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Microwave-Assisted Domino Heck Cyclization and Phosphorylation: Synthesis of Phosphorus Containing Heterocycles

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Abstract: A domino [Pd]-catalyzed intramolecular Heck cyclization followed by intermolecular phosphorylation, for the construction of phosphorus containing heterocyclic compounds, is presented. Notably, the process is accelerated by microwave heating conditions and enabled the construction of C–C and C–P bonds in short reaction times.

Introduction

Organic phosphorus compounds possessing aromatic rings are useful structures and can serve as ligands, nucleotides and pharmaceutically essential molecules.¹ Thus, investigating synthetic methods for the establishment of C-P bond is still of considerable interest and a challenging task. Conventionally, substitution reactions of toxic R2P(O)Cl with reactive organometallic nucleophiles facilitates the formation of C-P bond, under relatively harsh conditions and with poor functional group compatibility.² Notably, in 1982, Hirao et al. for the first time, demonstrated palladium catalyzed phosphorylation of Ar-I/Ar-Br.³ Consequently, the transition-metal mediated transformations became a useful tool for the construction of C(sp²)-P bond.⁴ Later, the functional group directed Pd(II)-catalyzed oxidative C(sp²)-P bond formations via ortho-C-H activation⁵ as well as direct oxidative dehydrogenative couplings have also been found feasible.6

On the other hand, reaction of phosphorus radicals (generated *via* homolytic fission of P–H bond of phosphine oxides in the presence of mild transition-metal salts or peroxides) with radical acceptors has been disclosed, for the accomplishment of organophosphorus compounds.⁷ Recently, the light-induced photocatalytic reaction has also been established as a green protocol.⁸ Particularly, tandem intermolecular radical addition across a radical acceptor and subsequent ring closure reaction of the resulting radical intermediate, is most useful synthetic strategy, because it permits the formation of cyclic products.⁹ Despite

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numerous reports on such a concept, the intermolecular radical phosphorylation over a double bond and intramolecular radical ring closure for the synthesis of phosphorus containing cyclic heterocycles is scarcely explored. For example, the research groups of Shang-Dong Yang reported the synthesis of phosphorylated oxindoles using annulation reaction and radical conditions. While the palladium-catalyzed intramolecular Heck and intermolecular Sonogashira¹⁰ or Suzuki¹¹ or oxidative couplings¹² were found to be efficient cascade processes, for the formation of heterocyclic products. We believe that there is no report using a domino Heck and phosphorylation strategy for the synthesis of fused bicyclic heterocycles containing phosphoryl moiety. In our efforts in developing synthetic domino transformations using transition-metal catalysis, 10a, 10b, 13 we envisaged that palladium catalyzed intramolecular Heck coupling and intermolecular phosphorylation of ortho-arylallyl ethers/orthoarylenamides/ortho-arylallyl amines would be feasible to furnish phosphorylated heterocyclic products. Herein, we present the microwave-assisted tandem Heck and phosphorylation strategy for the construction of 3,3'-disubstituted dihydrobenzofurans and oxindoles possessing a phosphoryl group.

Results and Discussion

We have initiated our screening studies, under microwaveassisted conditions with ortho-iodophenylallyl ether 1a and diphenylphosphine oxide 2a for the possible formation of desired phosphorylated dihydrobenzofuran 3aa. The microwave irradiation (closed Vessel & 300W) of 1a and 2a with Pd(OAc)₂ (5 mol%) and K₂CO₃ (3 equiv) in DMF (0.5 mL) for 15 min at 100 °C, gave a very poor yield of the expected phosphorylated dihydrobenzofuran product 3aa (Table 1, entry 1). There was no betterment upon increasing the irradiation time from 15 to 25 min (Table 1, entry 2). However, yields increased moderately with the use of phosphine ligands (Table 1, entries 3 to 6). Switching the catalyst to Pd(PPh₃)₂Cl₂, did not improve the yield (Table 1, entry 7). To our delight, based on our earlier experience, 5 mol% of catalyst $Pd_2(dba)_3$ and 15 min irradiation time was found to be suitable and furnished the product 3aa in 60% yield (Table 1, entry 8). Elevation of irradiation temperature to 120 °C, has further increased the yield of 3aa to 70% (Table 1, entry 9). While further increase of temperature, found to be inferior (Table 1, entry 10). Changing the base to DBU, gave a poor yield of 3aa (Table 1, entry 11). The product 3aa was isolated in moderate yield in the presence of Pd(PPh₃)₄ catalyst (Table 1, entry 12). On the other hand, attempts made by using water as the solvent

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along with quaternary ammonium salts, were inconclusive (i.e., neither the starting materials were recovered, nor the product was isolated) (Table 1, entries 13 to 16). With triethylamine as base, furnished 40% of **3aa** (Table 1, entry 17). Whereas conventional heating at 120 °C ranging of time period from 20 min to 24 h, resulted in poor yields of **3aa** (Table 1, entry 18 to 21). Further

attempts made with respect to the microwave power, lead to the formation of **3aa** in fair yields (Table 1, entries 22 to 24). It is worth mentioning that when slightly excess amount of **2a** (1.5 to 2.0 equiv) was used with respect to 1 equiv of 1a, aiming to further increase the product 3aa yield, inseparable undesired impurities were noted in their NMR spectra.





^aConditions: 68.5 mg (0.25 mmol) of 1-iodo-2-((2-methylallyl)oxy)benzene **1a**, 50.5 mg (0.25 mmol) of diphenylphosphine oxide **2a**, base (0.75 mmol, 3 equiv), Pdcatalyst (5 mol%) and solvent (0.5 mL). ^bIsolated yields of product **3aa**. ^cNo significant spot was observed on TLC; neither the starting material was recovered, nor any product was isolated. ^aReaction performed using conventional heating conditions. ^cReactions were conducted at different microwave powers.

From the above-optimized conditions (Table 1, entry 9), we have further explored the scope and generality of this domino protocol. To our delight, the process was smooth with 1-iodo-2-((2methylallyl)oxy)benzenes **1a-1h** and diphenylphosphine oxide **2a**

and furnished phosphorylated dihydrobenzofurans **3aa-3gc** (Table 2). For example, different substituents on the aromatic ring of **1b-1d**, such as Me, Et and 'Bu, were amenable and afforded the products in 65 to 70% yields (Table 2, **3ba-3da**). Also, this

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domino reaction was feasible with Ph substituted iodophenyl ether 1e and afforded 3ea in 61% yield.

Table 2: Scope for the synthesis of dihydrobenzofurans 3aa-3gc.^{a,b,c}



^aReaction conditions: All reactions were performed, under microwave irradiation (120 °C, 15 min, 300W, closed Vessel), *ortho*-iodophenyl allyl ethers **1a-1h** (0.25 mmol), diphenylphosphine oxides **2a-2c** (0.25 mmol), Pd₂(dba)₃ (5 mol%), K₂CO₃ (3 equiv), and solvent dimethylformamide (DMF) (0.5 mL), 120 °C, 15 minutes. ^bYields in the parentheses are isolated yields of chromatographically pure products **3aa-3gc**. ^cThe first alphabet of products **3aa-3gc** refers to the *ortho*-iodophenyl allyl ether, while the second letter indicates the diphenylphosphine oxides.

Further, halide substituents on the phenyl ring of iodoallyl ethers **1f-1h**, such as F, Cl, and Br, were well tolerated, under these conditions and furnished the products **3fa-3ha** in 55 to 62% yields. Notably, bromo substituted product **3ha** could be served as a useful synthon for further elaborations, under traditional transition metal catalyzed cross couplings. Furthermore, the protocol was also found suitable with other diarylphosphine oxides **2b-2c** (Table 2, **3ab-3gc**). However, the reaction was found to be sluggish and not successful with dimethylphosphite or diphenylphosphite **3ad** and **3ae** (Table 2). Whereas the reaction

with 2-methylallyl 2-iodobenzoate **3i** and diphenylphosphine oxide **2a** could not furnish the anticipated 6-membered heterocyclic **3ia**.

Encouraged by the successful synthesis of phosphorylated dihydrobenzofurans **3aa-3gc** (Table 2), we aimed to expand this domino strategy, for the synthesis of phosphorylated oxindoles. Thus, *ortho*-iodoarylenamides **4a-4c** were irradiated, under microwave heating along with diarylphosphine oxides **2a-2c** in the presence of Pd₂(dba)₃ catalyst, under standard conditions.

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Table 3: Synthesis of oxindoles 5aa-5cc and indolines 5da-5dc. a,b,c



^aReaction conditions: All reactions were performed under microwave irradiation (120°C, 15 min, 300W, closed Vessel), *ortho*-iodophenyl allyl amines/amides **4a-4d** (0.25 mmol), diphenylphosphine oxides **2a-2c** (0.25 mmol), Pd₂(dba)₃ (5 mol%), K₂CO₃ (3 equiv), and solvent dimethylformamide (DMF) (0.5 mL), 120 °C, 15 minutes. ^bYields in the parentheses are isolated yields of chromatographically pure products **5aa-5dc**. °The first alphabet of products **5aa-5dc** refers to the *ortho*-iodophenyl allyl amine/amide, while the second letter indicates the diphenylphosphine oxides.

To our delight, as anticipated, the protocol was also found compatible and delivered the corresponding phosphorylated oxindoles **5aa-5cc**, in 60 to 71% yield (Table 3). Moreover, to exemplify the utility of present synthetic strategy, preparation of indolines **5da** and **5dc** have also been achieved, albeit in moderate yields (Table 3). Thus, ascertains the scope and applicability of the present domino strategy.

Furthermore, to check the scope and limitation of the method, *ortho*-iodophenylenamide **4e** without α -methyl group was irradiated in the presence of **2a**, under established conditions. As

anticipated, the reaction was impeded right after intramolecular Heck coupling without undergoing the subsequent intermolecular phosphorylation and afforded simple intramolecular Heck coupled product **5e**. This can be reasoned based on the β -hydrogen availability that promotes *syn*-1,2-elimination of bicyclic pallidum species (Scheme 1a). The *E*-geometry of the double bond of **5e** is well conceived by the literature precedence.¹⁴

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Also, to explore the importance of α -methyl group, 1-(allyloxy)-2iodobenzene **4f** was reacted, under standard conditions, in which simple 3-methyl benzofuran **5f** was formed in 60% yield. Based on these observations, it was understood that α -methyl group is essential in order to prevent β -syn elimination of Pd-species of cyclic intermediate and to promote further intermolecular couplings (Scheme 1b).



Scheme 1: Reaction of *ortho*-iodophenyl allyl amide/ether 4e/4f with diphenylphosphine oxide 2a.

Conclusions

In conclusion, we have developed a domino process for the preparation of phosphorylated 3,3-disubstituted bicyclic fused heterocyclic products using [Pd]-catalyzed intramolecular Heck coupling and intermolecular phosphorylation. This tandem C–C and C–P bonds forming process was accelerated by microwave-assisted irradiation conditions.

Experimental Section

General: IR spectra were recorded on a Bruker Tensor 37 (FTIR) spectrophotometer. ¹H NMR spectra were recorded on Bruker Avance 400 (400 MHz) spectrometer at 295 K in CDCl₃; chemical shifts (δ ppm) and coupling constants (Hz) are reported in standard fashion with reference to either internal standard tetramethylsilane (TMS) (δ_{H} =0.00 ppm) or CHCl₃ (δ_{H} = 7.25 ppm). ¹³C NMR spectra were recorded on Bruker Avance 400 (100 MHz) spectrometer at RT in CDCl₃; chemical shifts (δ ppm) are reported relative to CHCl₃ [δ_{C} = 77.00 ppm (central line of triplet)]. In the ¹³C NMR, the nature of carbons (C, CH, CH₂ and CH₃) was

determined by recording the DEPT-135 spectra, and is given in parentheses and noted as s = singlet (for C), d = doublet (for CH), t = triplet (for CH_2) and g = quartet (for CH_3). In the ¹H-NMR, the following abbreviations were used throughout: s = singlet, d = doublet, t = triplet, q = quartet, qui =quintet, sept = septet, dd = doublet of doublet, m = multiplet and br. s = broad singlet. The assignment of signals was confirmed by ¹H, ¹³C CPD and DEPT spectra. High-resolution mass spectra (HR-MS) were recorded on an Agilent 6538 UHD Q-TOF electron spray ionization (ESI) mode and atmospheric pressure chemical ionization (APCI) modes. The microwave irradiation experiments were carried out in a dedicated CEM-Discover monomode microwave apparatus, operating at a frequency of 2.45 GHz with continuous irradiation power from 0 to 300 W and utilization of the standard absorbance level of 100 W. The reactions were carried out in 10 mL glass tubes, sealed with Teflon septum and placed in the microwave cavity. The reactions were irradiated at the required set temperature for the stipulated time and then cooled to ambient temperature with air jet cooling. Reactions were monitored by TLC on silica gel using a combination of hexane and ethyl acetate as eluents. Reactions were generally run under argon or a nitrogen atmosphere. Solvents were distilled prior to use; petroleum ether with a boiling range of 60 to 80 °C was used. The catalyst Pd₂(dba)₃ and the base K₂CO₃ were purchased from Sigma-Aldrich and used as received. The required chemicals such as, ortho-iodophenols, ortho-iodoanilines, allyl halides and diarylphosphine oxides were purchased from Sigma-Aldrich/TCI/local sources and used as received. Acme's silica gel (60-120 mesh) was used for column chromatography (approximately 20 g per one gram of crude material). It is worth noting that these sort of experimental procedures have already been published elsewhere. 10a, 10b, 13

GP (General procedure for the synthesis of 3,3'-Disubstituted Heterocyclic Compounds 3/5): To an oven dried 10 mL glass tube sealed with Teflon septum was equipped with a magnetic stir bar, were added *ortho*-iodoaryl allyl ether **1** *or ortho*iodophenyl allyl amines/enamide **4** (68-94 mg, 0.25 mmol), diarylphosphine oxides **2** (50.5-64.5 mg, 0.25 mmol), followed by Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) and solvent DMF (0.5mL), at room temperature under inert atmosphere. The resultant reaction mixture was subjected to microwave irradiation at 120 °C for 15 min 300 W, closed vessel. Progress of the reaction was monitored by TLC till the reaction is completed. The mixture was cooled to room temperature,

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quenched with aqueous NaHCO₃ solution and extracted with ethyl acetate (3×10 mL). The organic layers were washed with saturated NaCl solution, dried (Na₂SO₄) and filtered. Evaporation of the solvent(s) under reduced pressure and purification of the crude mixture by silica gel column chromatography (petroleum ether/ethyl acetate), furnished the 3,3'-disubstituted phosphorylated heterocyclic compounds **3/5** (up-to 70%) as oil/solid.

[(3-methyl-2,3-dihydro-1-benzofuran-3-

yl)methyl](diphenyl)phosphine oxide (3aa): GP was carried out with 1-iodo-2-[(2-methylprop-2-enyl)oxy]benzene 1a (68.2 mg, 0.25 mmol), diphenylphosphine oxide 2a (50.5 mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 50:50) furnished the product 3aa (60.6 mg, 70%) as a light yellow oily compound, [TLC control (petroleum ether/ethyl acetate 80:20), Rf(1a)=0.9, Rf(3aa)=0.1, UV detection]. IR (MIR-ATR, 4000-600 cm⁻¹): *v_{max}*=3054, 2968, 1595, 1476, 1442, 1178, 1111, 972, 828, 731, 698 cm⁻¹.¹H NMR (CDCl₃, 400 MHz): δ=7.81–7.76 (m, 2H, Ar-H), 7.70-7.64 (m, 2H, Ar-H), 7.50-7.36 (m, 6H, Ar-H), 7.09-7.04 (m, 2H, Ar–H), 6.78 (dd, 1H, J = 7.3 and 1.0 Hz, Ar–H), 6.74 (dd, 1H, J = 7.3 and 1.0 Hz, Ar-H), 4.74 (d, J = 9.3 Hz, 1H, OCH_AH_B), 4.22 (d, J = 9.3 Hz, 1H, OCH_AH_B), 2.72–2.69 (m, 2H, CH₂), 1.40 (s, 3H, CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ =158.6, 135.6 (d, J = 11.7 Hz), 134.4 (d, J = 98.3 Hz), 134.0 (d, J = 97.6 Hz), 131.6 (q, J = 4.2 Hz), 130.5 (d, J = 9.4 Hz), 130.3 (d, J = 9.4 Hz), 128.6 (d, J = 2.5 Hz), 128.5 (d, J = 3.0 Hz), 128.4, 122.5, 120.6, 109.9, 82.6 (d, J = 3.0 Hz), 44.7 (d, J = 3.7 Hz), 39.6 (d, J = 68.4 Hz), 25.8 (d, J = 3.0 Hz) ppm.³¹P NMR (CDCl₃, 162 MHz): δ =27.55 ppm. HR-MS (ESI+) m/z calculated for [C₂₂H₂₂O₂P]⁺=[M+H]⁺: 349.1352; found 349.1335.

(3,5-dimethyl-2,3-dihydro-1-benzofuran-3-

yl)(diphenyl)phosphine oxide (3ba): GP was carried out with 2iodo-4-methyl-1-[(2-methylprop-2-enyl)oxy]benzene **1b** (72 mg, 0.25 mmol), diphenylphosphine oxide **2a** (50.5 mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 50:50) furnished the product **3ba** (62.4 mg, 69%) as a light yellow oily compound, [TLC control (petroleum ether/ethyl acetate 80:20), Rf(1b)=0.9, Rf(3ba)=0.1, UV detection]. IR (MIR-ATR, 4000-600 cm⁻¹): v_{max}=3314, 3052, 2968, 1604, 1477, 1240, 1173, 1114, 974, 822, 735, 696 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): *δ*=7.80–7.75 (m, 2H, Ar-H), 7.69-7.64 (m, 2H, Ar-H), 7.50-7.37 (m, 6H, Ar-H), 6.85–6.83 (m, 2H, Ar–H), 6.63 (d, 1H, J = 7.8 Hz, Ar–H), 4.69 (d, J = 9.3 Hz, 1H, OCH_AH_B), 4.19 (d, J = 9.3 Hz, 1H, OCH_AH_B), 2.79– 2.62 (m, 2H, CH₂), 2.18 (s, 3H, CH₃), 1.42 (s, 3H, CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ=156.6, 135.3 (d, J = 11.7 Hz), 134.5 (d, J = 98.3 Hz), 134.0 (d, J = 97.6 Hz), 131.5 (q, J = 3.0 Hz), 130.5 (d, J = 8.8 Hz), 130.3 (d, J = 9.4 Hz), 129.9, 128.8, 128.6 (d, J = 11.8 Hz), 123.3, 109.5, 83.0 (d, J = 3.9 Hz), 44.7 (d, J = 4.4 Hz), 39.5 (d, J = 68.4 Hz), 25.7 (d, J = 3.0 Hz), 20.7 ppm.³¹P NMR (CDCl₃, 162 MHz): δ=27.68 ppm. HR-MS (ESI+) m/z calculated for [C₂₃H₂₃NaO₂P]⁺=[M+Na]⁺: 385.1328; found 385.1344.

(5-ethyl-3-methyl-2,3-dihydro-1-benzofuran-3-

yl)(diphenyl)phosphine oxide (3ca): GP was carried out with 4ethyl-2-iodo-1-[(2-methylprop-2-enyl)oxy]benzene 1c (75.5 mg, 0.25 mmol), diphenylphosphine oxide 2a (50.5 mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 50:50) furnished the product 3ca (56.4 mg, 60%) as a light yellow oily compound, [TLC control (petroleum ether/ethyl acetate 80:20), R_f(1c)=0.9, R_f(3ca)=0.1, UV detection]. IR (MIR-ATR, 4000-600 cm⁻¹): v_{max}=3417, 3055, 2964, 1488, 1441, 1185, 1115, 977, 813, 705 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ=7.80–7.75 (m, 2H, Ar–H), 7.69-7.64 (m, 2H, Ar-H), 7.51-7.37 (m, 6H, Ar-H), 6.89-6.87 (m, 2H, Ar-H), 6.65 (dd, 1H, J = 7.3 and 1.0 Hz, Ar-H), 4.70 (d, J = 9.3 Hz, 1H, OCH_AH_B), 4.20 (d, J = 9.3 Hz, 1H, OCH_AH_B), 2.79-2.64 (m, 2H, CH₂), 2.49 (q, J = 7.8 Hz, 1H, Ar–CH₂–CH₃), 1.42 (s, 3H, CH₃), 1.15 (t, J = 7.8 Hz, 1H, Ar-CH₂-CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): *δ*=156.8, 136.6, 135.5 (d, *J* = 11.7 Hz), 134.5 (d, J = 98.3 Hz), 134.0 (d, J = 97.6 Hz), 131.5 (q, J = 3.0 Hz), 130.5 (d, J = 8.8 Hz), 130.3 (d, J = 9.4 Hz), 128.6 (d, J = 1.5 Hz), 128.5 (d, J = 1.0 Hz), 127.7, 122.0, 109.6, 82.9 (d, J = 3.4 Hz), 44.8 (d, J = 4.4 Hz), 39.5 (d, J = 68.4 Hz), 28.3, 25.7 (d, J = 3.0 Hz), 16.0 ppm. ³¹P NMR (CDCl₃, 162 MHz): δ=27.84 ppm. HR-MS (ESI+) m/z calculated for $[C_{24}H_{25}NaO_2P]^+=[M+Na]^+$: 399.1484; found 399.1502.

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(5-tert-butyl-3-methyl-2,3-dihydro-1-benzofuran-3-

yl)(diphenyl)phosphine oxide (3da): GP was carried out with 4tert-butyl-2-iodo-1-[(2-methylprop-2-enyl)oxy]benzene 1d (82 mg, 0.25 mmol), diphenylphosphine oxide 2a (50.5 mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 50:50) furnished the product 3da (70.2 mg, 70%) as a light yellow oily compound, [TLC control (petroleum ether/ethyl acetate 80:20), Rf(1d)=0.9, Rf(3da)=0.1, UV detection]. IR (MIR-ATR, 4000-600 cm⁻¹): v_{max}=3405, 3056, 2962, 1602, 1486, 1440, 1182, 1115, 977, 817, 702 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): *δ*=7.81–7.76 (m, 2H, Ar-H), 7.70-7.65 (m, 2H, Ar-H), 7.50-7.38 (m, 6H, Ar-H), 7.12-7.09 (m, 2H, Ar–H), 6.68 (d, 1H, J=9.3 Hz, Ar–H), 4.70 (d, J=9.3 Hz, 1H, OCH_AH_B), 4.19 (d, J = 9.3 Hz, 1H, OCH_AH_B), 2.80–2.66 (m, 2H, CH₂), 1.43 (s, 3H, CH₃), 1.25 (s, 9H, 3 × CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ =156.5, 143.8, 135.3 (d, J = 12.5 Hz), 134.5 (d, J = 99.0 Hz), 134.0 (d, J = 98.3 Hz), 131.6 (t, J = 3.0 Hz), 130.5 (d, J = 9.3 Hz), 130.3 (d, J = 9.3 Hz), 128.7 (d, J = 4.0 Hz), 128.6 (d, J = 4.0 Hz), 125.4, 119.1, 109.2, 82.8 (d, J = 3.4 Hz), 44.9 (d, J = 3.7 Hz), 39.5 (d, J = 67.9 Hz), 34.4, 31.7, 25.8 (d, J = 2.5 Hz) ppm. ³¹P NMR (CDCl₃, 162 MHz): δ=28.27 ppm. HR-MS (ESI+) m/z calculated for [C₂₆H₂₉KO₂P]⁺=[M+K]⁺: 443.1537; found 443.1516.

(3-methyl-5-phenyl-2,3-dihydro-1-benzofuran-3-

yl)(diphenyl)phosphine oxide (3ea): GP was carried out with 3iodo-1,1'-biphenyl-4-yl 2-methylprop-2-enyl ether 1e (87.5 mg, 0.25 mmol), diphenylphosphine oxide 2a (50.5 mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 50:50) furnished the product 3ea (64.6 mg, 61%) as a light yellow solid compound, mp 160-165 °C, [TLC control (petroleum ether/ethyl acetate 80:20), Rf(1e)=0.9, Rf(3ea)=0.1, UV detection]. IR (MIR-ATR, 4000-600 cm⁻¹): vmax=3399, 3057, 2927, 1717, 1468, 1178, 1107, 973, 814, 704 cm⁻¹. ¹H NMR (CDCI₃, 400 MHz): δ=7.86–7.72 (m, 2H, Ar–H), 7.71–7.62 (m, 2H, Ar–H), 7.53–7.34 (m, 10H, Ar–H), 7.32–7.26 (m, 3H, Ar–H), 6.80 (d, 1H, J = 7.8 Hz, Ar–H), 4.83 (d, J = 9.3 Hz, 1H, OCH_AH_B), 4.29 (d, J = 9.3 Hz, 1H, OCH_AH_B), 2.89–2.65 (m, 2H, CH₂), 1.48 (s, 3H, CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ =158.5, 141.1, 136.1 (d, J = 8.8 Hz),

134.3 (d, J = 98.3 Hz), 134.2, 133.9 (d, J = 97.6 Hz), 131.6 (d, J = 2.9 Hz), 130.5 (d, J = 8.9 Hz), 130.3 (d, J = 9.4 Hz), 128.7 (d, J = 3.9 Hz), 128.6, 128.5 (d, J = 5.4 Hz), 127.6, 126.8, 126.6, 121.6, 110.2, 83.2 (d, J = 3.4 Hz), 44.9 (d, J = 3.7 Hz), 39.6 (d, J = 68.4 Hz), 26.0 (d, J = 3.0 Hz) ppm. ³¹P NMR (CDCl₃, 162 MHz): δ =27.67 ppm. HR-MS (ESl+) m/z calculated for [C₂₈H₂₆O₂P]⁺=[M+H]⁺: 425.1665; found 425.1650.

(5-fluoro-3-methyl-2,3-dihydro-1-benzofuran-3-

yl)(diphenyl)phosphine oxide (3fa): GP was carried out with 4fluoro-2-iodo-1-[(2-methylprop-2-enyl)oxy]benzene 1f (73 mg, 0.25 mmol), diphenylphosphine oxide 2a (50.5 mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 50:50) furnished the product 3fa (56.7 mg, 62%) as a light yellow oily compound, [TLC control (petroleum ether/ethyl acetate 80:20), R_f(1f)=0.9, R_f(3fa)=0.1, UV detection]. IR (MIR-ATR, 4000-600 cm⁻¹): *v_{max}*=3055, 2958, 1487, 1257, 1180, 1113, 976, 817, 730, 698 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ=7.80–7.74 (m, 2H, Ar–H), 7.69-7.64 (m, 2H, Ar-H), 7.53-7.38 (m, 6H, Ar-H), 6.77-6.69 (m, 2H, Ar-H), 6.64-6.61 (m, 1H, Ar-H), 4.77 (d, J = 9.3 Hz, 1H, OCH_AH_B), 4.23 (d, J = 9.3 Hz, 1H, OCH_AH_B), 2.75–2.62 (m, 2H, CH₂), 1.40 (s, 3H, CH₃) ppm.¹³C NMR (CDCI₃, 100 MHz): δ=157.5 (d, J_{C-F} = 238.4 Hz), 154.6, 136.6, 134.3 (d, J = 99.0 Hz), 133.8 (d, J = 98.3 Hz), 131.7 (d, J = 2.5 Hz), 130.5 (d, J = 8.9 Hz), 130.4(d, J = 9.3 Hz), 128.8 (d, J = 3.9 Hz), 128.7 (d, J = 3.4 Hz), 114.7 (d, J_{C-F} = 24.6 Hz), 110.3, 110.2, 109.9, 83.4 (d, J = 3.4 Hz), 45.1 (d, J = 1.5 Hz), 39.4 (d, J = 68.4 Hz), 25.8 (d, J = 3.0 Hz) ppm. ³¹P NMR (CDCI₃, 162 MHz): δ=27.24 ppm. HR-MS (ESI+) m/z calculated for $[C_{22}H_{21}FO_2P]^+=[M+H]^+: 367.1258$; found 367.1272.

(5-chloro-3-methyl-2,3-dihydro-1-benzofuran-3-

yl)(diphenyl)phosphine oxide (3ga): GP was carried out with 4chloro-2-iodo-1-[(2-methylprop-2-enyl)oxy]benzene 1g (77 mg, 0.25 mmol), diphenylphosphine oxide 2a (50.5 mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 50:50) furnished the product 3ga (57.3 mg, 60%) as a light yellow oily compound, [TLC control (petroleum ether/ethyl acetate 80:20), $R_f(1g)$ =0.9, $R_f(3ga)$ =0.1, UV detection]. IR (MIR-ATR, 4000–600

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cm⁻¹): ν_{max} =3411, 3057, 2923, 1602, 1468, 1180, 1108, 974, 814, 699 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ =7.80–7.75 (m, 2H, Ar–H), 7.67–7.62 (m, 2H, Ar–H), 7.52–7.37 (m, 6H, Ar–H), 6.99–6.96 (m, 2H, Ar–H), 6.63 (d, 1H, *J* = 8.3 Hz, Ar–H), 4.81 (d, *J* = 9.3 Hz, 1H, OC*H*_AH_B), 4.20 (d, *J* = 9.3 Hz, 1H, OCH_AH_B), 2.76–2.61 (m, 2H, CH₂), 1.39 (s, 3H, CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ =157.5, 137.2 (d, *J* = 11.0 Hz), 134.2 (d, *J* = 100.5 Hz), 133.7 (d, *J* = 98.3 Hz), 131.6 (d, *J* = 2.5 Hz), 130.4 (d, *J* = 9.3 Hz), 130.2 (d, *J* = 9.3 Hz), 128.7 (d, *J* = 6.9 Hz), 128.6 (d, *J* = 6.9 Hz), 128.3, 125.2, 123.1, 110.9, 83.3 (d, *J* = 3.4 Hz) ppm. ³¹P NMR (CDCl₃, 162 MHz): δ =27.23 ppm. HR-MS (ESI+) m/z calculated for [C₂₂H₂₁ClO₂P]⁺=[M+H]⁺: 383.0962; found 383.0977.

(5-bromo-3-methyl-2,3-dihydro-1-benzofuran-3-

yl)(diphenyl)phosphine oxide (3ha): GP was carried out with 4bromo-2-iodo-1-[(2-methylprop-2-enyl)oxy]benzene 1h (88 mg, 0.25 mmol), diphenylphosphine oxide 2a (50.5 mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 50:50) furnished the product 3ha (58.5 mg, 55%) as a light yellow oily compound, [TLC control (petroleum ether/ethyl acetate 80:20), R_f(1h)=0.9, R_f(3ha)=0.1, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): v_{max}=3415, 3058, 2927, 1602, 1472, 1183, 1112, 975, 815, 700 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): *δ*=7.84–7.72 (m, 2H, Ar–H), 7.71-7.59 (m, 2H, Ar-H), 7.55-7.35 (m, 6H, Ar-H), 7.03-6.93 (m, 2H, Ar–H), 6.63 (dd, 1H, J = 7.8 and 1.5 Hz, Ar–H), 4.81 (d, J = 9.3 Hz, 1H, OCHAHB), 4.25 (d, J = 9.3 Hz, 1H, OCHAHB), 2.80-2.59 (m, 2H, CH₂), 1.40 (s, 3H, CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ=157.5, 137.7 (d, J = 101.8 Hz), 137.1, 133.7 (d, J = 98.3 Hz), 131.7 (d, J = 3.0 Hz), 130.5 (d, J = 9.3 Hz), 130.2 (d, J = 9.3 Hz), 128.7 (d, J = 7.4 Hz), 128.6 (d, J = 6.9 Hz), 128.3, 125.2, 123.1, 111.0, 83.3 (d, J = 3.4 Hz), 45.0 (d, J = 3.7 Hz), 39.4 (d, J = 68.4 Hz), 26.0 (d, J = 3.4 Hz) ppm. ³¹P NMR (CDCl₃, 162 MHz): (ESI+) δ=27.30 ppm. HR-MS m/z calculated for $[C_{22}H_{21}^{79}BrO_2P]^+=[M+H]^+$: 427.0457; found 427.0465; [C₂₂H₂₁⁸¹BrO₂P]⁺=[M+H]⁺: 429.0437; found 427.0448.

(3-methyl-2,3-dihydro-1-benzofuran-3-yl)[bis(4-

methylphenyl)]phosphine oxide (3ab): GP was carried out with 1-iodo-2-[(2-methylprop-2-enyl)oxy]benzene 1a (68.2 mg, 0.25 mmol), bis(4-methylphenyl)phosphine oxide 2b (57.5 mg, 0.25

mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 50:50) furnished the product 3ab (60.8 mg, 65%) as a light yellow oily compound, [TLC control (petroleum ether/ethyl acetate 80:20), Rf(1a)=0.9, Rf(3ab)=0.1, UV detection]. IR (MIR-ATR, 4000-600 cm⁻¹): *v_{max}*=3421, 3038, 2966, 1600, 1467, 1177, 1108, 969, 809, 737, 647 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ=7.67 (dd, J = 8.3 and 1.0 Hz, 1H, Ar–H), 7.65 (dd, J = 8.3 and 1.0 Hz, 1H, Ar-H), 7.55 (dd, J = 8.3 and 1.0 Hz, 1H, Ar-H), 7.53 (dd, J = 8.3 and 1.0 Hz, 1H, Ar-H), 7.26 (dd, J = 8.1 and 2.7 Hz, 2H, Ar-H), 7.19 (dd, J = 8.1 and 2.7 Hz, 2H, Ar–H), 7.14–7.02 (m, 2H, Ar–H), 6.85–6.65 (m, 2H, Ar–H), 4.72 (d, J = 9.3 Hz, 1H, OCH_AH_B), 4.22 (d, J = 9.3 Hz, 1H, OCH_AH_B), 2.78–2.55 (m, 2H, CH₂), 2.38 (s, 3H, CH₃), 2.33 (s, 3H, CH₃), 1.39 (s, 3H, CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ=158.6, 142.0, 136.0 (d, J = 12.5 Hz), 131.3 (d, J = 100.5 Hz), 130.9 (d, J = 98.3 Hz), 130.6 (d, J = 9.4 Hz), 130.4 (d, J = 9.8 Hz), 129.5 (d, J = 4.4 Hz), 129.3 (d, J = 4.9 Hz), 128.4, 122.5, 120.6, 110.0, 82.6 (d, J = 2.5 Hz), 44.7 (d, J = 4.4 Hz), 39.7 (d, J = 68.4 Hz), 26.0 (d, J = 2.5 Hz), 21.5 (d, J = 1.0 Hz), 21.4 (d, J = 1.0 Hz) ppm. ³¹P NMR (CDCl₃, 162 MHz): δ =28.03 ppm. HR-MS (ESI+) m/z calculated for [C₂₄H₂₅NaO₂P]⁺=[M+Na]⁺: 399.1484; found 399.1503.

(5-ethyl-3-methyl-2,3-dihydro-1-benzofuran-3-yl)[bis(4-

methylphenyl)]phosphine oxide (3cb): GP was carried out with 4-ethyl-2-iodo-1-[(2-methylprop-2-enyl)oxy]benzene 1c (75.5 mg, 0.25 mmol), bis(4-methylphenyl)phosphine oxide 2b (57.5 mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 50:50) furnished the product 3cb (59.7 mg, 62%) as a light yellow oily compound, [TLC control (petroleum ether/ethyl acetate 80:20), Rf(1c)=0.9, Rf(3cb)=0.1, UV detection]. IR (MIR-ATR, 4000-600 cm⁻¹): v_{max}=3403, 2961, 1731, 1484, 1267, 1180, 1126, 977, 861, 697 cm^{-1.1}H NMR (CDCl₃, 400 MHz): δ =7.67 (dd, J = 7.8 and 1.0 Hz, 1H, Ar-H), 7.64 (dd, J = 7.8 and 1.0 Hz, 1H, Ar-H), 7.56 (dd, J = 7.8 and 1.0 Hz, 1H, Ar–H), 7.53 (dd, J = 7.8 and 1.0 Hz, 1H, Ar–H), 7.26 (dd, J = 8.1 and 2.7 Hz, 2H, Ar–H), 7.20 (dd, J = 8.1 and 2.7 Hz, 2H, Ar-H), 6.96-6.81 (m, 2H, Ar-H), 6.66 (dd, J = 8.8 and 1.0 Hz, 1H, Ar-H), 4.69 (d, J = 9.3 Hz, 1H, OCH_AH_B), 4.20 (d, J = 9.3 Hz, 1H, OCH_AH_B), 2.83–2.57 (m, 2H,

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CH₂), 2.50 (q, J = 7.8 Hz, 1H, Ar– CH_2 –CH₃), 2.38 (s, 3H, CH₃), 2.34 (s, 3H, CH₃), 1.41 (s, 3H, CH₃), 1.16 (t, J = 7.8 Hz, 1H, Ar– CH₂– CH_3) ppm. ¹³C NMR (CDCl₃, 100 MHz): $\bar{\delta}$ =156.7, 141.9 (q, J = 2.9 Hz), 136.5, 135.8 (d, J = 11.7 Hz), 131.4 (d, J = 101.2 Hz), 130.9 (d, J = 100.5 Hz), 130.6 (d, J = 9.4 Hz), 130.5 (d, J = 9.8Hz), 129.4 (d, J = 3.9 Hz), 129.3 (d, J = 3.9 Hz), 127.6, 122.0, 109.6, 82.9 (d, J = 3.4 Hz), 44.8 (d, J = 4.4 Hz), 39.7 (d, J = 68.4Hz), 28.3, 25.9 (d, J = 2.5 Hz), 21.5 (d, J = 1.0 Hz), 21.4 (d, J =1.0 Hz), 16.0 ppm.³¹P NMR (CDCl₃, 162 MHz): $\bar{\delta}$ =28.05 ppm. HR-MS (ESI+) m/z calculated for [C₂₆H₃₀O₂P]⁺=[M+H]⁺: 405.1978; found 405.1991.

(5-tert-butyl-3-methyl-2,3-dihydro-1-benzofuran-3-yl)[bis(4-

methylphenyl)]phosphine oxide (3db): GP was carried out with 4-tert-butyl-2-iodo-1-[(2-methylprop-2-enyl)oxy]benzene 1d (82 mg, 0.25 mmol), bis(4-methylphenyl)phosphine oxide 2b (57.5 mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 50:50) furnished the product 3db (67.6 mg, 63%) as a light yellow oily compound, [TLC control (petroleum ether/ethyl acetate 80:20), Rf(1d)=0.9, Rf(3db)=0.1, UV detection]. IR (MIR-ATR, 4000-600 cm⁻¹): vmax=2957, 1601, 1486, 1263, 1181, 1124, 977,861, 735, 693 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ=7.75–7.62 (m, 2H, Ar–H), 7.61–7.50 (m, 2H, Ar–H), 7.39–7.25 (m, 2H, Ar-H), 7.24-7.17 (m, 2H, Ar-H), 7.16-7.06 (m, 2H, Ar-H), 6.69 (d, 2H, J = 8.3 Hz, Ar-H), 4.70 (d, J = 9.3 Hz, 1H, OCH_AH_B , 4.19 (d, J = 9.3 Hz, 1H, OCH_AH_B), 2.80–2.56 (m, 2H, CH₂), 2.39 (s, 3H, CH₃), 2.35 (s, 3H, CH₃), 1.43 (s, 3H, CH₃), 1.26 (s, 9H, 3 × CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ=156.4, 143.7, 141.9 (q, J = 2.9 Hz), 131.5 (d, J = 12.5 Hz), 131.3 (d, J = 100.5 Hz), 131.0 (d, J = 99.0 Hz), 130.5 (d, J = 9.4 Hz), 130.4 (d, J = 9.8 Hz), 129.4 (d, J = 5.4 Hz), 129.3 (d, J = 5.4 Hz), 125.3, 119.1, 109.1, 82.8 (d, J = 3.4 Hz), 44.9 (d, J = 3.7 Hz), 39.7 (d, J = 68.4 Hz), 34.3, 31.7, 25.9 (d, J = 2.5 Hz), 21.5 (d, J = 1.0 Hz), 21.4 (d, J = 1.0 Hz) ppm. ³¹P NMR (CDCl₃, 162 MHz): δ =28.39 ppm. HR-MS (ESI+) m/z calculated for $[C_{28}H_{34}O_2P]^+=[M+H]^+$: 433.2291; found 433.2311.

bis(4-methylphenyl)(3-methyl-5-phenyl-2,3-dihydro-1-

benzofuran-3-yl)phosphine oxide (3eb): GP was carried out with 3-iodo-1,1'-biphenyl-4-yl 2-methylprop-2-enyl ether 1e (87.5 mg, 0.25 mmol), bis(4-methylphenyl)phosphine oxide 2b (57.5

mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 50:50) furnished the product 3eb (73.4 mg, 65%) as a white solid compound, mp 176-181 °C, [TLC control (petroleum ether/ethyl acetate 80:20), Rf(1e)=0.9, Rf(3eb)=0.1, UV detection]. IR (MIR-ATR, 4000-600 cm⁻¹): vmax=3047, 2925, 1596, 1475, 1397, 1284, 1142, 748, 697 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ=7.66 (dd, J = 7.8 and 1.0 Hz, 1H, Ar–H), 7.63 (dd, J = 7.8 and 1.0 Hz, 1H, Ar-H), 7.55 (dd, J = 7.8 and 1.0 Hz, 1H, Ar-H), 7.52 (dd, J = 7.8 and 1.0 Hz, 1H, Ar-H), 7.49-7.43 (m, 2H, Ar-H), 7.42-7.33 (m, 2H, Ar-H), 7.32-7.10 (m, 7H, Ar-H), 6.80 (d, J = 8.3 Hz, 1H, Ar–H), 4.81 (d, J = 9.3 Hz, 1H, OCH_AH_B), 4.28 (d, J = 9.3 Hz, 1H, OCH_AH_B), 2.85–2.61 (m, 2H, CH₂), 2.36 (s, 3H, CH₃), 2.32 (s, 3H, CH₃), 1.47 (s, 3H, CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ=158.5, 142.0 (q, J = 2.2 Hz), 141.3, 136.4 (d, J = 11.0 Hz), 134.2, 131.3 (d, J = 100.5 Hz), 130.9 (d, J = 99.8 Hz), 130.5 (d, J = 9.4 Hz), 130.4 (d, J = 9.8 Hz), 129.4 (d, J = 6.9 Hz), 129.3 (d, J = 6.9 Hz), 128.6, 127.4, 126.5, 121.5, 110.1, 83.2 (d, J = 3.4 Hz), 44.8 (d, J = 4.4 Hz), 39.8 (d, J = 67.9 Hz), 26.2 (d, J = 3.0 Hz), 21.5 (d, J = 1.0 Hz), 21.4 (d, J = 1.0 Hz) ppm. ³¹P NMR (CDCI₃, 162 MHz): δ=27.99 ppm. HR-MS (ESI+) m/z calculated for $[C_{30}H_{30}O_2P]^+=[M+H]^+$: 453.1978; found 453.1992.

bis(3,5-dimethylphenyl)(3-methyl-2,3-dihydro-1-benzofuran-

3-yl)phosphine oxide (3ac): GP was carried out with 1-iodo-2-[(2-methylprop-2-enyl)oxy]benzene 1a (68.2 mg, 0.25 mmol), bis(3,5-dimethylphenyl)phosphine oxide 2c (64.5 mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 50:50) furnished the product **3ac** (60.3 mg, 60%) as a light vellow oily compound, [TLC control (petroleum ether/ethyl acetate 80:20), Rf(1a)=0.9, Rf(3ac)=0.1, UV detection]. IR (MIR-ATR, 4000-600 cm⁻¹): v_{max}=3030, 2924, 1598, 1459, 1220, 1104, 1026, 914, 803 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ =7.37 (d, J = 1.0 Hz, 1H, Ar– H), 7.35 (d, J = 1.0 Hz, 1H, Ar–H), 7.28 (d, J = 1.0 Hz, 1H, Ar–H), 7.25 (d, J = 1.0 Hz, 1H, Ar-H), 7.16-7.01 (m, 4H, Ar-H), 6.85-6.70 (m, 2H, Ar–H), 4.71 (d, J = 9.3 Hz, 1H, OCH_AH_B), 4.22 (d, J = 9.3 Hz, 1H, OCH_AH_B), 2.82–2.54 (m, 2H, CH₂), 2.33 (s, 6H, 2 × CH₃), 2.29 (s, 6H, 2 × CH₃), 1.43 (s, 3H, CH₃) ppm. 13 C NMR (CDCl₃, 100 MHz): δ=158.7, 138.3 (d, J = 2.2 Hz), 138.2 (d, J =

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1.5 Hz), 135.9 (d, J = 11.7 Hz), 134.4 (d, J = 97.6 Hz), 133.9 (d, J = 96.8 Hz), 133.8 (q, J = 1.5 Hz), 128.4, 128.1 (d, J = 1.5 Hz), 127.9 (d, J = 1.5 Hz), 122.6, 120.5, 109.8, 82.7 (d, J = 3.0 Hz), 44.7 (d, J = 3.7 Hz), 39.5 (d, J = 67.4 Hz), 26.0 (d, J = 2.5 Hz), 21.3, 21.2 ppm. ³¹P NMR (CDCl₃, 162 MHz): δ =27.96 ppm. HR-MS (ESI+) m/z calculated for [C₂₆H₃₀O₂P]⁺=[M+H]⁺: 405.1978; found 405.1996.

(3,5-dimethyl-2,3-dihydro-1-benzofuran-3-yl)[bis(3,5-

dimethylphenyl)]phosphine oxide (3bc): GP was carried out with 2-iodo-4-methyl-1-[(2-methylprop-2-enyl)oxy]benzene 1b (72 mg, 0.25 mmol), bis(3,5-dimethylphenyl)phosphine oxide 2c (64.5 mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 50:50) furnished the product 3bc (64.7 mg, 62%) as a light yellow oily compound, [TLC control (petroleum ether/ethyl acetate 80:20), R_f(1e)=0.9, R_f(3bc)=0.1, UV detection]. IR (MIR-ATR, 4000-600 cm⁻¹): vmax=3393, 2923, 1710, 1485, 1225, 1185, 1109, 1026, 914, 806, 720, 659 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ=7.35 (d, J = 1.0 Hz, 1H, Ar–H), 7.32 (d, J = 1.0 Hz, 1H, Ar-H), 7.27 (d, J = 1.0 Hz, 1H, Ar-H), 7.24 (d, J = 1.0 Hz, 1H, Ar–H), 7.08 (d, J = 1.0 Hz, 1H, Ar–H), 7.05 (d, J = 1.0 Hz, 1H, Ar–H), 6.90–6.76 (m, 2H), 6.64 (d, J = 7.8 Hz, 1H, Ar– H), 4.66 (d, J = 9.3 Hz, 1H, OCH_AH_B), 4.18 (d, J = 9.3 Hz, 1H, OCH_AH_B), 2.72 (dd, J = 15.2 and 11.2 Hz, 1H), 2.59 (dd, J = 15.2 and 11.2 Hz, 1H), 2.32 (s, 6H, 2 × CH₃), 2.28 (s, 6H, 2 × CH₃), 2.17 (s, 3H, CH₃), 1.44 (s, 3H, CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): *δ*=156.7, 138.3 (d, *J* = 4.4 Hz), 138.2 (d, *J* = 4.4 Hz), 135.4 (d, J = 11.0 Hz), 134.6 (d, J = 96.8 Hz), 134.0 (d, J = 96.8 Hz), 133.2 (d, J = 2.0 Hz), 128.7, 128.0 (d, J = 8.9 Hz), 127.7 (d, J = 9.4 Hz), 123.4, 109.3, 83.1 (d, J = 3.9 Hz), 44.8 (d, J = 3.7 Hz), 39.4 (d, J = 67.9 Hz), 25.7 (d, J = 2.5 Hz), 21.3, 21.2, 20.7 ppm. ³¹P NMR (CDCl₃, 162 MHz): *δ*=27.88 ppm. HR-MS (ESI+) m/z for [C₂₇H₃₁NaO₂P]⁺=[M+Na]⁺: 441.1954; found calculated 441.1976.

bis(3,5-dimethylphenyl)(5-ethyl-3-methyl-2,3-dihydro-1-

benzofuran-3-yl)phosphine oxide (3cc): GP was carried out with 4-ethyl-2-iodo-1-[(2-methylprop-2-enyl)oxy]benzene 1c (75.5 mg, 0.25 mmol), bis(3,5-dimethylphenyl)phosphine oxide 2c (64.5 mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C,

15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 50:50) furnished the product 3cc (65.8 mg, 61%) as a light yellow oily compound, [TLC control (petroleum ether/ethyl acetate 80:20), Rf(1c)=0.9, Rf(3cc)=0.1, UV detection]. IR (MIR-ATR, 4000-600 cm⁻¹): vmax=3406, 2961, 2926, 1604, 1487, 1249, 1109, 975, 807, 654 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ =7.36 (d, J = 2.0 Hz, 1H, Ar–H), 7.33 (d, J = 2.0 Hz, 1H, Ar–H), 7.25 (d, J = 2.0 Hz, 1H, Ar-H), 7.09 (d, J = 2.0 Hz, 1H, Ar-H), 7.06 (d, J = 2.0 Hz, 1H, Ar–H), 6.90 (d, J = 2.0 Hz, 1H, Ar–H), 6.88 (dd, J = 7.83 and 2.0 Hz, 1H, Ar-H), 4.66 (d, J = 9.3 Hz, 1H, OCH_AH_B), 4.20 (d, J = 9.3 Hz, 1H, OCH_AH_B), 2.83–2.49 (m, 2H, CH₂), 2.48 (q, J = 7.8 Hz, 1H, Ar-CH₂-CH₃), 2.33 (s, 6H, 2 × CH₃), 2.29 (s, 6H, 2 × CH₃), 1.45 (s, 3H, CH₃), 1.15 (t, J = 7.8 Hz, 1H, Ar-CH₂-CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ=156.9, 138.3 (d, J = 5.1 Hz), 138.2 (d, J = 5.1 Hz), 136.5, 135.6 (d, J = 11.0 Hz), 134.6 (d, J = 97.6 Hz), 134.0 (d, J = 96.8 Hz), 133.2 (d, J = 2.5 Hz), 128.0 (d, J = 9.4 Hz), 127.9 (d, J = 9.4 Hz), 127.5, 122.1, 109.4, 83.1 (d, J = 4.4 Hz), 44.8 (d, J = 4.4 Hz), 39.4 (d, J = 67.9 Hz), 28.2, 25.7 (d, J = 2.5 Hz), 21.3, 21.2, 15.9 ppm. ³¹P NMR (CDCl₃, 162 MHz): δ=28.01 ppm. HR-MS (ESI+) m/z calculated for [C₂₈H₃₇NO₂P]⁺=[M+NH₄]⁺: 450.2556; found 450.2562.

(5-tert-butyl-3-methyl-2,3-dihydro-1-benzofuran-3-yl)[bis(3,5dimethylphenyl)]phosphine oxide (3dc): GP was carried out with 4-tert-butyl-2-iodo-1-[(2-methylprop-2-enyl)oxy]benzene 1d (82 mg, 0.25 mmol), bis(3,5-dimethylphenyl)phosphine oxide 2c (64.5 mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 50:50) furnished the product 3dc (81.1 mg, 71%) as a light yellow oily compound, [TLC control (petroleum ether/ethyl acetate 80:20), Rf(1d)=0.9, Rf(3dc)=0.1, UV detection]. IR (MIR-ATR, 4000-600 cm⁻¹): vmax=3393, 2955, 1605, 1489, 1258, 1183, 976, 811, 736 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ =7.36 (d, J = 2.0 Hz, 1H, Ar–H), 7.33 (d, J = 2.0 Hz, 1H, Ar–H), 7.29 (d, J = 2.0 Hz, 1H, Ar–H), 7.26 (d, J = 2.0 Hz, 1H, Ar– H), 7.16–7.02 (m, 4H, Ar–H), 6.71 (dd, J = 8.8 and 2.0 Hz, 1H, Ar–H), 4.65 (d, J = 9.3 Hz, 1H, OC H_A H_B), 4.19 (d, J = 9.3 Hz, 1H, OCH_AH_B), 2.80–2.55 (m, 2H, CH₂), 2.33 (s, 6H, 2 × CH₃), 2.29 (s, 6H, 2 × CH₃), 1.46 (s, 3H, CH₃), 1.24 (s, 9H, 3 × CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ =156.5, 143.7, 138.3 (d, J = 6.6 Hz), 138.2 (d, J = 6.6 Hz), 135.5 (d, J = 11.7 Hz), 134.6 (d, J = 96.8

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Hz), 134.0 (d, J = 96.8 Hz), 133.2 (d, J = 2.5 Hz), 128.1 (d, J = 9.4 Hz), 127.9 (d, J = 9.4 Hz), 125.3, 119.4, 109.1, 83.1 (d, J = 3.4 Hz), 45.0 (d, J = 3.7 Hz), 39.4 (d, J = 67.9 Hz), 31.7, 25.8 (d, J = 2.5 Hz), 21.3, 21.2 ppm. HR-MS (ESI+) m/z calculated for $[C_{30}H_{38}O_2P]^+=[M+H]^+: 461.2604;$ found 461.2625.

bis(3,5-dimethylphenyl)(3-methyl-5-phenyl-2,3-dihydro-1-

benzofuran-3-yl)phosphine oxide (3ec): GP was carried out with 3-iodo-1,1'-biphenyl-4-yl 2-methylprop-2-enyl ether 1e (87.5 mg, 0.25 mmol), bis(3,5-dimethylphenyl)phosphine oxide 2c (64.5 mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 50:50) furnished the product 3ec (72 mg, 60%) as a white solid compound, mp 215-220 °C, [TLC control (petroleum ether/ethyl acetate 80:20), R_f(1e)=0.9, R_f(3ec)=0.1, UV detection]. IR (MIR-ATR, 4000-600 cm⁻¹): v_{max}=2920, 1594, 1483, 1210, 1129, 1043, 908, 838, 757, 691 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): *δ*=7.46–7.33 (m, 5H, Ar–H), 7.33–7.21 (m, 6H, Ar–H), 7.08–6.98 (m, 2H, Ar–H), 6.82 (dd, 1H, J = 8.3 and 1.0 Hz, Ar–H), 4.78 (d, J = 9.3 Hz, 1H, OCH_AH_B), 4.27 (d, J = 9.3 Hz, 1H, OCH_AH_B), 2.87–2.57 (m, 2H, CH₂), 2.27 (s, 12H, 4 × CH₃), 1.52 (s, 3H, CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ =158.6, 141.0, 138.3 (d, J = 2.9 Hz), 138.2 (d, J = 2.9 Hz), 135.9 (d, J = 10.3 Hz), 134.5 (d, J = 97.6 Hz), 133.7 (d, J = 99.0 Hz), 133.3 (t, J = 2.9 Hz), 128.6, 127.9 (d, J = 8.9 Hz), 127.8 (d, J = 8.9 Hz), 127.3, 126.7, 126.5, 121.9, 109.9, 83.6 (d, J = 4.4 Hz), 44.8 (d, J = 4.4 Hz), 39.4 (d, J = 67.9 Hz), 26.0 (d, J = 2.9 Hz), 21.2 ppm. ³¹P NMR (CDCl₃, 162 MHz): δ =27.88 ppm. HR-MS (ESI+) m/z calculated for [C₃₂H₃₃KO₂P]⁺=[M+K]⁺: 519.1850; found 519.1831.

(5-chloro-3-methyl-2,3-dihydro-1-benzofuran-3-yl)[bis(3,5-

dimethylphenyl)]phosphine oxide (3gc): GP was carried out with 4-chloro-2-iodo-1-[(2-methylprop-2-enyl)oxy]benzene 1g (77 mg, 0.25 mmol), bis(3,5-dimethylphenyl)phosphine oxide 2c (64.5 mg, 0.25 mmol Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 50:50) furnished the product 3gc (66.7 mg, 61%) as a light yellow oily compound, [TLC control (petroleum ether/ethyl acetate 80:20), R_1 (1g)=0.9, R_1 (3gc)=0.1, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): v_{max} =3404, 2922, 1597, 1466, 1178, 972, 853, 740 cm^{-1.} ¹H NMR (CDCl₃, 400 MHz): δ=7.35 (d, *J* = 1.0 Hz, 1H, Ar–H), 7.32 (d, *J* = 1.0 Hz, 1H, Ar–H), 7.25 (d, *J* = 1.0 Hz, 1H, Ar–H), 7.22 (d, *J* = 1.0 Hz, 1H, Ar–H), 7.10 (d, *J* = 1.0 Hz, 1H, Ar–H), 7.05 (d, *J* = 1.0 Hz, 1H, Ar–H), 7.00–6.94 (m, 2H, Ar–H), 6.63 (dd, *J* = 7.8 and 1.0 Hz, 1H, Ar–H), 4.79 (d, *J* = 9.3 Hz, 1H, OCH_AH_B), 4.24 (d, *J* = 9.3 Hz, 1H, OCH_AH_B), 2.76–2.53 (m, 2H, CH₂), 2.33 (s, 6H, 2 × CH₃), 2.29 (s, 6H, 2 × CH₃), 1.42 (s, 3H, CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ =157.6, 138.4 (d, *J* = 5.1 Hz), 138.3 (d, *J* = 5.1 Hz), 137.1, 134.1 (d, *J* = 98.3 Hz), 133.7 (d, *J* = 96.8 Hz), 133.4 (q, *J* = 3.0 Hz), 128.2, 127.9 (d, *J* = 8.9 Hz), 127.8 (d, *J* = 8.9 Hz), 125.1, 110.7, 83.5 (d, *J* = 3.9 Hz), 45.0 (d, *J* = 4.4 Hz), 39.3 (d, *J* = 67.9 Hz), 26.2 (d, *J* = 3.4 Hz), 21.3, 21.2 ppm. ³¹P NMR (CDCl₃, 162 MHz): δ =27.51 ppm. HR-MS (ESI+) m/z calculated for [C₂₆H₂₉ClO₂P]⁺=[M+H]⁺: 439.1588; found 439.1567.

3-[bis(4-methylphenyl)phosphoryl]-1,3-dimethyl-1,3-dihydro-

2H-indol-2-one (5ab): GP was carried out with N-(2-iodophenyl)-N,2-dimethylacrylamide 4a (75 mg, 0.25 mmol), bis(4methylphenyl)phosphine oxide 2b (57.5 mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 45:55) furnished the product 5ab (63.6 mg, 63%) as a light yellow solid compound, mp 145-150 °C, [TLC control (petroleum ether/ethyl acetate 70:30), Rf(4a)=0.6, Rf(5ab)=0.1, UV detection]. IR (MIR-ATR, 4000-600 cm⁻¹): vmax=3032, 2919, 2857, 1600, 1484, 1453, 1269, 1178, 1030, 856, 736, 695 cm $^{\text{-1}}$ ^1H NMR (CDCI₃, 400 MHz): δ =7.44 (dd, J = 8.3 and 1.0 Hz, 1H, Ar–H), 7.41 (dd, J = 8.3 and 1.0 Hz, 1H, Ar-H), 7.33 (dd, J = 8.3 and 1.0 Hz, 1H, Ar-H), 7.30 (dd, J = 8.3 and 1.0 Hz, 1H, Ar-H), 7.22-7.07 (m, 6H, Ar–H), 6.79 (ddd, J = 7.6, 7.6 and 1.5 Hz, 1H, Ar–H), 6.65 (dd, J = 7.8 and 1.5 Hz, 1H, Ar–H), 3.01 (d, J = 15.2 and 10.8 Hz, 1H, CH₂), 2.79 (d, J = 15.2 and 10.8 Hz, 1H, CH₂), 2.98 (s, 3H, CH₃), 2.33 (s, 3H, CH₃), 2.30 (s, 3H, CH₃), 1.39 (d, *J* = 2.0 Hz, 3H, CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ =179.5 (d, J = 4.4 Hz), 142.9, 141.7 (d, J = 2.9 Hz), 141.6 (d, J = 2.9 Hz), 131.6 (d, J = 2.2 Hz), 130.7 (d, J = 9.8 Hz), 130.6 (d, J = 101.2 Hz), 130.5 (d, J = 9.4 Hz), 129.8 (d, J = 101.2 Hz), 129.1 (d, J = 11.8 Hz), 128.8 (d, J = 11.8 Hz), 127.8, 124.9, 122.1, 107.7, 45.5 (d, J = 3.7 Hz), 37.5 (d, J = 71.9 Hz), 26.8 (d, J = 11.8 Hz), 26.2, 21.4 ppm. ³¹P NMR (CDCl₃, 162 MHz): δ=26.59 ppm. HR-MS (ESI+) m/z calculated for [C₂₅H₂₇NO₂P]⁺=[M+H]⁺: 404.1774; found 404.1790.

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1-benzyl-3-[bis(4-methylphenyl)phosphoryl]-3-methyl-1,3dihydro-2H-indol-2-one (5bb): GP was carried out with Nbenzyl-N-(2-iodophenyl)-2-methylacrylamide 4b (94 mg, 0.25 mmol), bis(4-methylphenyl)phosphine oxide 2b (57.5 mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 41:59) furnished the product 5bb (73.7 mg, 62%) as a light yellow oily compound, [TLC control (petroleum ether/ethyl acetate 70:30), R_f(**4b**)=0.7, R_f(**5bb**)=0.1, UV detection]. IR (MIR-ATR, 4000-600 cm⁻¹): v_{max}=3411, 3055, 2927, 1704, 1609, 1477, 1443, 1370, 1257, 1183, 736, 698 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ=7.43–7.31 (m, 4H, Ar–H), 7.22–7.05 (m, 10H, Ar–H), 6.95 (ddd, J = 7.8, 7.8 and 1.0 Hz, 1H, Ar–H), 6.66 (ddd, J = 7.8, 7.8 and 1.0 Hz, 1H, Ar–H), 6.48 (dd, J = 7.8 and 1.0 Hz, 1H, Ar–H), 4.95 (d, J = 15.2 Hz, 1H, CH₂), 4.32 (d, J = 15.2 Hz, 1H, CH₂), 2.95 (dd, J = 15.2 and 10.8 Hz, 1H, CH₂), 2.80 (d, J = 15.2 and 10.8 Hz, 1H, CH₂), 2.27 (s, 3H, CH₃), 2.26 (s, 3H, CH₃), 1.39 (d, J = 2.0 Hz, 3H, CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ =179.7 (d, J = 5.1 Hz), 141.9, 141.7 (d, J = 2.9 Hz), 141.6 (d, J = 2.9 Hz), 136.1, 131.7, 130.7 (d, J = 9.8 Hz), 130.6 (d, J = 9.8 Hz), 130.4 (d, J = 102.0 Hz), 130.2 (d, J = 102.0 Hz), 129.1 (d, J = 12.3 Hz), 128.9 (d, J = 12.3 Hz), 128.6, 127.6, 127.3, 127.1, 124.9, 122.1, 108.8, 45.6 (d, J = 3.7 Hz), 43.8, 37.3 (d, J = 71.9 Hz), 26.7 (d, J = 11.3 Hz), 21.4 ppm. ³¹P NMR (CDCl₃, 162 MHz): *δ*=26.72 ppm. HR-MS (ESI+) m/z calculated for [C₃₁H₃₄N₂O₂P]⁺=[M+NH₄]⁺: 497.2352; found 497.2381.

1-benzyl-3-[bis(3,5-dimethylphenyl)phosphoryl]-3-methyl-

1,3-dihydro-2H-indol-2-one (5bc): GP was carried out with *N*benzyl-*N*-(2-iodophenyl)-2-methylacrylamide **4b** (94 mg, 0.25 mmol), bis(3,5-dimethylphenyl)phosphine oxide **2c** (64.5 mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 45:55) furnished the product **5bc** (77 mg, 61%) as a light yellow oily compound, [TLC control (petroleum ether/ethyl acetate 70:30), *R*₁(**4b**)=0.7, *R*₁(**5bc**)=0.1, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): *v*_{max}=3038, 2922, 1604, 1451, 1178, 1105, 802, 699, 656 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): *δ*=7.30–7.12 (m, 10H, Ar–H), 7.06–7.00 (m, 3H, Ar–H), 6.71 (ddd, *J* = 7.8, 7.8 and 1.0 Hz, 1H, Ar–H), 6.55 (dd, *J* = 7.8 and 1.0 Hz, 1H, Ar–H), 5.07 (d, *J* = 15.2

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Hz, 1H, CH₂), 4.43 (d, *J* = 15.2 Hz, 1H, CH₂), 3.04 (dd, *J* = 15.2 and 10.8 Hz, 1H, CH₂), 2.87 (d, *J* = 15.2 and 10.8 Hz, 1H, CH₂), 2.27 (s, 12H, 4 × CH₃), 1.49 (d, *J* = 1.5 Hz, 3H, CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ =179.8 (d, *J* = 5.1 Hz), 142.1, 137.9 (d, *J* = 7.3 Hz), 137.8 (d, *J* = 7.3 Hz), 136.2, 133.5 (d, *J* = 98.3 Hz), 133.2 (d, *J* = 98.3 Hz), 133.1 (d, *J* = 2.9 Hz), 133.0 (d, *J* = 2.9 Hz), 131.6 (d, *J* = 2.2 Hz), 128.6, 128.2 (d, *J* = 9.3 Hz), 128.1 (d, *J* = 9.3 Hz), 127.5, 127.3, 127.1, 125.0, 121.9, 108.6, 45.6 (d, *J* = 3.7 Hz), 43.9, 37.4 (d, *J* = 71.4 Hz), 26.8 (d, *J* = 11.3 Hz), 21.2 ppm. ³¹P NMR (CDCl₃, 162 MHz): δ =26.49 ppm. HR-MS (ESI+) m/z calculated for [C₃₃H₃₅NO₂P]*=[M+H]*: 508.2400; found 508.2424.

3-[bis(4-methylphenyl)phosphoryl]-1,3,5,7-tetramethyl-1,3dihydro-2H-indol-2-one (5cb): GP was carried out with N-(2iodo-4,6-dimethylphenyl)-N,2-dimethylacrylamide 4c (82.2 mg, 0.25 mmol), bis(4-methylphenyl)phosphine oxide 2b (57.5 mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 50:50) furnished the product 5cb (76.4 mg, 71%) as a light yellow solid compound, mp 200-205 °C, [TLC control (petroleum ether/ethyl acetate 68:32), Rf(4c)=0.8, Rf(5cb)=0.1, UV detection]. IR (MIR-ATR, 4000-600 cm⁻¹): vmax=3385, 3053, 2923, 1597, 1484, 1442, 1178, 1108, 1018, 701 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ=7.39–7.31 (m, 4H, Ar–H), 7.14–7.07 (m, 4H, Ar–H), 6.65 (dd, J = 7.8 and 1.0 Hz, 1H, Ar-H), 6.61 (dd, J = 7.8 and 1.0 Hz,1H, Ar–H), 3.27 (s, 1H, CH₃), 3.05 (dd, J = 15.2 and 10.8 Hz, 1H, CH₂), 2.71 (d, J = 15.2 and 10.8 Hz, 1H, CH₂), 2.42 (s, 3H, CH₃), 2.33 (s, 3H, CH₃), 2.32 (s, 3H, CH₃), 2.01 (s, 3H, CH₃), 1.36 (d, J = 1.5 Hz, 3H, CH₃) ppm. ¹³C NMR (CDCI₃, 100 MHz): δ=180.2 (d, J = 3.7Hz), 141.5 (d, J = 2.9 Hz), 141.3 (d, J = 2.9 Hz), 138.5, 131.2, 132.0, 130.8 (d, J = 9.8 Hz), 130.5 (d, J = 9.8 Hz), 130.3 (d, J = 98.3 Hz), 129.6 (d, J = 97.6 Hz), 128.9, 128.8, 128.7, 123.3, 119.0, 44.8 (d, J = 3.7 Hz), 38.0 (d, J = 71.2 Hz), 29.7, 27.3 (d, J = 12.8 Hz), 21.4, 20.5, 18.8 ppm. ³¹P NMR (CDCI₃, 162 MHz): δ=26.42 ppm. HR-MS (ESI+) m/z calculated for [C₂₇H₃₀NNaO₂P]⁺=[M+Na]⁺: 454.1906; found 454.1918.

3-[bis(3,5-dimethylphenyl)phosphoryl]-1,3,5,7-tetramethyl-

1,3-dihydro-2H-indol-2-one (5cc): GP was carried out with *N*-(2-iodo-4,6-dimethylphenyl)-*N*,2-dimethylacrylamide **4c** (82.2 mg, 0.25 mmol), bis(3,5-dimethylphenyl)phosphine oxide **2c** (64.5 mg, 0.25 mmol), $Pd_2(dba)_3$ (11.4 mg, 5 mol%), K_2CO_3 (103.9 mg, 0.75

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mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 45:55) furnished the product 5cc (80 mg, 70%) as a light yellow oily compound, [TLC control (petroleum ether/ethyl acetate 70:30), R_f(4c)=0.7, R_f(5cc)=0.1, UV detection]. IR (MIR-ATR, 4000-600 cm⁻¹): *v_{max}*=3034, 2919, 1600, 1480, 1269, 1177, 1130, 857, 733, 696 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ =7.06 (d, J = 1.0 Hz, 1H, Ar-H), 7.03 (d, J = 1.0 Hz, 1H, Ar-H), 7.00 (d, J = 1.0 Hz, 1H, Ar-H), 6.97 (d, J = 1.0 Hz, 1H, Ar–H), 6.95 (d, J = 1.0 Hz, 1H, Ar–H), 6.92 (d, J = 1.0 Hz, 1H, Ar-H), 6.58 (d, J = 1.0 Hz, 1H, Ar-H), 6.51 (d, J = 1.0 Hz, 1H, Ar-H), 3.26 (s, 1H, CH₃), 3.00 (dd, J = 15.2 and 10.8 Hz, 1H, CH₂), 2.64 (d, J = 15.2 and 10.8 Hz, 1H, CH₂), 2.38 (s, 3H, CH₃), 2.18 (s, 12H, 4 × CH₃), 1.94 (s, 3H, CH₃), 1.32 (d, J = 1.5 Hz, 3H, CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ =180.2 (d, J = 3.0 Hz), 138.7, 137.7 (d, J = 11.0 Hz), 137.6 (d, J = 11.0 Hz), 133.5 (d, J = 98.3 Hz), 133.1 (d, J = 97.6 Hz), 133.0 (d, J = 2.5 Hz), 132.7 (d, J = 2.5 Hz), 132.0, 130.8, 128.3, 128.3 (d, J = 8.8 Hz), 128.0 (d, J = 9.4 Hz), 123.3, 118.7, 44.7 (d, J = 3.7 Hz), 38.2 (d, J = 71.4 Hz), 29.6, 27.1 (d, J = 12.3 Hz), 21.2, 21.1, 20.4, 18.7 ppm. ³¹P NMR (CDCl₃, 162 MHz): δ=26.06 ppm. HR-MS (ESI+) m/z calculated for $[C_{29}H_{35}NO_2P]^+=[M+H]^+$: 460.2400: found 460.2415.

1-benzyl-3-(diphenylphosphoryl)-3-methylindoline (5da): GP was carried out with N-benzyl-N-(2-iodophenyl)-N-(2-methylprop-2-enyl)amine 4d (90.7 mg, 0.25 mmol), diphenylphosphine oxide 2a (50.5 mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 60:40) furnished the product 5da (58.1 mg, 50%) as a light yellow oily compound, [TLC control (petroleum ether/ethyl acetate 75:25), Rf(4d)=0.9, *R*_f(**5da**)=0.1, UV detection]. IR (MIR-ATR, 4000-600 cm⁻¹): vmax=3043, 2970, 1601, 1472, 1265, 1180, 1110, 730 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ =7.76 (dd, J = 7.8 and 1.5 Hz, 1H, Ar– H), 7.73 (dd, J = 7.8 and 1.5 Hz, 1H, Ar–H), 7.67 (dd, J = 7.8 and 1.5 Hz, 1H, Ar-H), 7.64 (dd, J = 7.8 and 1.5 Hz, 1H, Ar-H), 7.53-7.39 (m, 4H, Ar-H), 7.38-7.18 (m, 4H, Ar-H), 7.05-6.92 (m, 2H, Ar–H), 6.60 (ddd, J = 7.3, 7.3 and 1.0 Hz, 1H, Ar–H), 6.42 (dd, J = 7.8 and 1.0 Hz, 1H, Ar-H), 4.17 (s, 2H CH₂), 3.54 (d, J = 9.3 Hz, 1H, NC H_AH_B), 3.08 (d, J = 9.3 Hz, 1H, NC H_AH_B), 2.75–2.65 (m, 2H, CH₂), 1.43 (s, 3H, CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz):

δ=150.6, 138.1 (d, *J* = 11.7 Hz), 134.7 (d, *J* = 97.6 Hz), 134.5 (d, *J* = 97.6 Hz), 131.4 (d, *J* = 2.9 Hz), 130.5 (d, *J* = 8.9 Hz), 130.4 (d, *J* = 8.9 Hz), 128.6, 128.5, 128.4, 128.3, 127.9, 127.8, 127.0, 122.0, 117.7, 107.4, 65.8 (d, *J* = 3.4 Hz), 52.7, 43.1 (d, *J* = 4.4 Hz), 39.0 (d, *J* = 67.9 Hz), 25.6 (d, *J* = 2.5 Hz) ppm. ³¹P NMR (CDCl₃, 162 MHz): δ=27.96 ppm. HR-MS (ESI+) m/z calculated for [C₂₉H₂₉NOP]⁺=[M+H]⁺: 438.1981; found 438.2005.

1-benzyl-3-[bis(3,5-dimethylphenyl)phosphoryl]-3-

methylindoline (5dc): GP was carried out with N-benzyl-N-(2iodophenyl)-N-(2-methylprop-2-enyl)amine 4d (90.7 mg, 0.25 mmol), bis(3,5-dimethylphenyl)phosphine oxide 2c (64.5 mg, 0.25 mmol), Pd₂(dba)₃ (11.4 mg, 5 mol%), K₂CO₃ (103.9 mg, 0.75 mmol) DMF (0.5 mL), under microwave irradiation (120 °C, 15 min, 300 W, closed vessel). Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 60:40) furnished the product 5dc (63.9 mg, 52%) as a white solid compound, mp 119-124 °C, [TLC control (petroleum ether/ethyl acetate 75:25), Rf(4d)=0.9, Rf(5dc)=0.1, UV detection]. IR (MIR-ATR, 4000-600 cm⁻¹): vmax=3052, 2967, 1600, 1485, 1441, 1266, 1181, 1108, 1021, 730, 697 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ=7.36 (dd, J = 7.8 and 1.0 Hz, 1H, Ar–H), 7.33 (dd, J = 7.8 and 1.0 Hz, 1H, Ar-H), 7.31-7.21 (m, 7H, Ar-H), 7.10-6.95 (m, 4H, Ar–H), 6.60 (ddd, J = 7.3, 7.3 and 1.0 Hz, 1H, Ar–H), 6.42 (dd, J = 7.3 and 1.5 Hz, 1H, Ar–H), 4.25 (d, J = 15.2 Hz, 1H, CH₂), 4.12 (d, J = 15.2 Hz, 1H, CH₂), 3.53 (d, J = 9.3 Hz, 1H, NCH_AH_B), 3.12 (d, J = 9.3 Hz, 1H, NCH_AH_B), 2.77–2.57 (m, 2H, CH₂), 2.32 (s, 6H, 2 × CH₃), 2.28 (s, 6H, 2 × CH₃), 1.45 (s, 3H, CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ=150.6, 138.3, 138.2, 138.1 (d, J = 2.9 Hz), 138.0 (d, J = 2.9 Hz), 134.7 (d, J = 96.8 Hz), 134.4 (d, J = 96.8 Hz), 133.0 (d, J = 2.9 Hz), 128.4, 128.0 (d, J = 9.3 Hz), 127.9 (d, J = 8.9 Hz), 127.7, 127.0, 122.1, 117.6, 107.3, 66.0 (d, J = 3.4 Hz), 52.7, 43.0 (d, J = 4.4 Hz), 39.0 (d, J = 67.9 Hz), 25.8 (d, J = 2.5 Hz), 21.3, 21.2 ppm. ³¹P NMR (CDCI₃, 162 MHz): δ=28.34 ppm. HR-MS (ESI+) m/z calculated for [C₃₃H₃₇NOP]⁺=[M+H]⁺: 494.2607; found 494.2631.

solvent DMF (5 mL). And allowed the reaction mixture to stir at room temperature for 8 to 12h. Progress of the reaction was monitored by TLC till the reaction is completed. Purification of the crude material by silica gel column chromatography (petroleum

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ether/ethyl acetate, 100:0 to 99:01) furnished the product **1c** (90%) as a white colour liquid. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} =2965, 2926, 2867, 1657, 1598, 1562, 1486, 1449, 1389, 1277, 1239, 1153, 1047, 1009, 898, 810, 759 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ=7.61 (d, 1H, *J* = 7.6 Hz, Ar–H), 7.08 (dd, 1H, *J* = 8.3 and 2.5 Hz, Ar–H), 6.70 (d, 1H, *J* = 8.3 Hz, Ar–H), 5.18 (s, 1H), 5.00 (s, 1H), 4.45 (s, 2H), 2.54 (q, *J* = 7.8 Hz, 2H, *CH*₂–CH₃), 1.86 (s, 3H, CH₃), 1.18 (t, *J* = 7.6 Hz, 3H, CH₂–CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ=155.2, 140.4, 138.7, 138.6, 128.6, 112.8, 112.1, 86.5, 72.6, 27.5, 19.5, 15.7 ppm. HR-MS (ESI+) m/z calculated for [C₁₂H₁₆IO]⁺=[M+H]⁺: 303.0240; found 303.0236.

3-iodo-1,1'-biphenyl-4-yl 2-methylprop-2-enyl ether (1e): Reaction was carried out with 3-iodo-1,1'-biphenyl-4-ol (1 equiv), 3-bromo-2-methylprop-1-ene (2 equiv), K₂CO₃ (2 equiv), and the solvent DMF (5 mL). And allowed the reaction mixture to stir at room temperature for 8 to 12h. Progress of the reaction was monitored by TLC till the reaction is completed. Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 99:01) furnished the product 1e (95%) as a white colour jelly compound. IR (MIR-ATR, 4000-600 cm⁻¹): v_{max}=3066, 3030, 2977, 2918, 1595, 1476, 1276, 1243, 1057, 1009, 901, 813, 760 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ=8.02 (d, 1H, J = 2.4 Hz, Ar–H), 7.58–7.48 (m, 3H), 7.47–7.38 (m, 2H), 7.37–7.27 (m, 1H), 6.85 (d, 1H, J = 8.3 Hz, Ar–H), 5.22 (s, 1H), 5.04 (s, 1H), 4.52 (s, 2H), 1.89 (s, 3H, CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ=156.6, 140.1, 139.3, 137.0, 135.8, 128.8 (2C), 127.9, 127.1, 126.7 (2C), 113.0, 112.2, 86.9, 72.6, 19.5 ppm. HR-MS (ESI+) m/z calculated for [C₁₆H₁₆IO]⁺=[M+H]⁺: 351.0240; found 351.0251.

4-bromo-2-iodo-1-[(2-methylprop-2-enyl)oxy]benzene (1h): Reaction was carried out with 4-bromo-2-iodophenol (1 equiv), 3bromo-2-methylprop-1-ene (2 equiv), K₂CO₃ (2 equiv), and the solvent DMF (5 mL). And allowed the reaction mixture to stir at room temperature for 8 to 12h. Progress of the reaction was monitored by TLC till the reaction is completed. Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 99:01) furnished the product **1h** (92%) as a white colour liquid. IR (MIR-ATR, 4000–600 cm⁻¹): *v*_{max}=3077, 2922, 1657, 1566, 1457, 1374, 1275, 1233, 1035, 903, 800, 678, 628 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ=7.87 (d, 1H, *J* = 2.4 Hz, Ar–H), 7.36 (dd, 1H, *J* = 8.8 and 2.5 Hz, Ar–H), 6.64 (d, 1H, *J* = 8.8 Hz, Ar–H), 5.16 (s, 1H), 5.02 (s, 1H), 4.44 (s, 2H), 1.84 (s, 3H, CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): *δ*=156.5, 141.2, 139.8, 132.0, 113.5, 113.2, 113.1, 87.2, 72.8, 19.4 ppm.

2-methylprop-2-enyl 2-iodobenzoate (1i): Reaction was carried out with 2-iodobenzoic acid (1 equiv), 3-bromo-2-methylprop-1ene (2 equiv), TBAI (1 equiv), K₂CO₃ (2 equiv), and the solvent DMF (5 mL). And allowed the reaction mixture to stir at room temperature for 8 to 12h. Progress of the reaction was monitored by TLC till the reaction is completed. Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 99:01) furnished the product 1i (85%) as a white colour liquid. IR (MIR-ATR, 4000-600 cm⁻¹): vmax=3078, 2937, 1724, 1582, 1433, 1370, 1285, 1242, 1127, 1101, 1041, 903, 739 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ =8.00 (dd, 1H, J = 7.8 and 1.0 Hz, Ar-H), 7.83 (dd, 1H, J = 7.8 and 1.5 Hz, Ar-H), 7.40 (ddd, 1H, J = 7.3, 7.3 and 1.0 Hz, Ar–H), 7.15 (ddd, 1H, J = 7.8, 7.8 and 1.5 Hz, Ar-H), 5.10 (s, 1H), 5.00 (s, 1H), 4.76 (s, 2H), 1.86 (s, 3H, CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ=166.1, 141.3, 139.5, 135.0, 132.6, 130.9, 127.9, 113.7, 94.1, 68.9, 19.7 ppm. HR-MS (ESI+) m/z calculated for [C₁₁H₁₂IO₂]⁺=[M+H]⁺: 302.9876; found 302.9880.

N-benzyl-N-(2-iodophenyl)-N-(2-methylprop-2-enyl)amine

(4d): Reaction was carried out with 2-iodoaniline (1 equiv), 3bromo-2-methylprop-1-ene (2 equiv), K2CO3 (2 equiv), and the solvent DMF (5 mL). And allowed the reaction mixture to stir at room temperature to 70 °C for 12h. Progress of the reaction was monitored by TLC till the reaction is completed. Then in the next step protection of free NH was carried out with (bromomethyl)benzene (1.2 equiv), NaH (2 equiv) and the solvent THF at 0 °C under inert atmosphere, progress of the reaction was monitored by TLC till the reaction is completed. Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 99:01) furnished the product 4d (60%) as a light yellow colour liquid. IR (MIR-ATR, 4000-600 cm⁻ ¹): *v*_{max}=3065, 2918, 2829, 1584, 1501, 1460, 1367, 1268, 1203, 1013, 938, 751 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): *δ*=7.85 (dd, 1H, J = 7.8 and 1.0 Hz, Ar–H), 7.33–7.24 (m, 3H), 7.23–7.16 (m, 3H), 6.91 (dd, 1H, J = 8.3 and 1.5 Hz, Ar–H), 6.75 (ddd, 1H, J = 7.3, 7.3 and 1.5 Hz, Ar-H), 4.93 (s, 1H), 4.85 (s, 1H), 4.12 (s, 2H, CH₂), 3.48 (s, 2H, CH₂), 1.76 (s, 3H, CH₃) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ=151.7, 142.4, 140.1, 137.6, 129.0 (2C), 128.4, 128.0 (2C), 127.0, 125.6, 124.2, 113.7, 99.4, 58.7, 57.9, 21.1 ppm. HR-MS (ESI+) m/z calculated for $[C_{17}H_{19}IN]^+=[M+H]^+$: 364.0557; found 364.0553.

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Acknowledgments

We are grateful to the Department of Science and Technology-Science and Engineering Research Board (DST-SERB) [NO.:EMR/2017/005312], New Delhi, for financial support. K.R. thanks MHRD, New Delhi, for the award of a research fellowship.

Keywords: Domino • Microwaves • short reaction times • benzofurans • oxindoles.

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Layout 2:

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* This process is accelerated by microwave-assisting conditions and enabled the construction of C-C and C-P bonds in short reaction times for the synthesis of dihydrobenzofurans, oxindoles and indolines.