | C25 | 104.0(3) | C20 | C19 | C1 | 120.3(3) |
| C24 | N4 | C26 | 105.6(4) | C20 | C19 | C18 | 118.4(3) |
| C14 | C30 | C31 | 119.37(18) | C20A | C19 | C18 | 113.9(6) |
| C30 | C31 | C3 | 120.4(2) | C22 | C21 | C32 | 121.19(14) |
| C31 | C3 | C4 | 120.8(2) | C21 | C22 | C23 | 120.51(14) |
| C3 | C4 | C5 | 119.01(16) | C22 | C23 | C33 | 120.37(15) |
| C15 | C4 | C3 | 117.68(15) | O3 | C24 | C7 | 119.6(2) |
| C15 | C4 | C5 | 123.19(15) | N4 | C24 | O3 | 113.05(18) |
| C6 | C5 | C4 | 129.24(15) | N4 | C24 | C7 | 127.3(3) |
| C33 | C6 | C27 | 116.44(13) | O3A | C24 | C7 | 114.5(14) |
| C5 | C6 | C33 | 118.38(14) | N4A | C24 | C7 | 130.5(19) |
| C5 | C6 | C27 | 124.88(14) | N4A | C24 | O3A | 109(3) |
| C33 | C7 | C32 | 120.87(13) | C26 | C25 | O3 | 108.6(2) |
| C33 | C7 | C24 | 118.94(13) | N4A | C25 | C26 | 96(3) |
| C32 | C7 | C24 | 120.18(13) | C25 | C26 | N4 | 108.7(3) |
| C32 | C8 | C18 | 115.99(13) | C25 | C26 | O3A | 114.7(16) |
| C9 | C8 | C32 | 119.13(14) | C25 | C26 | N4A | 41.1(18) |
| C9 | C8 | C18 | 124.54(14) | O3A | C26 | N4A | 76(2) |
| C8 | C9 | C10 | 129.29(14) | C6 | C27 | C28 | 114.31(14) |
| C11 | C10 | С9 | 118.82(15) | C2 | C28 | C27 | 120.6(3) |
| C17 | C10 | С9 | 123.89(15) | C2 | C28 | C29 | 121.8(2) |
| C17 | C10 | C11 | 117.26(16) | C29 | C28 | C27 | 117.61(18) |
| C12 | C11 | C10 | 121.74(18) | C24 | O3A | C26 | 86.8(17) |
| C13 | C12 | C11 | 119.92(19) | C24 | N4A | C26 | 81(2) |
| C16 | C13 | C12 | 119.53(19) | C25 | N4A | C24 | 114(4) |
| C30 | C14 | C15 | 120.9(2) | C25 | N4A | C26 | 43.0(15) |

Table 6 Torsion Angles for 3ya.

| A | В | С | D | Angle/° | A | B | С | D | Angle/° |
|-----|-----|------|-----|-------------|-----|-----|-----|------|-------------|
| C33 | C6 | C27 | C28 | -63.7(2) | C8 | C9 | C10 | C17 | -44.9(2) |
| C33 | C7 | C24 | 03 | -76.2(3) | C8 | C18 | C19 | C1 | -54.8(2) |
| C33 | C7 | C24 | N4 | 102.5(3) | C8 | C18 | C19 | C20 | 115.3(9) |
| C33 | C7 | C24 | O3A | 102(2) | C8 | C18 | C19 | C20A | 150.2(9) |
| C33 | C7 | C24 | N4A | -107(6) | C9 | C8 | C18 | C19 | 120.22(17) |
| C32 | C7 | C24 | 03 | 102.4(3) | C9 | C10 | C11 | C12 | -178.48(15) |
| C32 | C7 | C24 | N4 | -79.0(3) | C9 | C10 | C17 | C16 | 178.90(16) |
| C32 | C7 | C24 | O3A | -79(2) | C10 | C11 | C12 | C13 | -1.5(3) |
| C32 | C7 | C24 | N4A | 72(6) | C11 | C10 | C17 | C16 | -2.8(3) |
| C32 | C8 | C9 | C10 | -175.88(13) | C11 | C12 | C13 | C16 | -0.5(3) |
| C32 | C8 | C18 | C19 | -66.55(18) | C12 | C13 | C16 | C17 | 0.8(3) |
| C32 | C21 | C22 | C23 | -1.3(2) | C13 | C16 | C17 | C10 | 0.9(3) |
| 03 | C25 | 5C26 | N4 | 1.5(4) | C14 | C30 | C31 | C3 | 0.0(3) |
| C30 | C31 | C3 | C4 | -0.3(3) | C15 | C4 | C5 | C6 | -52.8(3) |
| C30 | C14 | C15 | C4 | 0.7(4) | C17 | C10 | C11 | C12 | 3.1(2) |
| C31 | C30 | C14 | C15 | -0.2(4) | C18 | C8 | C9 | C10 | -2.9(2) |
| C31 | C3 | C4 | C5 | 176.79(17) | C21 | C32 | C7 | C33 | -1.6(2) |
| C31 | C3 | C4 | C15 | 0.7(3) | C21 | C32 | C7 | C24 | 179.86(13) |
| C3 | C4 | C5 | C6 | 131.29(19) | C21 | C32 | C8 | C9 | 121.28(16) |
| C3 | C4 | C15 | C14 | -0.9(3) | C21 | C32 | C8 | C18 | -52.33(18) |
| C4 | C5 | C6 | C33 | -178.72(15) | C21 | C22 | C23 | C33 | -2.2(2) |
| C4 | C5 | C6 | C27 | -5.2(3) | C23 | C33 | C6 | C5 | 117.18(17) |
| C5 | C4 | C15 | C14 | -176.82(19) | C23 | C33 | C6 | C27 | -56.90(19) |
| C5 | C6 | C27 | C28 | 122.70(19) | C23 | C33 | C7 | C32 | -1.7(2) |
| C6 | C33 | 5C7 | C32 | 175.35(13) | C23 | C33 | C7 | C24 | 176.78(14) |
| C6 | C33 | 5C7 | C24 | -6.1(2) | C24 | 03 | C25 | C26 | -1.4(4) |
| C6 | C33 | C23 | C22 | -173.48(14) | C24 | N4 | C26 | C25 | -0.9(4) |
| C6 | C27 | C28 | C2 | 99.6(3) | C25 | 03 | C24 | N4 | 0.9(4) |
| C6 | C27 | C28 | C29 | -79.9(2) | C25 | 03 | C24 | C7 | 179.7(2) |
| C7 | C33 | 6C6 | C5 | -59.91(19) | C25 | C26 | 03A | C24 | -1(3) |
| C7 | C33 | 6C6 | C27 | 126.01(15) | C25 | C26 | N4A | C24 | -141(7) |
| C7 | C33 | C23 | C22 | 3.7(2) | C26 | N4 | C24 | 03 | 0.0(4) |
| C7 | C32 | 2 C8 | С9 | -55.86(19) | C26 | N4 | C24 | C7 | -178.74(19) |
| C7 | C32 | 2 C8 | C18 | 130.53(14) | C26 | C25 | N4A | C24 | 42(7) |
| C7 | C32 | 2C21 | C22 | 3.2(2) | O3A | C24 | N4A | C25 | -49(8) |
| C7 | C24 | O3A | C26 | -179.0(7) | O3A | C24 | N4A | C26 | -21(4) |
| C7 | C24 | N4A | C25 | 159(3) | O3A | C26 | N4A | C24 | 18(4) |
| C7 | C24 | N4A | C26 | -173.0(15) | O3A | C26 | N4A | C25 | 159(4) |

Table 6 Torsion Angles for 3ya.

| Α | B | С | D | Angle/° | Α | В | С | D | Angle/° |
|----|-----|-----|-----|-------------|-----|-----|-----|-----|---------|
| C8 | C32 | C7 | C33 | 175.54(13) | N4A | C24 | O3A | C26 | 24(5) |
| C8 | C32 | C7 | C24 | -3.0(2) | N4A | C25 | C26 | O3A | -23(5) |
| C8 | C32 | C21 | C22 | -174.07(14) | N4A | C26 | O3A | C24 | -16(3) |
| C8 | C9 | C10 | C11 | 136.74(17) | | | | | |

Table 7 Hydrogen Atom Coordinates (Å×10⁴) and Isotropic Displacement Parameters (Å²×10³) for 3ya.

| Atom | x | у | Z | U(eq) |
|------|-------|------|------|-------|
| H1A | 7003 | 7624 | 2520 | 124 |
| H1B | 5533 | 7128 | 2957 | 124 |
| H1C | 6655 | 6313 | 2543 | 124 |
| H2A | 9826 | -688 | 3411 | 148 |
| H2B | 8721 | -882 | 4477 | 148 |
| H30 | 1639 | 1358 | 198 | 96 |
| H31 | 789 | 1899 | 1697 | 90 |
| Н3 | 2267 | 2188 | 2851 | 72 |
| Н5 | 4752 | 2841 | 2865 | 57 |
| H9 | 9967 | 4067 | 2759 | 55 |
| H11 | 12466 | 4585 | 2579 | 67 |
| H12 | 14049 | 6104 | 1790 | 82 |
| H13 | 13338 | 8230 | 1097 | 84 |
| H14 | 3957 | 1102 | -135 | 100 |
| H15 | 5458 | 1399 | 999 | 84 |
| H16 | 11049 | 8826 | 1188 | 88 |
| H17 | 9437 | 7321 | 1992 | 71 |
| H18A | 8441 | 5221 | 5074 | 57 |
| H18B | 9006 | 6297 | 3948 | 57 |
| H21 | 8263 | 3100 | 5892 | 60 |
| H22 | 7301 | 1167 | 6689 | 63 |
| H23 | 6406 | 351 | 5549 | 60 |
| H25 | 8568 | 4174 | -119 | 101 |
| H25A | 8431 | 3908 | -32 | 101 |
| H26 | 6715 | 5644 | -32 | 101 |
| H27A | 5862 | -195 | 2989 | 66 |
| H27B | 6361 | -558 | 4214 | 66 |
| H29A | 9167 | 163 | 1569 | 141 |
| H29B | 7977 | 1230 | 1380 | 141 |
| H29C | 7668 | -112 | 1450 | 141 |

| Atom | x | У | z | U(eq) |
|------|------|------|------|-------|
| H20A | 5060 | 6308 | 4866 | 117 |
| H20B | 6272 | 5632 | 5721 | 117 |
| H20C | 5640 | 7297 | 4771 | 87 |
| H20D | 6614 | 6223 | 5672 | 87 |

Table 7 Hydrogen Atom Coordinates (Å×10⁴) and Isotropic Displacement Parameters (Å²×10³) for 3ya.

Table 8 Atomic Occupancy for 3ya.

| Atom | Occupancy | Atom | Occupancy | Atom | Occupancy |
|------|-----------|------|-----------|------|-----------|
| 03 | 0.905(14) | N4 | 0.953(16) | H25 | 0.905(14) |
| H25A | 0.047(16) | H26 | 0.953(16) | O3A | 0.095(14) |
| N4A | 0.047(16) | C20 | 0.64(3) | H20A | 0.64(3) |
| H20B | 0.64(3) | C20A | 0.36(3) | H20C | 0.36(3) |
| H20D | 0.36(3) | | | | |

9. Characterization Data and NMR Spectra of Oxazole Derivatives

2-(2,6-bis((*E*)-4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (3aa)



The general procedure was applied to **1a** (8.0 mg, 0.05 mmol), **2a** (19.5 mg, 2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (2.9 mg, 7 mol%), PhOCH₂COOH (11.4 mg, 1.5 equiv), MnO₂ (1.3 mg, 0.3 equiv) in toluene (0.5 mL) at 80 °C for 34 h under Ar. Purification by column chromatography on silica gel

(petroleum ether/ethyl acetate = 20/1, v/v) afforded **3aa** as a white solid (18.8 mg, 80% yield). Melting point: 81.4–82.8°C; ¹ H NMR (400 MHz, CDCl₃): δ = 7.32 (s, 3H), 7.25 – 7.11 (m, 10H), 6.75 (s, 1H), 6.53 (s, 2H), 4.72 (s, 4H), 2.94 (s, 4H), 2.20 (s, 3H), 1.58 (s, 6H); ¹³C NMR (100 MHz, CDCl₃): δ = 159.7, 148.3, 145.5, 142.8, 139.5, 137.7, 132.5, 129.1, 128.6, 128.2, 128.1, 126.7, 125.7, 123.6, 112.5,40.3, 23.2, 10.9. HRMS (ESI): m/z calcd for C₃₄H₃₃ONNa [M+Na]⁺: 494.2454, found: 494.2464.

5-methyl-2-(4-methyl-2,6-bis((*E*)-4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)oxazole (3ba)



The general procedure was applied to **1b** (17.3 mg, 0.1 mmol), **2a** (39.0 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 24 h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3ba** as a colorless oil

(33.1 mg, 68% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.23 (d, *J* = 7.5 Hz, 4H), 7.21 – 7.10 (m, 8H), 6.72 (s, 1H), 6.51 (s, 2H), 4.71 (s, 4H), 2.90 (s, 4H), 2.35 (s, 3H), 2.19 (s, 3H), 1.58 (s, 6H); ¹³C NMR (100 MHz, CDCl₃): δ = 160.0, 148.2, 145.5, 142.9, 139.8, 139.0, 137.9, 132.4, 128.9, 128.7, 128.2, 126.7, 123.6, 123.0, 112.4, 40.3, 23.3, 21.7, 11.0. HRMS (ESI): m/z calcd for C₃₅H₃₅ONNa [M+Na]⁺: 508.2611, found: 508.2614.

2-(4-methoxy-2,6-bis((*E*)-4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (3ca)



The general procedure was applied to **1c** (18.9 mg, 0.1 mmol), **2a** (39.0 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 25 h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3ca** as a white solid

(31.7 mg, 63% yield). Melting point: 61.9–63.2°C; ¹ H NMR (400 MHz, CDCl₃): δ = 7.35 – 7.28 (m, 4H), 7.25 – 7.23 (m, 3H), 7.23 – 7.20 (m, 3H), 6.92 (s, 2H), 6.78 (d, *J* = 1.3 Hz, 1H), 6.59 (s, 2H), 4.79 (s, 4H), 3.87 (s, 3H), 2.98 (s, 4H), 2.26 (d, *J* = 1.3 Hz, 3H), 1.66 (s, 6H); ¹³C NMR (100 MHz, CDCl₃): δ = 159.9, 159.6, 148.2, 147.1, 143.0, 139.5, 137.7, 132.5, 128.7, 128.2, 126.8, 123.6, 118.6, 113.6, 112.5, 55.4, 40.3, 23.3, 11.0. HRMS (ESI): m/z calcd for C₃₅H₃₅O₂NNa [M+Na]⁺: 524.2560, found: 524.2563.

2-(4-ethoxy-2,6-bis((E)-4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (3da)



The general procedure was applied to **1d** (20.3 mg, 0.1 mmol), **2a** (39.0 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 25 h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3da** as a white solid

(31.5 mg, 61% yield). Melting point: 71.4–72.6°C; ¹ H NMR (400 MHz, CDCl₃): δ = 7.34 – 7.29 (m, 4H), 7.26 – 7.20 (m, 6H), 6.92 (s, 2H), 6.78 (d, J = 1.4 Hz, 1H), 6.59 (s, 2H), 4.79 (s, 4H), 4.10 (q, *J* = 7.0 Hz, 2H), 2.98 (s, 4H), 2.27 (d, *J* = 1.2 Hz, 3H), 1.66 (s, 6H), 1.46 (t, *J* = 7.0 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ = 160.0, 159.0, 148.2, 147.1, 143.0, 139.6, 137.8, 132.4, 128.7, 128.2, 126.8, 123.5, 118.4, 114.1, 112.5, 63.6, 40.3, 23.3, 14.9, 11.0. HRMS (ESI): m/z calcd for C₃₆H₃₇O₂NNa [M+Na]⁺: 538.2717, found: 538.2721.

2-(4-isopropyl-2,6-bis((*E*)-4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (3ea)



The general procedure was applied to **1e** (20.1 mg, 0.1 mmol), **2a** (39.0 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 40 h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3ea** as a colorless oil

(26.7 mg, 52% yield). ¹ H NMR (400 MHz, CDCl₃): $\delta = 7.35 - 7.28$ (m, 4H), 7.26 - 7.19 (m, 8H), 6.79 (d, J = 1.4 Hz, 1H), 6.57 (s, 2H), 4.77 (s, 4H), 3.05 - 2.92 (m, 5H), 2.27 (d, J = 1.2 Hz, 3H), 1.65 (s, 6H), 1.31 (d, J = 6.9 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃): $\delta = 160.1$, 149.7, 148.2, 145.4, 143.1, 139.9, 137.9, 132.3, 128.7, 128.2, 126.7, 126.3, 123.5, 123.3, 112.4, 40.4, 34.1, 23.9, 23.3, 11.0. HRMS (ESI): m/z calcd for C₃₇H₃₉ONNa [M+Na]⁺: 536.2924, found: 536.2927.

3,5-bis((*E*)-4-methyl-1-phenylpenta-1,4-dien-2-yl)-4-(5-methyloxazol-2-yl)benzonitrile (3fa)



The general procedure was applied to **1f** (18.4 mg, 0.1 mmol), **2a** (39.0 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 25 h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 15/1, v/v) afforded **3fa** as a colorless oil

(31.1 mg, 63% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.64 (s, 2H), 7.33 (t, *J* = 7.3 Hz, 4H), 7.28 – 7.26 (m, 1H), 7.26 – 7.20 (m, 5H), 6.86 (d, *J* = 1.4 Hz, 1H), 6.58 (s, 2H), 4.76 (d, *J* = 31.7 Hz, 4H), 2.96 (s, 4H), 2.30 (d, *J* = 1.2 Hz, 3H), 1.64 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 157.9, 149.2, 146.6, 142.2, 137.8, 137.0, 133.9, 131.5, 130.3, 128.6, 128.4, 127.3, 124.1, 118.7, 113.1, 40.1, 23.2, 11.0. HRMS (ESI): m/z calcd for C₃₅H₃₂ON₂Na [M+Na]⁺: 519.2407, found: 519.2411.

2-(2,6-bis((*E*)-4-methyl-1-phenylpenta-1,4-dien-2-yl)-4-(trifluoromethyl)phenyl)-5-methyloxa zole (3ga)



The general procedure was applied to **1g** (22.7 mg, 0.1 mmol), **2a** (39.0 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 51 h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3ga** as a colorless oil

(34.6 mg, 64% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.61 (s, 2H), 7.36 – 7.30 (m, 4H), 7.26 – 7.22 (m, 6H), 6.85 (d, *J* = 1.3 Hz, 1H), 6.61 (s, 2H), 4.76 (d, *J* = 20.6 Hz, 4H), 3.00 (s, 4H), 2.30 (d, *J* = 1.2 Hz, 3H), 1.64 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 158.5, 149.0, 146.3, 142.4, 138.5, 137.3, 133.6, 131.1 (q, *J* = 32.1 Hz), 129.3 (d, *J* = 0.9 Hz), 128.7, 128.3, 127.1, 124.8 (q, *J* = 3.8 Hz), 124.0 (q, *J* = 271.3 Hz),123.9, 113.0, 40.2, 23.1, 11.0. ¹⁹F NMR (376 MHz, CDCl₃): δ = -62.79. HRMS (ESI): m/z calcd for C₃₅H₃₂ONF₃Na [M+Na]⁺: 562.2328, found: 562.2338.

2-(4-fluoro-2,6-bis((*E*)-4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (3ha)



The general procedure was applied to **1h** (17.7 mg, 0.1 mmol), **2a** (39.0 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 24 h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3ha** as a colorless oil

(36.7 mg, 75% yield). ¹ H NMR (400 MHz, CDCl₃): $\delta = 7.36 - 7.29$ (m, 4H), 7.26 - 7.20 (m, 6H), 7.10 (d, J = 9.3 Hz, 2H), 6.81 (s, 1H), 6.58 (s, 2H), 4.80 (d, J = 13.5 Hz, 4H), 2.98 (s, 4H), 2.28 (s, 3H), 1.66 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): $\delta = 162.5$ (d, J = 248.2 Hz), 159.0, 148.4, 147.9 (d, J = 8.5 Hz), 142.5, 138.4 (d, J = 1.6 Hz), 137.3, 133.1, 128.5, 128.2, 126.9, 123.6, 122.1 (d, J = 2.8 Hz), 114.9 (d, J = 21.7 Hz), 112.7, 40.1, 23.1, 10.9. ¹⁹F NMR (376 MHz, CDCl₃): $\delta = -111.51$ (t, J = 9.3 Hz). HRMS (ESI): m/z calcd for C₃₄H₃₂ONFNa [M+Na]⁺: 512.2360, found: 512.2362.

2-(4-chloro-2,6-bis((*E*)-4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (3ia)



The general procedure was applied to **1i** (19.3 mg, 0.1 mmol), **2a** (39.0 mg, 2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 18 h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3ia** as a white solid

(38.4 mg, 76% yield). Melting point: 74.1–75.3°C; ¹ H NMR (400 MHz, CDCl₃): δ = 7.29 (s, 2H), 7.23 (d, *J* = 7.4 Hz, 3H), 7.19 – 7.11 (m, 7H), 6.74 (s, 1H), 6.51 (s, 2H), 4.70 (d, *J* = 17.5 Hz, 4H), 2.88 (s, 4H), 2.19 (s, 3H), 1.57 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 158.8, 148.7, 147.1,

142.5, 138.5, 137.4, 135.0, 133.3, 128.6, 128.3, 128.0, 127.0, 124.5, 123.8, 112.8, 40.1, 23.2, 11.0. HRMS (ESI): m/z calcd for C₃₄H₃₂ONClNa [M+Na]⁺: 528.2065, found: 528.2070.

methyl 3,5-bis((*E*)-4-methyl-1-phenylpenta-1,4-dien-2-yl)-4-(5-methyloxazol-2-yl)benzoate (3ja)



The general procedure was applied to **1j** (21.7 mg, 0.1 mmol), **2a** (39.0 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 18 h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3ja** as a white solid

(44.2 mg, 84% yield). Melting point: 104.0–104.9°C; ¹ H NMR (400 MHz, CDCl₃): δ = 7.98 (s, 2H), 7.32 – 7.27 (m, 3H), 7.24 – 7.15 (m, 7H), 6.81 (s, 1H), 6.57 (s, 2H), 4.69 (d, *J* = 8.0 Hz, 4H), 3.92 (s, 3H), 2.92 (s, 4H), 2.25 (s, 3H), 1.57 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 166.8, 158.8, 148.9, 145.8, 142.5, 139.1, 137.5, 133.2, 130.6, 130.0, 129.2, 128.7, 128.3, 126.9, 123.9, 112.8, 52.5, 40.1, 23.2, 11.0. HRMS (ESI): m/z calcd for C₃₆H₃₅O₃NNa [M+Na]⁺: 552.2509, found: 552.2512.

(E)-5-methyl-2-(3-(4-methyl-1-phenylpenta-1,4-dien-2-yl)-[1,1'-biphenyl]-2-yl)oxazole (3ka)



The general procedure was applied to 1k (23.5 mg, 0.1 mmol), 2a (39.0 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 18 h under Ar. Purification by column chromatography on silica gel (petroleum

ether/ethyl acetate = 20/1, v/v) afforded **3ka** as a colorless oil (32.1 mg, 82% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.46 – 7.36 (m, 2H), 7.34 – 7.28 (m, 1H), 7.25 – 7.09 (m, 10H), 6.57 (d, *J* = 14.9 Hz, 2H), 4.71 (s, 2H), 2.91 (s, 2H), 2.06 (s, 3H), 1.58 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.4, 148.3, 145.7, 143.5, 142.8, 141.2, 139.5, 137.8, 132.9, 129.6, 128.9, 128.8, 128.7, 128.5, 128.2, 128.0, 126.9, 126.8, 126.4, 123.3, 112.5, 40.2, 23.3, 10.8. HRMS (ESI): m/z calcd for C₂₈H₂₅ONNa [M+Na]⁺: 414.1828, found: 414.1835.

(E)-2-(2-fluoro-6-(4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (3la)



The general procedure was applied to **11** (17.7 mg, 0.1 mmol), **2a** (39.0 mg, 2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 18 h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl

acetate = 20/1, v/v) afforded **3la** as a colorless oil (29.0 mg, 87% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.47 - 7.40 (m, 1H), 7.40 - 7.33 (m, 2H), 7.32 - 7.27 (m, 4H), 7.14 (t, *J* = 8.9 Hz, 1H), 6.94 (s, 1H), 6.64 (s, 1H), 4.75 (d, *J* = 17.3 Hz, 2H), 3.01 (s, 2H), 2.38 (s, 3H), 1.65 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ = 161.1 (d, *J* = 250.2 Hz), 155.4, 149.5, 146.8, 142.6, 138.7 (d, *J* = 2.3 Hz), 137.5, 133.1, 131.1 (d, *J* = 9.1 Hz), 128.7, 128.3, 127.1, 125.3 (d, *J* = 3.1 Hz), 124.2, 115.7 (d, *J* = 13.9 Hz), 114.6 (d, *J* = 22 Hz), 112.7, 40.0, 23.1, 11.1. ¹⁹F NMR (376 MHz, CDCl₃): δ = -113.13. HRMS (ESI): m/z calcd for C₂₂H₂₀ONFNa [M+Na]⁺: 356.1421, found: 356.1427.

(E)-2-(2-iodo-6-(4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (3ma)



The general procedure was applied to 1m (28.4 mg, 0.1 mmol), 2a (39.0 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 33 h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl

acetate = 20/1, v/v) afforded **3ma** as a white solid (33.8 mg, 77% yield). Melting point: 64.5–65.8°C; ¹ H NMR (400 MHz, CDCl₃): δ = 7.85 (d, *J* = 7.9 Hz, 1H), 7.42 (d, *J* = 7.7 Hz, 1H), 7.31 (t, *J* = 7.5 Hz, 2H), 7.25 – 7.16 (m, 3H), 7.13 (t, *J* = 7.8 Hz, 1H), 6.87 (s, 1H), 6.55 (s, 1H), 4.76 (d, *J* = 15.7 Hz, 2H), 2.94 (s, 2H), 2.36 (s, 3H), 1.63 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ = 160.4, 148.8, 147.3, 142.5, 138.5, 137.9, 137.3, 133.6, 133.1, 131.1, 129.0, 128.6, 128.3, 127.1, 123.5, 112.8, 99.4, 40.0, 23.2, 11.1. HRMS (ESI): m/z calcd for C₂₂H₂₁ONI [M+H]⁺:442.0662, found:442.0661.

(E)-5-methyl-2-(2-methyl-6-(4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)oxazole (3na)



The general procedure was applied to **1n** (17.3 mg, 0.1 mmol), **2a** (39.0 mg, 2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 18 h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl

acetate = 20/1, v/v) afforded **3na** as a colorless oil (19.9 mg, 60% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.36 – 7.27 (m, 4H), 7.25 – 7.18 (m, 4H), 6.86 (d, *J* = 1.5 Hz, 1H), 6.56 (s, 1H), 4.76 (s, 2H), 2.94 (s, 2H), 2.33 (s, 6H), 1.63 (s, 3H).¹³C NMR (100 MHz, CDCl₃): δ = 159.9, 148.5, 145.3, 142.9, 139.8, 138.8, 137.9, 132.5, 129.5, 129.1, 128.6, 128.2, 127.0, 126.9, 126.7, 123.4, 112.5, 40.0, 23.2, 20.5, 11.0. HRMS (ESI): m/z calcd for C₂₃H₂₃ONNa [M+Na]⁺:352.1672, found:352.1677.

2-(3-fluoro-2,6-bis((E)-4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (30a)



The general procedure was applied to **1o** (17.7 mg, 0.1 mmol), **2a** (39.0 mg, 2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 24 h under Ar. Purification by column chromatography on silica gel (petroleum

ether/ethyl acetate = 20/1, v/v) afforded **30a** as a colorless oil (36.5 mg, 75% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.42 – 7.33 (m, 5H), 7.33 – 7.27 (m, 6H), 7.21 (t, *J* = 8.9 Hz, 1H), 6.88 (s, 1H), 6.70 (s, 1H), 6.61 (s, 1H), 4.81 (d, *J* = 12.8 Hz, 2H), 4.69 (d, *J* = 8.3 Hz, 2H), 3.06 (s, 2H), 3.00 (s, 2H), 2.34 (s, 3H), 1.66 (d, *J* = 16.0 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 158.9 (d, *J* = 243.3 Hz), 158.5 (d, *J* = 1.2 Hz), 148.7, 142.7 (d, *J* = 21.7 Hz), 141.5 (d, *J* = 3.7 Hz), 138.9, 137.6 (d, *J* = 21.7 Hz), 134.5, 133.2, 132.8, 132.3 (d, *J* = 17.2 Hz), 129.4 (d, *J* = 8.6 Hz), 128.7 (d, *J* = 19.9 Hz), 128.2 (d, *J* = 2.9 Hz), 126.9 (d, *J* = 7.2 Hz), 123.7, 116.8 (d, *J* = 23.5 Hz), 112.7, 112.6 (d, *J* = 1.6 Hz), 40.3, 23.2, 22.8 (d, *J* = 1.7 Hz), 11.0. ¹⁹F NMR (376 MHz, CDCl₃): δ = -114.17 (dd, *J* = 9.1, 5.3 Hz). HRMS (ESI): m/z calcd for C₃₄H₃₂ONFNa [M+Na]⁺: 512.2360, found: 512.2371.

(E)-5-methyl-2-(5-methyl-2-(4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)oxazole (3pa)



The general procedure was applied to **1p** (17.3 mg, 0.1 mmol), **2a** (39.0 mg, 2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 18 h under Ar.

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3pa** as a colorless oil (23.4 mg, 71% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.68 (s, 1H), 7.31 (d, *J* = 4.4 Hz, 4H), 7.25 – 7.15 (m, 3H), 6.80 (s, 1H), 6.54 (s, 1H), 4.64 (d, *J* = 15.0 Hz, 2H), 3.11 (s, 2H), 2.32 (d, *J* = 27.6 Hz, 6H), 1.55 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ = 161.3, 148.9, 143.0, 140.7, 140.6, 138.1, 137.0, 131.3, 130.5, 130.4, 129.9, 128.7, 128.3, 126.7, 126.0, 124.1, 112.3, 40.7, 23.2, 21.1, 11.1. HRMS (ESI): m/z calcd for C₂₃H₂₃ONNa [M+Na]⁺: 352.1672, found: 352.1677.

(E)-2-(5-bromo-2-(4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (3qa)



The general procedure was applied to 1q (23.6 mg, 0.1 mmol), 2a (39.0 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 18 h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl

acetate = 20/1, v/v) afforded **3qa** as a colorless oil (37.2 mg, 95% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.52 (d, *J* = 8.0 Hz, 1H), 7.33 (d, *J* = 6.7 Hz, 1H), 7.25 – 7.09 (m, 6H), 6.81 (s, 1H), 6.50 (s, 1H), 4.69 (d, *J* = 15.0 Hz, 2H), 2.88 (s, 2H), 2.28 (s, 3H), 1.57 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ = 158.6, 149.0, 147.6, 142.5, 138.4, 137.3, 133.6, 131.5, 130.9, 129.0, 128.6, 128.3, 128.2, 127.1, 124.8, 123.6, 112.8, 39.9, 23.2, 11.1. HRMS (ESI): m/z calcd for C₂₂H₂₀ONBrNa [M+Na]⁺: 416.0620, found: 416.0625.

(E)-2-(5-iodo-2-(4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (3ra)



The general procedure was applied to 1r (28.4 mg, 0.1 mmol), 2a (39.0 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 18 h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl

acetate = 20/1, v/v) afforded **3ra** as a colorless oil (35.5 mg, 80% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 8.13 (d, *J* = 2.0 Hz, 1H), 7.59 (dd, *J* = 8.1, 2.0 Hz, 1H), 7.25 – 7.20 (m, 4H), 7.18 – 7.09 (m, 1H), 6.99 (d, *J* = 8.1 Hz, 1H), 6.73 (s, 1H), 6.46 (s, 1H), 4.54 (d, *J* = 32.4 Hz, 2H), 3.03 (s, 2H), 2.22 (s, 3H), 1.46 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.4, 149.4, 142.8, 142.7, 139.9, 138.4, 137.8, 137.6, 132.4, 131.8, 128.7, 128.4, 128.1, 127.0, 124.4, 112.6, 92.2, 40.5, 23.2, 11.1. HRMS (ESI): m/z calcd for C₂₂H₂₀ONINa [M+Na]⁺: 464.0482, found: 464.0489.

(E)-2-(2,4-dimethoxy-6-(4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (3sa)



The general procedure was applied to **1s** (21.9 mg, 0.1 mmol), **2a** (39.0 mg, 2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 39 h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl

acetate = 5/1, v/v) afforded **3sa** as a white solid (16.0 mg, 43% yield). Melting point: 79.1–80.3°C; ¹ H NMR (400 MHz, CDCl₃): δ = 7.34 – 7.27 (m, 2H), 7.24 – 7.18 (m, 3H), 6.83 (d, *J* = 1.4 Hz, 1H), 6.59 (s, 2H), 6.46 (d, *J* = 2.3 Hz, 1H), 4.76 (s, 2H), 3.86 (s, 3H), 3.80 (s, 3H), 2.91 (s, 2H), 2.31 (d, *J* = 1.2 Hz, 3H), 1.63 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ = 161.5, 159.9, 157.8, 148.6, 147.8, 143.0, 139.3, 137.7, 132.6, 128.7, 128.2, 126.8, 123.6, 112.5, 109.6, 106.3, 97.2, 56.2, 55.5, 39.9, 23.3, 11.2. HRMS (ESI): m/z calcd for C₂₄H₂₅O₃NNa [M+Na]⁺: 398.1727, found: 398.1732.

2-(2,6-bis((E)-4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)-5-(tert-butyl)oxazole (3ta)



The general procedure was applied to **1t** (20.1 mg, 0.1 mmol), **2a** (39.0 mg, 2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 18 h under Ar. Purification by column chromatography on silica gel (petroleum

ether/ethyl acetate = 20/1, v/v) afforded **3ta** as a white solid (36.7 mg, 71% yield). Melting point: 88.2–90.4°C; ¹ H NMR (400 MHz, CDCl₃): δ = 7.37 – 7.30 (m, 3H), 7.25 – 7.12 (m, 10H), 6.70 (s, 1H), 6.55 (s, 2H), 4.76 (d, *J* = 7.3 Hz, 4H), 2.89 (s, 4H), 1.61 (s, 6H), 1.10 (s, 9H). ¹³C NMR (100 MHz, CDCl₃): δ = 160.9, 159.4, 145.6, 143.0, 139.5, 137.7, 132.7, 129.2, 128.7, 128.2, 128.2, 126.8, 125.8, 120.4, 112.4, 40.2, 31.4, 28.8, 23.4. HRMS (ESI): m/z calcd for C₃₇H₃₉ONNa [M+Na]⁺:536.2924, found:536.2931.

2-(2,6-bis((*E*)-4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)-5-phenyloxazole (3ua)



The general procedure was applied to 1u (22.1 mg, 0.1 mmol), 2a (39.0 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 18 h under Ar. Purification by column chromatography on silica gel (petroleum

ether/ethyl acetate = 20/1, v/v) afforded **3ua** as a white solid (35.6 mg, 67% yield). Melting point: 103.4–104.8°C; ¹ H NMR (400 MHz, CDCl₃): δ = 7.59 (d, *J* = 7.5 Hz, 2H), 7.51 – 7.41 (m, 4H), 7.38 – 7.27 (m, 11H), 7.25 – 7.19 (m, 2H), 6.72 (s, 2H), 4.81 (s, 4H), 3.03 (s, 4H), 1.66 (s, 6H); ¹³C NMR (100 MHz, CDCl₃): δ = 160.4, 151.2, 145.7, 142.9, 139.9, 137.7, 132.7, 129.5, 129.0, 128.7, 128.6, 128.4, 128.2, 128.1, 126.8, 125.0, 124.3, 122.9, 112.5, 40.4, 23.3. HRMS (ESI): m/z calcd for C₃₉H₃₅ONNa [M+Na]⁺: 556.2611, found: 556.2619.

2-(2,6-bis((*E*)-4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)-5-(4-methoxyphenyl)oxazole (3va)



The general procedure was applied to 1v (25.1 mg, 0.1 mmol), 2a (39.0 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 18 h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3va** as a white solid (34.7 mg, 62%)

yield). Melting point: 102.1–103.3°C; ¹ H NMR (400 MHz, CDCl₃): δ = 7.50 (d, *J* = 7.0 Hz, 2H), 7.47 – 7.40 (m, 3H), 7.34 – 7.25 (m, 9H), 7.25 – 7.19 (m, 2H), 6.87 (d, *J* = 7.8 Hz, 2H), 6.70 (d, *J* = 1.8 Hz, 2H), 4.80 (s, 4H), 3.82 (s, 3H), 3.01 (s, 4H), 1.64 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.8, 159.7, 151.2, 145.6, 142.9, 140.0, 137.7, 132.6, 129.4, 128.7, 128.6, 128.2, 126.8, 125.8, 125.2, 121.4, 121.0, 114.4, 112.5, 55.4, 40.4, 23.3. HRMS (ESI): m/z calcd for C₄₀H₃₇O₂NNa [M+Na]⁺:586.2717, found:586.2725.

2-(2,6-bis((*E*)-4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)-5-(3-chlorophenyl)oxazole (3wa)



The general procedure was applied to 1w (25.5 mg, 0.1 mmol), 2a (39.0 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 18 h under Ar. Purification by column chromatography on silica gel (petroleum

ether/ethyl acetate = 20/1, v/v) afforded **3wa** as a white solid (30.7 mg, 54% yield). Melting point: 107.8–109.9°C; ¹ H NMR (400 MHz, CDCl₃): δ = 7.52 (s, 1H), 7.45 – 7.35 (m, 5H), 7.25 – 7.19 (m, 10H), 7.16 (t, *J* = 7.1 Hz, 2H), 6.62 (s, 2H), 4.74 (s, 4H), 2.95 (s, 4H), 1.59 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 161.0, 149.8, 145.7, 142.8, 139.7, 137.6, 135.1, 132.8, 130.3, 129.7, 128.7, 128.6, 128.3, 128.3, 126.9, 124.8, 124.2, 123.8, 122.3, 112.6, 40.5, 23.3. HRMS (ESI): m/z calcd for C₃₉H₃₄ONNa [M+Na]⁺:590.2221, found:590.2227.

methyl 2-(2,6-bis((*E*)-4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)oxazole-5-carboxylate (3xa)



The general procedure was applied to 1x (20.3 mg, 0.1 mmol), 2a (39.0 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 39 h under Ar. Purification by column chromatography on silica gel (petroleum

ether/ethyl acetate = 20/1, v/v) afforded **3xa** as a white solid (17.5 mg, 34% yield). Melting point: 91.1–92.3°C; ¹ H NMR (400 MHz, CDCl₃): δ = 7.82 (s, 1H), 7.48 – 7.39 (m, 3H), 7.34 – 7.30 (m, 3H), 7.29 (s, 1H), 7.25 – 7.19 (m, 6H), 6.55 (s, 2H), 4.80 (d, *J* = 4.2 Hz, 4H), 3.87 (s, 3H), 3.03 (s, 4H), 1.66 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 164.4, 158.3, 145.8, 142.6, 142.1, 139.0, 137.4, 134.9, 133.1, 130.0, 128.6, 128.3, 128.1, 127.0, 124.5, 112.8, 52.2, 40.6, 23.3. HRMS (ESI): m/z calcd for C₃₅H₃₄O₃NNa [M+Na]⁺:538.2353, found:538.2359.

2-(2,6-bis((*E*)-4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)oxazole (3ya)

The general procedure was applied to **1y** (14.5 mg, 0.1 mmol), **2a** (39.0 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 18 h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3ya** as a white solid (32.8 mg, 72% yield). Melting point: 85.1–86.9°C; ¹ H NMR (400 MHz, CDCl₃): δ = 7.68 (s, 1H), 7.46 – 7.37 (m, 3H), 7.31 (t, *J* = 7.6 Hz, 4H), 7.25 – 7.19 (m, 7H), 6.56 (s, 2H), 4.80 (s, 4H), 2.98 (s, 4H), 1.66 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 161.4, 145.8, 142.8, 139.4, 138.4, 137.7, 132.8, 129.4, 128.7, 128.2, 128.1, 128.0, 126.8, 125.5, 112.6, 40.4, 23.3. HRMS (ESI): m/z calcd for C₃₃H₃₂ON [M+H]⁺:458.2478, found:458.2481.

2-(2,6-bis((*E*)-4-methyl-1-(p-tolyl)penta-1,4-dien-2-yl)phenyl)-5-methyloxazole (3ab)

The general procedure was applied to 1a (15.9 mg, 0.1 mmol), 2b (42.5 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%),
PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 17 h under Ar. Purification by

column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3ab** as a colorless oil (33.1 mg, 66% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.43 – 7.35 (m, 3H), 7.21 – 7.09 (m, 8H), 6.80 (s, 1H), 6.55 (s, 2H), 4.79 (s, 4H), 3.00 (s, 4H), 2.35 (s, 6H), 2.22 (s, 3H), 1.66 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.9, 148.3, 145.7, 143.0, 138.9, 136.4, 134.9, 132.5, 129.1, 128.9, 128.6, 128.0, 125.8, 123.6, 112.4, 40.4, 23.3, 21.3, 11.0. HRMS (ESI): m/z calcd for C₃₆H₃₈ON [M+H]⁺: 500.2948, found: 500.2949.

$\label{eq:22-(2,6-bis((E)-1-(4-methoxyphenyl)-4-methylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole} 22-(2,6-bis((E)-1-(4-methoxyphenyl)-4-methylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (2,6-bis((E)-1-(4-methoxyphenyl)-4-methylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (2,6-bis((E)-1-(4-methoxyphenyl)-4-methylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (2,6-bis((E)-1-(4-methoxyphenyl)-5-methylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (2,6-bis((E)-1-(4-methoxyphenyl)-5-methylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (2,6-bis((E)-1-(4-methoxyphenyl)-5-methylpenta-1,4-dien-2-yl)phenyl (2,0-2)(2,$



The general procedure was applied to **1a** (15.9 mg, 0.1 mmol), **2c** (46.5 mg, 2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 18 h

under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3ac** as a colorless oil (32.9 mg, 62% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.41 – 7.33 (m, 3H), 7.22 – 7.14 (m, 4H), 6.88 – 6.83 (m, 4H), 6.79 (d, *J* = 1.3 Hz, 1H), 6.51 (s, 2H), 4.79 (s, 4H), 3.81 (s, 6H), 2.97 (s, 4H), 2.26 (d, *J* = 1.2 Hz, 3H), 1.66 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.9, 158.5, 148.3, 145.9, 143.0, 138.1, 132.2, 130.4, 129.9, 129.2, 128.0, 125.8, 123.6, 113.6, 112.3, 55.3, 40.4, 23.4, 11.0. HRMS (ESI): m/z calcd for C₃₆H₃₇O₃NNa [M+Na]⁺: 554.2666, found: 554.2672.

2-(2,6-bis((*E*)-1-(4-(tert-butyl)phenyl)-4-methylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (3ad)



The general procedure was applied to **1a** (15.9 mg, 0.1 mmol), **2d** (53.0 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 19 h under Ar.

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3ad** as a white solid (40.9 mg, 70% yield). Melting point: 112.6–114.7°C. ¹ H NMR (400 MHz, CDCl₃): δ = 7.44 – 7.33 (m, 7H), 7.22 (d, *J* = 8.0 Hz, 4H), 6.80 (s, 1H), 6.57 (s, 2H), 4.82 (s, 4H), 3.02 (s, 4H), 2.30 (m, 3H), 1.70 (s, 6H), 1.35 (s, 18H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.8, 149.6, 148.3, 145.8, 143.0, 138.9, 134.9, 132.5, 129.2, 128.4, 128.1, 125.7, 125.1, 123.6, 112.4, 40.5, 34.6, 31.5, 23.4, 11.1. HRMS (ESI): m/z calcd for C₄₂H₄₉ONNa [M+Na]⁺: 606.3706, found: 606.3710.

2-(2,6-bis((*E*)-1-(4-fluorophenyl)-4-methylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (3ae)



The general procedure was applied to **1a** (15.9 mg, 0.1 mmol), **2e** (43.5 mg, 2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 23 h under Ar. Purification by

column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3ae** as a colorless oil (31.4 mg, 62% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.43 – 7.33 (m, 3H), 7.22 – 7.15 (m, 4H), 7.04 – 6.95 (m, 4H), 6.81 (d, *J* = 1.3 Hz, 1H), 6.51 (s, 2H), 4.76 (d, *J* = 8.7 Hz, 4H), 2.93 (s, 4H), 2.27 (d, *J* = 1.3 Hz, 3H), 1.63 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 161.8 (d, *J* = 244.6 Hz), 159.8, 148.4, 145.5, 142.8, 139.6, 133.7 (d, *J* = 3.2 Hz), 131.5, 130.2 (d, *J* = 7.8 Hz), 129.3, 128.2, 125.7, 123.6, 115.2 (d, *J* = 21.2 Hz), 112.5, 40.3, 23.3, 11.0. ¹⁹F NMR (376 MHz, CDCl₃): δ = -115.49. HRMS (ESI): m/z calcd for C₃₄H₃₂F₂ON [M+H]⁺: 508.2446, found: 508.2454.

2-(2,6-bis((E)-1-(4-chlorophenyl)-4-methylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (3af)



The general procedure was applied to 1a (15.9 mg, 0.1 mmol), 2f (47.5 mg, 2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (5.9 mg, 7

mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 20 h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3af** as a colorless oil (32.9 mg, 61% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.57 (d, *J* = 8.0 Hz, 4H), 7.46 – 7.38 (m, 3H), 7.34 (d, *J* = 8.0 Hz, 4H), 6.83 (s, 1H), 6.58 (s, 2H), 4.78 (d, *J* = 13.8 Hz, 4H), 2.96 (s, 4H), 2.28 (s, 3H), 1.64 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.6, 148.6, 145.2, 142.5, 141.6, 141.3, 131.4, 129.4, 129.0, 128.9, 128.3, 125.6, 125.3 (q, *J* = 3.7 Hz), 123.7, 112.8, 40.5, 23.3, 11.0. HRMS (ESI): m/z calcd for C₃₄H₃₁Cl₂ONNa [M+Na]⁺: 562.1675, found: 562.1676.

2-(2,6-bis((E)-4-methyl-1-(m-tolyl)penta-1,4-dien-2-yl)phenyl)-5-methyloxazole (3ag)



The general procedure was applied to **1a** (15.9 mg, 0.1 mmol), **2g** (42.5 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 34 h under Ar. Purification by column

chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3ag** as a colorless oil (31.5 mg, 63% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.43 – 7.33 (m, 3H), 7.21 (t, J = 7.7 Hz, 2H), 7.10 – 7.02 (m, 6H), 6.81 (s, 1H), 6.55 (s, 2H), 4.76 (s, 4H), 2.98 (s, 4H), 2.34 (s, 6H), 2.29 (s, 3H), 1.64 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.8, 148.3, 145.6, 143.0, 139.5, 137.8, 137.7, 132.7, 129.6, 129.1, 128.1, 127.5, 125.8, 125.7, 123.7, 112.5, 40.4, 23.2, 21.6, 11.0. HRMS (ESI): m/z calcd for C₃₆H₃₇ONNa [M+Na]⁺: 522.2767, found: 536.2777.

2-(2,6-bis((*E*)-1-(3-methoxyphenyl)-4-methylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (3ah)



The general procedure was applied to 1a (15.9 mg, 0.1 mmol), 2h (46.5 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 24 h under Ar. Purification by

column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3ah** as a colorless oil (31.3 mg, 59% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.44 – 7.33 (m, 3H), 7.22 (t, J = 7.9 Hz, 2H), 6.85 – 6.76 (m, 7H), 6.55 (s, 2H), 4.78 (s, 4H), 3.79 (s, 6H), 2.98 (s, 4H), 2.28 (s, 3H), 1.65 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.8, 159.5, 148.4, 145.5, 143.0, 139.8, 139.2, 132.5, 129.2, 129.2, 128.1, 125.8, 123.6, 121.2, 113.9, 112.6, 112.5, 55.2, 40.5, 23.3, 11.0. HRMS (ESI): m/z calcd for C₃₆H₃₇O₃NNa [M+Na]⁺: 554.2666, found: 554.2672.

2-(2,6-bis((E)-1-(3-chlorophenyl)-4-methylpenta-1,4-dien-2-yl)phenyl)-5-methyloxazole (3ai)



The general procedure was applied to **1a** (15.9 mg, 0.1 mmol), **2i** (47.5 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 24 h under Ar. Purification by column

chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3ai** as a colorless oil (29.7 mg, 55% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.45 – 7.33 (m, 3H), 7.25 – 7.17 (m, 6H), 7.11 (d, *J* = 7.3 Hz, 2H), 6.83 (s, 1H), 6.50 (s, 2H), 4.76 (d, *J* = 16.7 Hz, 4H), 2.94 (s, 4H), 2.30 (s, 3H), 1.63 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.6, 148.6, 145.2, 142.6, 141.0,

139.5, 134.1, 131.2, 129.5, 129.3, 128.7, 128.2, 126.9, 126.8, 125.7, 123.8, 112.8, 40.4, 23.2, 11.0. HRMS (ESI): m/z calcd for C₃₄H₃₁ONCl₂Na [M+Na]⁺: 562.1675, found: 562.1681.

2-(2,6-bis((E)-4-methyl-1-(o-tolyl)penta-1,4-dien-2-yl)phenyl)-5-methyloxazole (3aj)



The general procedure was applied to **1a** (15.9 mg, 0.1 mmol), **2j** (42.5 mg, 2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 60 h under Ar. Purification by column

chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3aj** as a colorless oil (13.5 mg, 27% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.44 – 7.39 (m, 1H), 7.35 (s, 1H), 7.34 (d, *J* = 1.5 Hz, 1H), 7.18 – 7.13 (m, 8H), 6.85 (d, *J* = 1.3 Hz, 1H), 6.54 (s, 2H), 4.64 (s, 2H), 4.60 (s, 2H), 2.82 (s, 4H), 2.33 (d, *J* = 0.8 Hz, 3H), 2.23 (s, 6H), 1.53 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.8, 148.5, 144.8, 143.0, 140.5, 137.2, 136.7, 131.4, 129.8, 129.1, 128.9, 128.4, 127.1, 126.0, 125.5, 123.8, 112.8, 40.2, 22.9, 20.1, 11.1. HRMS (ESI): m/z calcd for C₃₆H₃₇ONNa [M+Na]⁺: 522.2767, found: 522.2769.

2-(2,6-bis((*E*)-4-methyl-1-(thiophen-2-yl)penta-1,4-dien-2-yl)phenyl)-5-methyloxazole (3ak)



The general procedure was applied to **1a** (15.9 mg, 0.1 mmol), **2k** (40.5 mg, 2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 42 h under Ar. Purification by column

chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3ak** as a colorless oil (16.9 mg, 35% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.41 – 7.32 (m, 3H), 7.23 (dd, *J* = 5.0, 1.3 Hz, 2H), 7.00 – 6.94 (m, 4H), 6.75 (s, 1H), 6.68 (s, 2H), 4.76 (s, 4H), 3.11 (s, 4H), 2.24 (s, 3H), 1.72 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.5, 148.7, 145.7, 141.7, 140.3, 137.8, 129.5, 128.2, 127.8, 126.9, 125.4, 125.3, 123.7, 111.9, 41.3, 23.4, 11.0. HRMS (ESI): m/z calcd for C₃₀H₂₉ONS₂Na [M+Na]⁺: 506.1583, found: 506.1588.

2-(2,6-bis((*E*)-1-phenylprop-1-en-2-yl)phenyl)-5-methyloxazole (3al)



The general procedure was applied to **1a** (15.9 mg, 0.1 mmol), **2l** (29.0 mg, 2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 13 h under Ar. Purification by column chromatography on silica gel (petroleum

ether/ethyl acetate = 20/1, v/v) afforded **3al** as a white solid (28.2 mg, 72% yield). Melting point: 104.6–105.5°C. ¹ H NMR (400 MHz, CDCl₃): δ = 7.44 – 7.38 (m, 1H), 7.32 – 7.27 (m, 5H), 7.21 – 7.13 (m, 7H), 6.74 (d, *J* = 1.3 Hz, 1H), 6.33 (s, 2H), 2.23 (d, *J* = 1.2 Hz, 3H), 1.97 (d, *J* = 1.5 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.8, 148.5, 147.2, 138.2, 138.1, 129.8, 129.7, 129.0, 128.2, 127.2, 126.5, 125.6, 123.6, 19.8, 11.0. HRMS (ESI): m/z calcd for C₂₈H₂₅ONNa [M+Na]⁺: 414.1828, found: 414.1837.

2-(2,6-bis((*E*)-1-phenylhex-1-en-2-yl)phenyl)-5-methyloxazole (3am)



The general procedure was applied to **1a** (15.9 mg, 0.1 mmol), **2m** (39.5 mg, 2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 17

h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3am** as a colorless oil (30.4 mg, 64% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.47 (t, *J* = 8.4 Hz, 1H), 7.39 – 7.30 (m, 6H), 7.26 – 7.20 (m, 6H), 6.81 (s, 1H), 6.43 (s, 2H), 2.38 – 2.32 (m, 4H), 2.31 (s, 3H), 1.42 – 1.32 (m, 4H), 1.31 – 1.20 (m, 4H), 0.83 (t, *J* = 7.2 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.7, 148.2, 145.7, 143.2, 138.3, 130.1, 129.3, 128.8, 128.2, 127.7, 126.5, 126.5, 123.5, 31.8, 30.5, 22.9, 14.0, 11.0. HRMS (ESI): m/z calcd for C₃₄H₃₇ONNa [M+Na]⁺: 498.2767, found: 498.2764.

2-(2,6-bis((*E*)-1-phenylhex-1-en-2-yl)phenyl)-5-methyloxazole (3an)



The general procedure was applied to **1a** (15.9 mg, 0.1 mmol), **2n** (52.0 mg, 2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 19 h under Ar.

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **3an** as a colorless oil (39.1 mg, 68% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.84 – 7.78 (m, 6H), 7.67 (s, 2H), 7.54 – 7.39 (m, 7H), 7.35 (dd, *J* = 8.5, 1.7 Hz, 2H), 6.82 (d, *J* = 1.3 Hz, 1H), 6.57 (s, 2H), 2.41 (d, *J* = 8 Hz, 4H), 2.31 (d, *J* = 1.2 Hz, 3H), 1.46 – 1.36 (m, 4H), 1.30 – 1.22 (m, 4H), 0.83 (t, *J* = 7.3 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.8, 148.3, 145.8, 143.6, 135.8, 133.5, 132.3, 130.2, 129.4, 128.0, 127.7, 127.6, 127.5, 127.4, 126.6, 126.1, 125.7, 123.6, 31.9, 30.6, 22.9, 14.0, 11.1. HRMS (ESI): m/z calcd for C₄₂H₄₁ONNa [M+Na]⁺: 598.3080, found: 598.3085.

2-(2,6-bis((*E*)-1,5-diphenylpent-1-en-2-yl)phenyl)-5-methyloxazole (3ao)



The general procedure was applied to **1a** (15.9 mg, 0.1 mmol), **2o** (55.0 mg, 2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 21 h under Ar. Purification by column chromatography on silica gel (petroleum

ether/ethyl acetate = 20/1, v/v) afforded **3ao** as a white solid (36.0 mg, 60% yield). Melting point: 57.9–59.8°C. ¹ H NMR (400 MHz, CDCl₃): δ = 7.41 – 7.34 (m, 1H), 7.25 – 7.17 (m, 5H), 7.17 – 7.03 (m, 13H), 7.01 – 6.96 (m, 4H), 6.71 (s, 1H), 6.34 (s, 2H), 2.45 (t, *J* = 7.6 Hz, 4H), 2.33 (t, *J* = 7.6 Hz, 4H), 2.18 (s, 3H), 1.69 – 1.57 (m, 4H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.6, 148.3, 145.5, 142.5, 142.3, 138.0, 130.6, 129.5, 128.8, 128.5, 128.3, 128.2, 127.8, 126.5, 125.7, 123.5, 35.8, 31.5, 30.0, 11.0. HRMS (ESI): m/z calcd for C₄₄H₄₁ONNa [M+Na]⁺: 622.3080, found: 622.3083.

2-(2,6-bis((*E*)-1,5,5-triphenylpent-1-en-2-yl)phenyl)-5-methyloxazole (3ap)



The general procedure was applied to **1a** (15.9 mg, 0.1 mmol), **2p** (74.0 mg, 2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 20 h under Ar. Purification by column chromatography on silica gel (petroleum

ether/ethyl acetate = 20/1, v/v) afforded **3ap** as a white solid (52.6 mg, 70% yield). Melting point: 130.4–131.9°C. ¹ H NMR (400 MHz, CDCl₃): $\delta = \delta$ 7.43 – 7.35 (m, 1H), 7.24 (s, 1H), 7.21 – 7.11 (m, 15H), 7.10 – 7.03 (m, 12H), 7.00 (d, J = 6.7 Hz, 4H), 6.67 (s, 1H), 6.35 (s, 2H), 3.77 (t, J = 7.6 Hz, 2H), 2.38 – 2.27 (m, 4H), 2.19 – 2.04 (m, 7H). ¹³C NMR (100 MHz, CDCl₃): $\delta = 159.4$, 148.1, 145.5, 144.8, 142.2, 137.9, 130.7, 129.4, 128.6, 128.4, 128.2, 127.9, 127.8, 126.6, 126.5, 126.1, 123.5, 51.2, 34.2, 30.6, 11.0. HRMS (ESI): m/z calcd for $C_{56}H_{49}ONNa [M+Na]^+$: 774.3706, found: 774.3712.

2-(2,6-bis((E)-4-methoxy-1-phenylbut-1-en-2-yl)phenyl)-5-methyloxazole (3aq)

The general procedure was applied to **1a** (15.9 mg, 0.1 mmol), **2q** (40.0 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 100 °C for 20 h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 5/1, v/v) afforded **3aq** as a colorless oil (29.7 mg, 62% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.51 – 7.45 (m, 1H), 7.39 – 7.28 (m, 6H), 7.25 – 7.18 (m, 6H), 6.78 (s, 1H), 6.46 (s, 2H), 3.37 (t, *J* = 7.2 Hz, 4H), 3.23 (s, 6H), 2.66 (t, *J* = 7.3 Hz, 4H), 2.28 (d, *J* = 1.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.4, 148.5, 145.0, 138.9, 137.7, 132.1, 129.5, 128.8, 128.3, 127.9, 126.8, 126.6, 123.6, 70.8, 58.6, 32.5, 11.1. HRMS (ESI): m/z calcd for C₃₂H₃₃O₃NNa [M+Na]⁺: 502.2353, found: 502.2357.

2-(2,6-bis((E)-1-phenyl-4-(3-phenylpropoxy)but-1-en-2-yl)phenyl)-5-methyloxazole (3ar)



The general procedure was applied to **1a** (15.9 mg, 0.1 mmol), **2r** (66.0 mg, 2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 36 h under Ar. Purification by column

chromatography on silica gel (petroleum ether/ethyl acetate = 5/1, v/v) afforded **3ar** as a colorless oil (37.1 mg, 54% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.37 – 7.31 (m, 1H), 7.25 – 7.19 (m, 5H), 7.18 – 7.09 (m, 11H), 7.09 – 7.01 (m, 6H), 6.66 (s, 1H), 6.35 (s, 2H), 3.29 (t, *J* = 7.2 Hz, 4H), 3.19 (t, *J* = 6.4 Hz, 4H), 2.58 – 2.48 (m, 8H), 2.16 (s, 3H), 1.75 – 1.65 (m, 4H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.5, 148.5, 145.1, 142.2, 139.2, 137.8, 132.0, 129.5 128.9, 128.6, 128.4, 128.3, 128.0, 126.8, 126.5, 125.8, 123.6, 70.0, 68.9, 32.6, 32.5, 31.5, 11.1. HRMS (ESI): m/z calcd for C₄₈H₄₉O₃NNa [M+Na]⁺: 710.3605, found: 710.3609.

2-(2,6-bis((*E*)-5-(methoxymethoxy)-1-phenylpent-1-en-2-yl)phenyl)-5-methyloxazole (3as)



The general procedure was applied to **1a** (15.9 mg, 0.1 mmol), **2s** (51.0 mg, 2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 17 h under Ar. Purification by column

chromatography on silica gel (petroleum ether/ethyl acetate = 5/1, v/v) afforded **3as** as a colorless oil (36.3 mg, 64% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.49 – 7.43 (m, 1H), 7.38 – 7.28 (m, 6H), 7.24 – 7.15 (m, 6H), 6.78 (s, 1H), 6.40 (s, 2H), 4.48 (s, 4H), 3.41 (t, *J* = 6.5 Hz, 4H), 3.26 (s, 6H), 2.41 (t, *J* = 8 Hz, 4H), 2.28 (s, 3H), 1.71 – 1.58 (m, 4H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.6, 148.4, 145.4, 141.9, 137.9, 130.9, 129.5, 128.8, 128.2, 127.7, 126.7, 126.6, 123.5, 96.4, 67.6, 55.2, 28.6, 28.4, 11.0. HRMS (ESI): m/z calcd for C₃₆H₄₁O₅NNa [M+Na]⁺: 590.2877, found: 590.2881.

2-(2,6-bis((E)-4-(1,3-dioxan-2-yl)-1-phenylbut-1-en-2-yl)phenyl)-5-methyloxazole (3at)



The general procedure was applied to **1a** (15.9 mg, 0.1 mmol), **2t** (54.0 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 19 h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 4/1, v/v) afforded **3at** as a white solid (39.0 mg, 66%)

yield). Melting point: 93.4–95.1°C. ¹ H NMR (400 MHz, CDCl₃): δ = 7.43 (t, *J* = 7.7 Hz, 1H), 7.35 – 7.27 (m, 6H), 7.23 – 7.15 (m, 6H), 6.74 (s, 1H), 6.35 (s, 2H), 4.40 (t, *J* = 5.2 Hz, 2H), 4.00 (dd, *J* = 11.5, 4.8 Hz, 4H), 3.62 (t, *J* = 11.3 Hz, 4H), 2.50 (t, *J* = 7.6 Hz, 4H), 2.26 (s, 3H), 2.07 – 1.94 (m, 2H), 1.77 – 1.63 (m, 4H), 1.26 (d, *J* = 12.9 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.5, 148.2, 145.3, 141.5, 137.9, 130.9, 129.4, 128.8, 128.2, 127.6, 126.9, 126.5, 123.4, 102.0, 66.9, 33.7, 26.5, 25.9, 11.1. HRMS (ESI): m/z calcd for C₃₈H₄₂O₅N [M+H]⁺: 592.3057, found: 592.3067.

di-tert-butyl

4,4'-((1*E*,1'*E*)-(2-(5-methyloxazol-2-yl)-1,3-phenylene)bis(2-phenylethene-1,1-diyl))bis(piperi dine-1-carboxylate) (3au)



The general procedure was applied to **1a** (15.9 mg, 0.1 mmol), **2u** (71.3 mg, 2.5 equiv), Cp*Rh(CH₃CN)₃(SbF₆)₂ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 100 °C for 26 h under Ar. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 4/1,

v/v) afforded **3au** as a white solid (40.8 mg, 56% yield). Melting point: 156.7–157.9 °C. ¹ H NMR (400 MHz, CDCl₃): $\delta = 7.44 - 7.39$ (m, 1H), 7.35 - 7.29 (m, 4H), 7.27 (s, 1H), 7.26 - 7.20 (m, 3H), 7.10 (d, J = 7.5 Hz, 4H), 6.78 (d, J = 1.4 Hz, 1H), 6.42 (s, 2H), 4.21 - 3.92 (m, 4H), 2.82 (tt, J = 12.1, 3.2 Hz, 2H), 2.56 (t, J = 12.8 Hz, 4H), 2.29 (d, J = 1.3 Hz, 3H), 1.61 (d, J = 12.7 Hz, 4H), 1.40 (s, 22H). ¹³C NMR (100 MHz, CDCl₃): $\delta = 159.8, 154.9, 148.1, 144.1, 143.7, 137.5, 132.1, 128.9, 128.6, 128.3, 128.1, 128.1, 126.8, 123.1, 79.4, 43.9, 39.2, 31.0, 28.6, 11.0. HRMS (ESI): m/z calcd for C₄₆H₅₅O₅N₃Na [M+Na]⁺: 752.4034, found: 752.4040.$

2-(2,6-bis((*E*)-1,2-diphenylvinyl)phenyl)-5-methyloxazole (3av)



The general procedure was applied to **1a** (15.9 mg, 0.1 mmol), **2v** (44.5 mg, 2.5 equiv), $Cp*Rh(CH_3CN)_3(SbF_6)_2$ (5.9 mg, 7 mol%), PhOCH₂COOH (22.8 mg, 1.5 equiv), MnO₂ (2.6 mg, 0.3 equiv) in toluene (1.0 mL) at 80 °C for 30 h under Ar. Purification by column chromatography on silica gel (petroleum

ether/ethyl acetate = 8/1, v/v) afforded **3av** as a colorless oil (15.4 mg, 30% yield). ¹ H NMR (400 MHz, CDCl₃): δ = 7.51 – 7.40 (m, 3H), 7.17 – 7.06 (m, 12H), 7.04 – 6.95 (m, 8H), 6.65 (s, 2H), 6.32 (s, 1H), 1.84 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.0, 147.9, 146.4, 141.8, 139.9, 137.5, 130.7, 130.1, 129.6, 129.4, 128.0, 127.9, 126.8, 126.8, 123.0, 10.5. HRMS (ESI): m/z calcd for C₃₈H₃₀ON [M+H]⁺: 516.2322, found: 516.2324.

(4*E*,4'*E*)-4,4'-(2-(5-methyloxazol-2-yl)-1,3-phenylene)bis(5-phenylpent-4-en-1-ol) (4)



¹ H NMR (400 MHz, CDCl₃): δ = 7.49 (t, *J* = 7.7 Hz, 1H), 7.38 – 7.28 (m, 6H), 7.25 – 7.19 (m, 2H), 7.16 (d, *J* = 7.5 Hz, 4H), 6.81 (s, 1H), 6.38 (s, 2H), 3.46 (t, *J* = 6.3 Hz, 4H), 2.45 (t, *J* = 7.5 Hz, 4H), 2.31 (s, 3H), 1.67 – 1.54 (m, 7.10 m), 1.67 – 1.54 (m).

4H). ¹³C NMR (100 MHz, CDCl₃): $\delta = 160.1$, 148.9, 145.5, 141.8, 137.8, 131.0, 129.9, 128.8, 128.3, 127.7, 126.8, 126.5, 123.0, 62.3, 31.3, 28.1, 11.1. HRMS (ESI): m/z calcd for C₃₂H₃₄O₃N [M+H]⁺: 480.2533, found: 480.2530.

2-(2,6-bis((*E*)-4,5-dibromo-4-methyl-1-phenylpent-1-en-2-yl)phenyl)-5-methyloxazole (5)



Melting point: 126.1–127.8 °C. ¹ H NMR (400 MHz, CDCl₃): δ = 7.54 (s, 3H), 7.35 (t, *J* = 7.5 Hz, 4H), 7.25 – 7.19 (m, 6H), 6.96 (s, 1H), 6.71 (s, 2H), 3.68 – 3.58 (m, 4H), 3.15 – 2.96 (m, 4H), 2.41 (s, 3H), 1.56 (d, J = 2.8 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 159.2, 149.0, 149.0, 145.2, 137.5,

137.4, 136.6, 130.0, 130.0, 129.0, 129.0, 128.9, 128.5, 127.1, 125.9, 123.8, 123.8, 66.6, 66.5, 45.1, 42.4, 30.6, 30.6, 11.3.

(*E*)-5-methyl-2-(2-(4-methyl-1-phenylpenta-1,4-dien-2-yl)-5-(phenylethynyl)phenyl)oxazole (6)



¹ H NMR (400 MHz, CDCl₃): δ = 7.99 (s, 1H), 7.51 – 7.38 (m, 3H), 7.32 – 7.21 (m, 8H), 7.20 – 7.13 (m, 1H), 6.76 (s, 1H), 6.53 (s, 1H), 4.56 (d, *J* = 25.5 Hz, 2H), 3.07 (s, 2H), 2.25 (s, 3H), 1.49 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ =160.3, 149.2, 143.2, 142.8, 140.5, 137.8, 132.6, 132.3, 131.8, 130.9, 128.8, 128.5, 128.4, 126.9, 126.5, 124.4, 123.3, 122.4, 112.6, 90.3, 88.7, 40.5, 23.2,

11.2.

2-(2,6-bis((*E***)-4-methyl-1-phenylpenta-1,4-dien-2-yl)phenyl)oxazole-5-carboxylic acid** (7)



Melting point: 124.1–125.8 °C. ¹ H NMR (400 MHz, CDCl₃): δ = 7.88 (s, 1H), 7.47 – 7.33 (m, 3H), 7.25 – 7.12 (m, 9H), 6.51 (s, 2H), 4.75 (d, *J* = 6.2 Hz, 4H), 2.99 (s, 4H), 1.62 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): δ = 165.3, 161.6, 145.9, 142.6, 141.7, 138.8, 137.3, 136.3, 133.3, 130.2, 128.6, 128.3, 128.2, 127.0, 124.2, 112.9, 40.7, 23.3.

3,5-bis((*E*)-4-methyl-1-phenylpenta-1,4-dien-2-yl)-4-(5-methyloxazol-2-yl)-*N*-(prop-2-yn-1-yl)benzamide (8)



¹ H NMR (400 MHz, CDCl₃): $\delta = 7.73$ (s, 2H), 7.36 – 7.29 (m, 4H), 7.28 – 7.21 (m, 6H), 6.84 (s, 1H), 6.60 (s, 2H), 6.30 (s, 1H), 4.74 (d, J = 13.1 Hz, 4H), 4.30 (s, 2H), 2.96 (s, 4H), 2.32 (s, 1H), 2.29 (s, 3H), 1.61 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): $\delta = 166.7$, 158.8, 148.9, 146.1, 142.6, 139.0, 137.5, 134.3, 133.3, 128.7, 128.3, 127.0, 126.7, 123.9, 112.9, 79.5, 72.2, 40.2, 30.1, 23.2, 11.1.

10. ¹H, ¹³C and ¹⁹F NMR spectra



220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 fl (ppm)




















7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85 6.80 6.75 6.70 6.65 6.60 6.55 6.50 6.45 6.40 6.35 f1 (ppm)

 $\mathcal{L}^{7.232}_{7.220}$ $\chi^{7.108}_{7.092}$ -6.584






























































































