Supporting Information

Metal-free synthesis of carboxamides via Lossen rearrangement

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1. General Information

¹H and ¹³C NMR spectra were recorded on a Bruker AVANCE NEO 400 spectrometer (400 MHz for ¹H, 101 MHz for ¹³C, and 162 MHz for ³¹P) in CDCl₃ or DMSO-*d*. Chemical shifts (δ) were measured in ppm relative to TMS $\delta = 0$ for ¹H, or to chloroform $\delta = 77.0$ for ¹³C as internal standard. ³¹P NMR spectra were recorded on the same instrument. Data are reported as follows: Chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), and Coupling constants, *J*, are reported in hertz. Mass data were measured with Waters Xevo TQ-XS mass spectrometer. The starting materials were purchased from Aldrich, Acros Organics, J&K Chemicals, or TCI and used without further purification. Solvents were dried and purified according to the "Purification of Laboratory Chemicals book" procedure. Column chromatography was performed on silica gel (particle size 200-400 mesh ASTM). The hydroxylamine derivatives were prepared according to the literature ^[1]. **2. General Procedure**

$$Ar^{1} H_{H}^{O} OBz + H_{H}^{O} H_{Ar^{2}}^{O} \frac{Na_{2}CO_{3} (50 \text{ mol}\%)}{CH_{3}CN, \text{ Air, } 60 \degree C} Ar^{1} H_{Ar^{2}}^{H} Ar^{2}$$

Conditions: In a 20 ml Schlenk tube containing a magnetic stir bar, 1 (0.2 mmol), 2 (0.4 mmol), Na₂CO₃ (50 mol %) in 4 mL CH₃CN under Air at 60 °C for 10 h. After completion, the reaction was quenched by adding a saturated aqueous solution of NaHCO₃ (2 mL), concentrated under reduced pressure. The residue was diluted with EtOAc (20 mL), washed sequentially with saturated aqueous solution of NaHCO₃ (10 mL) and brine (10 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under vacuum. The residue was purified by flash chromatography on silica gel to afford the desired compound 3.

$$Ar \overset{O}{\underset{H}{\overset{}}} OBz + Ph_{3}P \overset{O}{\overset{}} COR \overset{K_{2}CO_{3} (50 \text{ mol}\%)}{CH_{3}CN, \text{ Air, 90 °C}} Ar \overset{O}{\underset{H}{\overset{}}} \overset{O}{\underset{PPh_{3}}{\overset{}}} COR$$

Conditions: In a 20 ml Schlenk tube containing a magnetic stir bar, 1 (0.2 mmol), 4 (0.24 mmol), K_2CO_3 (50 mol %) in 4 mL CH₃CN under Air at 90 °C for 10 h. After completion, the reaction was quenched by adding a saturated aqueous solution of NaHCO₃ (2 mL), concentrated under reduced pressure. The residue was diluted with EtOAc (20 mL), washed sequentially with saturated aqueous solution of NaHCO₃ (10 mL) and brine (10 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under vacuum. The residue was purified by flash chromatography on silica gel to afford the desired compound 5.



Conditions: In a 20 ml Schlenk tube containing a magnetic stir bar, 1 (0.2 mmol), 6 (0.24 mmol), K_2CO_3 (50 mol %) in 4 mL CH₃CN under Air at 90 °C for 10 h. After

completion, the reaction was quenched by adding a saturated aqueous solution of NaHCO₃ (2 mL), concentrated under reduced pressure. The residue was diluted with EtOAc (20 mL), washed sequentially with saturated aqueous solution of NaHCO₃ (10 mL) and brine (10 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under vacuum. The residue was purified by flash chromatography on silica gel to afford the desired compound 7.

	N ^{-OBz} H Ph ₃ P	√ ^{OEt}	base (50 mol%) solvent, Air,T °C	
entry	base	т	solvent	yield(%) ^b
1	Na ₂ CO ₃	90	CH ₃ CN	76
2	K ₂ CO ₃	90	CH ₃ CN	89
3	KHCO ₃	90	CH ₃ CN	N.D.
4	NaHCO ₃	90	CH ₃ CN	N.D.
5	KOAc	90	CH ₃ CN	N.D.
6	NaHCO ₃	90	CH ₃ CN	N.D.
7 ^c	K ₂ CO ₃	90	CH ₃ CN	89
8 ^c	K ₂ CO ₃	70	CH ₃ CN	70
9 ^c	K ₂ CO ₃	90	DCE	44
10 ^c	K ₂ CO ₃	90	THF	68

3. Optimization Studies of β-Ketophosphorus Ylides^a

^{*a*} The reaction was performed with 0.2 mmol of *N*-(benzoyloxy)-4-methylbenzamide, P-ylide (2.0 eq.), and K₂CO₃ (50 mol%) in 4 mL CH₃CN under Air at 90 °C. ^{*b*} Isolated yield. ^{*c*} P-ylide (1.2 eq.)

4. Optimization Studies of Imidazolidindiones ^a

	N ^{-OBz} H BnHN	CO ₂ Et sc	ase (50 mol%) Ivent, Air,T °C	
entry	base	т	solvent	yield(%) ^b
1	Na ₂ CO ₃	90	CH ₃ CN	79
2	K ₂ CO ₃	90	CH ₃ CN	82
3	KHCO₃	90	CH₃CN	N.D.
4	Ft ₂ N	90	CH₃CN	N.D.
5	DBU	90	CH ₃ CN	N.D.
6	K ₂ CO ₃	70	CH ₃ CN	74
7	K ₂ CO ₃	90	Toluene	21
8	K ₂ CO ₃	90	DCE	51
9	K ₂ CO ₃	90	THF	81

^{*a*} The reaction was performed with 0.2 mmol of **1**, **6** (1.2 eq.), and K_2CO_3 (50 mol%) in 4 mL CH₃CN under Air at 90 °C ^{*b*} Isolated yield.

5. Gram-Scale Reactions



Conditions: In a 200 ml round-bottom flask containing a magnetic stir bar, *N*-(benzoyloxy)benzamide (20 mmol), diphenylphosphine oxide (2.0 eq.), Na_2CO_3 (50 mol %) in 100 mL CH₃CN under Air at 60 °C for 10 h. After completion, the reaction

was quenched by adding a saturated aqueous solution of NaHCO₃ (200 mL), concentrated under reduced pressure. The residue was diluted with EtOAc (200 mL), washed sequentially with saturated aqueous solution of NaHCO₃ (100 mL) and brine (100 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under vacuum. The residue was purified by flash chromatography on silica gel to afford the desired compound 3a.



Conditions: In a 200 ml round-bottom flask containing a magnetic stir bar, *N*-(benzoyloxy)-4-methylbenzamide (20 mmol), P-ylide (1.2 eq.), K_2CO_3 (50 mol %) in 100 mL CH₃CN under Air at 90 °C for 10 h. After completion, the reaction was quenched by adding a saturated aqueous solution of NaHCO₃ (200 mL), concentrated under reduced pressure. The residue was diluted with EtOAc (200 mL), washed sequentially with saturated aqueous solution of NaHCO₃ (100 mL) and brine (100 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under vacuum. The residue was purified by flash chromatography on silica gel to afford the desired compound 5a.



Conditions: In a 200 ml Schlenk tube containing a magnetic stir bar, *N*-(benzoyloxy)-4-methylbenzamide (20 mmol), ethyl benzyl-D-valinate (1.2 eq.), K_2CO_3 (50 mol %) in 120 mL CH₃CN under Air at 90 °C for 10 h. After completion, the reaction was quenched by adding a saturated aqueous solution of NaHCO₃ (200 mL), concentrated under reduced pressure. The residue was diluted with EtOAc (200 mL), washed sequentially with saturated aqueous solution of NaHCO₃ (100 mL) and brine (100 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under vacuum. The residue was purified by flash chromatography on silica gel to afford the desired compound 7b. **6. Investigation of the Reaction Mechanism**



Condition a: In a 20 ml round-bottom flask containing a magnetic stir bar, *N*-(benzoyloxy)benzamide (0.2 mmol), diphenylphosphine oxide (2.0 eq.), Na₂CO₃ (50 mol %), PPh₃ (2.0 eq.) in 4 mL CH₃CN under Air at 60 °C for 10 h. After completion, the reaction was quenched by adding a saturated aqueous solution of NaHCO₃ (2 mL), concentrated under reduced pressure. The residue was diluted with EtOAc (20 mL), washed sequentially with saturated aqueous solution of NaHCO₃ (10 mL) and brine (10 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under vacuum. The residue was purified by flash chromatography on silica gel to afford the desired compound 3a, TLC detected no product **8**.

Condition b: In a 20 ml round-bottom flask containing a magnetic stir bar, **9** (0.2 mmol), diphenylphosphine oxide (2.0 eq.), Na₂CO₃ (50 mol %) in 4 mL CH₃CN under Air at 60 °C for 10 h. There was no rection was detected.

Condition c: In a 20 ml round-bottom flask containing a magnetic stir bar, *p*-Tolyl isocyanate (0.2 mmol), diphenylphosphine oxide (2.0 eq.), Na₂CO₃ (50 mol %) in 4 mL CH₃CN under Air at 60 °C for 10 h. After completion, the reaction was quenched by adding a saturated aqueous solution of NaHCO₃ (2 mL), concentrated under reduced pressure. The residue was diluted with EtOAc (20 mL), washed sequentially with saturated aqueous solution of NaHCO₃ (10 mL) and brine (10 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under vacuum. The residue was purified by flash chromatography on silica gel to afford the desired compound 3a.

7. Characterization Data of New Product



1-(diphenylphosphoryl)-N-phenylformamide (3a)

¹H NMR (400 MHz, CDCl₃) δ 10.01 (s, 1H), 8.15 – 7.85 (m, 4H), 7.75 (d, J = 8.0 Hz, 2H), 7.56 (td, J = 7.3, 1.4 Hz, 2H), 7.53 – 7.42 (m, 4H), 7.31 (t, J = 8.0 Hz, 2H), 7.15 (d, J = 7.4 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 167.9 (d, J = 123.3 Hz), 136.9 (d, J = 9.5 Hz), 132.7 (d, J = 2.8 Hz), 131.8, 131.7, 129.3, 128.9, 128.7, 128.6, 128.3, 125.4, 120.0. ³¹P NMR (162 MHz, CDCl₃) δ 15.6. HRMS (ESI-TOF) *m/z*: [M + H]⁺ Calcd for C₁₉H₁₇NO₂P⁺: 322.0991, found 322.0991.



1-(diphenylphosphoryl)-N-(p-tolyl)formamide (3b)

¹H NMR (400 MHz, CDCl₃) δ 9.57 (s, 1H), 8.13 – 7.93 (m, 3H), 7.58 (dd, *J* = 11.6, 4.8 Hz, 4H), 7.49 (td, *J* = 7.5, 3.2 Hz, 4H), 7.14 (d, *J* = 8.3 Hz, 2H), 2.31 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 167.6 (d, *J* = 123.0 Hz), 135.3, 134.2 (d, *J* = 9.6 Hz), 132.7, 132.7, 131.8, 131.7, 129.5, 129.3, 128.8, 128.6, 128.3, 119.8, 20.9. ³¹P NMR (162 MHz, CDCl₃) δ 15.6. HRMS (ESI-TOF) *m/z*: [M + H]⁺ Calcd for C₂₀H₁₉NO₂P⁺: 336.1148, found 336.1154.



1-(diphenylphosphoryl)-N-(m-tolyl)formamide (3c)

¹H NMR (400 MHz, CDCl₃) δ 9.54 (s, 1H), 8.06 – 7.78 (m, 4H), 7.64 – 7.46 (m, 3H), 7.43-7.38 (m, 5H), 7.15 (dd, *J* = 16.0, 8.2 Hz, 1H), 6.90 (d, *J* = 7.6 Hz, 1H), 2.24 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 166.8 (d, *J* = 123.0 Hz), 138.1, 135.7 (d, *J* = 9.5 Hz), 131.8, 131.8, 130.8, 130.7, 128.2, 127.9, 127.8, 127.7, 127.2, 125.4, 119.5, 116.0, 20.4. ³¹P NMR (162 MHz, CDCl₃) δ 15.7. HRMS (ESI-TOF) *m/z*: [M + H]⁺ Calcd for C₂₀H₁₉NO₂P⁺: 336.1148, found 336.1154.



1-(diphenylphosphoryl)-N-(o-tolyl)formamide (3d)

¹H NMR (400 MHz, CDCl₃) δ 9.52 (s, 1H), 7.88-7.84 (m, 5H), 7.54 – 7.31 (m, 6H), 7.19 – 7.02 (m, 2H), 6.97 (t, *J* = 7.3 Hz, 1H), 2.22 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 167.9 (d, *J* = 122.5 Hz), 134.6 (d, *J* = 9.0 Hz), 132.9 (d, *J* = 2.8 Hz), 131.8, 131.7, 130.8, 130.0, 129.4, 129.1, 128.9, 128.8, 128.4, 128.3, 126.8, 126.1, 122.2, 17.8. ³¹P NMR (162 MHz, CDCl₃) δ 15.7. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₂₀H₁₉NO₂P⁺: 336.1148, found 336.1154.



1-(diphenylphosphoryl)-N-(3-methoxyphenyl)formamide (3e)

¹H NMR (400 MHz, CDCl₃) δ 9.60 (s, 1H), 8.11 – 7.84 (m, 4H), 7.69 – 7.41 (m, 8H), 7.27 – 7.17 (m, 2H), 6.78 – 6.66 (m, 1H), 3.77 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 168.0 (d, J = 122.9 Hz), 160.1, 137.9, 137.8, 132.8, 131.8, 131.7, 129.8, 129.1, 128.8, 128.7, 128.1, 112.0, 111.9, 105.2, 55.3. ³¹P NMR (162 MHz, CDCl₃) δ 15.8. HRMS (ESI-TOF) *m/z*: [M+Na]⁺ Calcd for C₂₀H₁₈NO₃PNa⁺: 374.0917, found 374.0925

1-(diphenylphosphoryl)-N-(2-methoxyphenyl)formamide (3f)

¹H NMR (400 MHz, DMSO) δ 9.99 (s, 1H), 8.11 (d, *J* = 7.9 Hz, 1H), 7.93 – 7.84 (m, 4H), 7.77 – 7.68 (m, 2H), 7.66 – 7.60 (m, 4H)., 7.19 (dd, *J* = 7.3, 1.5 Hz, 1H), 7.14 (dd, *J* = 8.3, 1.3 Hz, 1H), 7.03 – 6.88 (m, 1H), 3.91 (s, 3H). ¹³C NMR (101 MHz, DMSO) δ 168.1 (d, *J* = 122.2 Hz), 149.5, 133.5, 133.5, 131.9, 131.8, 129.9, 129.6, 129.5, 128.9, 126.7, 126.0, 125.9, 121.0, 120.9, 111.9, 56.5. ³¹P NMR (162 MHz, DMSO) δ 14.8.

HRMS (ESI-TOF) m/z: $[M+Na]^+$ Calcd for $C_{20}H_{18}NO_3PNa^+$: 374.0917, found 374.0925

1-(diphenylphosphoryl)-N-mesitylformamide (3g)

¹H NMR (400 MHz, CDCl₃) δ 8.90 (s, 1H), 7.97 (ddd, J = 12.1, 8.3, 1.3 Hz, 4H), 7.59 (dd, J = 7.5, 1.6 Hz, 2H), 7.55 – 7.47 (m, 4H), 6.88 (s, 2H), 2.26 (s, 3H), 2.13 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 168.3 (d, J = 132.8 Hz), 134.7, 132.7, 132.7, 131.7, 131.6, 129.6, 129.0, 128.8, 128.7, 128.6, 20.9, 18.3. ³¹P NMR (162 MHz, CDCl₃) δ 16.2. HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for C₂₂H₂₃NO₂P⁺: 364.1461, found 364.1464.



N-(benzo[*d*][1,3]dioxol-5-yl)-1-(diphenylphosphoryl)formamide (3h)

¹H NMR (400 MHz, CDCl₃) δ 9.47 (s, 1H), 7.87 (dd, J = 12.0, 7.4 Hz, 4H), 7.52 (t, J = 7.0 Hz, 2H), 7.43 (td, J = 7.4, 3.0 Hz, 4H), 7.33 (d, J = 1.3 Hz, 1H), 7.19 (s, 1H), 6.99 (dd, J = 8.3, 1.5 Hz, 1H), 6.68 (d, J = 8.4 Hz, 1H), 5.87 (s, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 166.4 (d, J = 123.3 Hz), 146.8, 144.1, 131.8, 131.8, 130.8, 130.7, 130.2, 130.1, 128.2, 127.8, 127.7, 127.2, 112.2, 107.1, 101.2, 100.4. ³¹P NMR (162 MHz, CDCl₃) δ 15.7. HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for C₂₀H₁₇NO₄P⁺: 366.0890, found 366.0896.



1-(diphenylphosphoryl)-N-(2-fluoro-5-methylphenyl)formamide (3i)

¹H NMR (400 MHz, CDCl₃) δ 9.47 (s, 1H), 8.08 (d, J = 7.3 Hz, 1H), 7.99 – 7.72 (m, 4H), 7.57 – 7.47 (m, 2H), 7.47 – 7.33 (m, 4H), 6.91 (dd, J = 10.4, 8.5 Hz, 1H), 6.83 – 6.68 (m, 1H), 2.20 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 168.5 (d, J = 122.5 Hz), 150.8 (d, J = 244.1 Hz), 134.3, 134.3, 133.0, 132.9, 131.9, 131.8, 129.1, 128.9, 128.8, 128.1, 126.5, 126.4, 124.7, 124.6, 124.6, 122.1, 115.0, 114.8, 21.0. ³¹P NMR (162 MHz, CDCl₃) δ 15.7. HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for C₂₀H₁₈FNO₂P⁺: 354.1054, found 354.1053.



N-(4-bromophenyl)-1-(diphenylphosphoryl)formamide (3j)

¹H NMR (400 MHz, CDCl₃) δ 9.84 (s, 1H), 7.93 (ddd, J = 12.1, 8.3, 1.3 Hz, 4H), 7.70 - 7.55 (m, 4H), 7.55 - 7.47 (m, 4H), 7.47 - 7.39 (m, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 168.2 (d, J = 122.6 Hz), 135.9, 135.8, 132.9, 132.9, 132.1, 131.8, 131.7, 129.1, 128.9, 128.8, 128.0, 121.6, 118.3. ³¹P NMR (162 MHz, CDCl₃) δ 15.9. HRMS (ESI-TOF) *m/z*: [M+Na]⁺ Calcd for C₁₉H₁₅BrNO₂PNa⁺: 421.9916, found 421.9924.



1-(di-*p*-tolylphosphoryl)-*N*-phenylformamide (31)

¹H NMR (400 MHz, CDCl₃) δ 9.58 (s, 1H), 7.95 – 7.76 (m, 4H), 7.69 (d, *J* = 7.9 Hz, 2H), 7.43 – 7.23 (m, 6H), 7.16 (d, *J* = 7.4 Hz, 1H), 2.40 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 168.4 (d, *J* = 122.7 Hz), 143.4, 136.9, 136.8, 131.8, 131.7, 129.5, 129.4, 129.0, 126.0, 125.4, 125.0, 119.8, 21.6. ³¹P NMR (162 MHz, CDCl₃) δ 16.8. HRMS (ESI-TOF) *m/z*: [M+Na]⁺ Calcd for C₂₁H₂₀NO₂PNa⁺: 372.1124, found 372.1133.



1-(bis(4-methoxyphenyl)phosphoryl)-N-phenylformamide (3m)

¹H NMR (400 MHz, CDCl₃) δ 9.65 (s, 1H), 7.93 – 7.78 (m, 4H), 7.71 (d, *J* = 7.9 Hz, 2H), 7.33 (t, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 7.4 Hz, 1H), 6.99 (dd, *J* = 8.9, 2.4 Hz, 4H), 3.83 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 168.8 (d, *J* = 124.1 Hz), 163.1, 136.9, 136.8, 133.8, 133.6, 129.0, 125.3, 120.4, 119.8, 119.4, 114.4, 114.2, 55.3. ³¹P NMR (162 MHz, CDCl₃) δ 16.8. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₂₁H₂₁NO₄P⁺: 404.1022, found 404.1032.



1-(bis(3,5-dimethylphenyl)phosphoryl)-*N*-phenylformamide (3n)

¹H NMR (400 MHz, DMSO) δ 10.89 (s, 1H), 7.80 (d, J = 7.9 Hz, 2H), 7.44 (d, J = 12.1 Hz, 4H), 7.34 (t, J = 7.9 Hz, 2H), 7.29 (s, 2H), 7.15 (s, 1H), 2.33 (s, 12H). ¹³C NMR (101 MHz, DMSO) δ 169.3 (d, J = 122.1 Hz), 169.3 (d, J = 122.1 Hz), 138.6 (d, J = 12.5 Hz), 137.8 (d, J = 9.5 Hz), 134.6, 130.7, 129.7, 129.3, 129.2, 129.2, 125.6, 121.3, 21.3. ³¹P NMR (162 MHz, DMSO) δ 14.9. HRMS (ESI-TOF) m/z: [M+Na]⁺ Calcd for C₂₃H₂₄NO₂PNa⁺: 400.1437, found 400.1436.



1-(bis(4-fluorophenyl)phosphoryl)-*N*-phenylformamide (30)

¹H NMR (400 MHz, CDCl₃) δ 9.56 (s, 1H), 8.05 – 7.87 (m, 4H), 7.69 (d, *J* = 7.9 Hz, 2H), 7.35 (t, *J* = 8.0 Hz, 2H), 7.25 – 7.11 (m, 5H). ¹³C NMR (101 MHz, CDCl₃) δ 167.6 (d, *J* = 125.3 Hz), 164.5, 164.4, 136.6, 136.5, 134.5, 134.4, 134.3, 129.2, 125.8, 125.1, 125.0, 124.0, 119.9, 116.5 (d, *J* = 13.6 Hz), 116.3 (d, *J* = 13.7 Hz).³¹P NMR (162 MHz, CDCl₃) δ 14.0. HRMS (ESI-TOF) *m/z*: [M+Na]⁺ Calcd for C₁₉H₁₄F₂NO₂PNa⁺: 380.0622, found 380.0630.



1-(bis(4-chlorophenyl)phosphoryl)-N-phenylformamide (3p)

¹H NMR (400 MHz, CDCl₃) δ 9.54 (s, 1H), 7.87 (dd, *J* = 11.6, 8.5 Hz, 4H), 7.68 (d, *J* = 8.0 Hz, 2H), 7.49 (dd, *J* = 8.4, 2.3 Hz, 4H), 7.35 (t, *J* = 7.9 Hz, 2H), 7.19 (s, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 167.2 (d, J = 125.2 Hz), 139.9, 136.5, 136.4, 133.1, 133.0, 129.3, 129.2, 127.4, 126.4, 125.9, 120.0. ³¹P NMR (162 MHz, CDCl₃) δ 13.7. HRMS (ESI-TOF) *m/z*: [M+Na]⁺ Calcd for C₁₉H₁₄Cl₂NO₂PNa⁺: 412.0031, found 412.0039.



ethyl 3-oxo-3-(phenylamino)-2-(triphenyl- λ^5 -phosphaneylidene)propanoate (5a) ¹H NMR (400 MHz, CDCl₃) δ 10.91 (s, 1H), 7.80 – 7.54 (m, 6H), 7.54 – 7.29 (m, 11H), 7.16 – 7.01 (m, 2H), 6.85 – 6.70 (m, 1H), 3.62 (q, *J* = 7.1 Hz, 2H), 0.44 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.1, 170.0, 168.1, 168.0, 140.0, 139.9, 133.2, 133.1, 131.5, 131.5, 128.4, 128.3, 127.8, 126.9, 121.6, 119.3, 58.2, 56.7 (d, *J* = 127.8 Hz), 13.51. ³¹P NMR (162 MHz, CDCl₃) δ 18.5. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₂₉H₂₇NO₃P⁺: 468.1723, found 468.1731.



ethyl 3-oxo-3-(p-tolylamino)-2-(triphenyl- λ^5 -phosphaneylidene)propanoate (5b) ¹H NMR (400 MHz, CDCl₃) δ 10.91 (s, 1H), 7.83 – 7.62 (m, 6H), 7.55 – 7.45 (m, 3H), 7.45 – 7.36 (m, 8H), 6.97 (d, *J* = 8.3 Hz, 2H), 3.69 (q, *J* = 7.1 Hz, 2H), 2.21 (s, 3H), 0.52 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.1, 169.9, 168.0, 167.9, 137.4, 137.4, 133.2, 133.1, 131.4, 131.4, 130.8, 128.9, 128.36, 128.2, 127.8, 126.9, 119.2, 58.1, 56.5 (d, J = 127.9 Hz), 20.6, 13.5. ³¹P NMR (162 MHz, CDCl₃) δ 18.4. HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for C₃₀H₂₉NO₃P⁺: 482.1880, found 482.1888.



ethyl 3-((4-(chloromethyl)phenyl)amino)-3-oxo-2-(triphenyl-λ⁵-phosphaneylidene) -propanoate (5c)

¹H NMR (400 MHz, CDCl₃) δ 11.07 (s, 1H), 8.07 – 7.96 (m, 2H), 7.77 – 7.65 (m, 6H), 7.52 (ddd, J = 5.2, 3.1, 1.6 Hz, 5H), 7.44 – 7.36 (m, 4H), 7.30 – 7.23 (m, 2H), 5.24 (s, 2H), 3.70 (q, J = 7.1 Hz, 2H), 0.53 (t, J = 7.1 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.2, 170.1, 168.2, 168.1, 166.5, 140.2, 140.2, 133.3, 133.2, 132.8, 131.6, 131.6, 130.3, 129.6, 129.1, 128.9, 128.5, 128.4, 128.2, 127.7, 126.8, 119.3, 66.8, 58.3, 56.9 (d, J = 127.7 Hz), 13.5. ³¹P NMR (162 MHz, CDCl₃) δ 18.5. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₃₀H₂₈ClNO₃P⁺: 516.1490, found 516.1500.



Ethyl 3-((4-chlorophenyl)amino)-3-oxo-2-(triphenyl- λ^5 -phosphaneylidene) - propanoate (5d)

¹H NMR (400 MHz, CDCl₃) δ 11.07 (s, 1H), 7.70 (ddd, J = 12.7, 5.2, 3.3 Hz, 6H), 7.52 (ddt, J = 8.6, 3.1, 1.6 Hz, 3H), 7.48 – 7.38 (m, 8H), 7.13 – 7.06 (m, 2H), 3.70 (q, J = 7.1 Hz, 2H), 0.53 (t, J = 7.1 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.2, 170.1, 168.2, 168.1, 138.6, 138.5, 133.2, 133.1, 131.7, 131.6, 128.5, 128.4, 128.3, 127.5, 126.6, 126.3, 120.5, 58.3, 56.8 (d, J = 127.2 Hz), 13.5. ³¹P NMR (162 MHz, CDCl₃) δ 18.6. HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for C₂₉H₂₆ClNO₃P⁺: 502.1333, found 502.1338.



ethyl 3-((4-bromophenyl)amino)-3-oxo-2-(triphenyl-λ⁵-phosphaneylidene) -propanoate (5e)

¹H NMR (400 MHz, CDCl₃) δ 11.04 (s, 1H), 7.71 (dd, J = 12.7, 8.0 Hz, 6H), 7.64 – 7.35 (m, 11H), 7.35 – 7.09 (m, 2H), 3.69 (q, J = 7.1 Hz, 2H), 0.53 (t, J = 7.1 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.1, 168.1, 139.2, 133.3, 133.2, 131.7, 131.7, 131.4, 128.6, 128.5, 127.6, 126.7, 120.9, 113.9, 58.4, 56.9 (d, J = 127.2 Hz), 13.6. ³¹P NMR (162 MHz, CDCl₃) δ 18.5. HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for C₂₉H₂₆ClNO₃P⁺: 546.0828, found 546.0834.



ethyl 3-oxo-3-((4-(trifluoromethoxy)phenyl)amino)-2-(triphenyl-λ⁵-phosphane -ylidene)propanoate (5f)

¹H NMR (400 MHz, CDCl₃) δ 11.08 (s, 1H), 7.71 (ddd, J = 12.7, 5.2, 3.3 Hz, 6H), 7.62 – 7.39 (m, 11H), 7.02 (d, J = 8.4 Hz, 2H), 3.70 (q, J = 7.1 Hz, 2H), 0.53 (t, J = 7.1 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.3, 170.2, 168.3, 168.2, 143.5, 138.9, 133.4, 133.3, 131.8, 131.7, 128.6, 128.5, 127.7, 126.7, 121.5, 120.2, 77.4, 58.4, 56.9 (d, J = 127.3 Hz), 13.6. ³¹P NMR (162 MHz, CDCl₃) δ 18.5. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₃₀H₂₆F₃NO₄P⁺: 552.1546, found 552.1556.



ethyl 3-oxo-3-((4-(trifluoromethyl)phenyl)amino)-2-(triphenyl-λ⁵-phosphane -ylidene)propanoate (5g)

¹H NMR (400 MHz, CDCl₃) δ 11.20 (s, 1H), 7.70 – 7.57 (m, 6H), 7.52 (d, J = 8.5 Hz, 2H), 7.49 – 7.43 (m, 3H), 7.42 – 7.35 (m, 6H), 7.33 (d, J = 8.6 Hz, 2H), 3.63 (q, J = 7.1 Hz, 2H), 0.45 (t, J = 7.1 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.3, 170.1, 168.3, 168.2, 143.2, 133.3, 133.2, 131.8, 131.7, 128.6, 128.5, 127.4, 126.5, 125.8, 125.7, 123.3, 123.2, 123.0, 118.7, 114.1, 58.6, 57.2 (d, J = 127.0 Hz), 13.5. ³¹P NMR (162 MHz, CDCl₃) δ 18.6. HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for C₃₀H₂₆F₃NO₄P⁺: 536.1597, found 536.1606.



methyl 4-(3-ethoxy-3-oxo-2-(triphenyl- λ^5 -phosphaneylidene)propanamido) -benzoate (5h)

¹H NMR (400 MHz, CDCl₃) δ 11.27 (s, 1H), 7.79 (d, J = 8.8 Hz, 2H), 7.69 – 7.59 (m, 6H), 7.54 – 7.42 (m, 5H), 7.41 – 7.33 (m, 6H)), 3.74 (s, 3H), 3.63 (q, J = 7.1 Hz, 2H), 0.46 (t, J = 7.1 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.1, 170.0, 168.2, 168.1, 166.9, 144.4, 133.2, 133.1, 131.7, 131.6, 130.4, 128.5, 128.4, 127.2, 126.3, 122.6, 118.1, 58.4, 57.3 (d, J = 126.5 Hz), 51.5, 13.4. ³¹P NMR (162 MHz, CDCl₃) δ 18.7. HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for C₃₁H₂₉NO₅P⁺: 526.1778, found 526.1785.

ethyl 3-((3-bromophenyl)amino)-3-oxo-2-(triphenyl-λ⁵-phosphaneylidene) -propanoate (5i)

¹H NMR (400 MHz, CDCl₃) δ 11.03 (s, 1H), 7.71 (d, J = 1.5 Hz, 1H), 7.68 – 7.56 (m, 6H), 7.50 – 7.31 (m, 9H), 7.31 – 7.21 (m, 1H), 6.99 – 6.88 (m, 2H), 3.61 (q, J = 7.1 Hz, 2H), 0.44 (t, J = 7.1 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.2, 170.1, 168.1, 168.0, 141.4, 133.3, 133.2, 131.70, 131.7, 129.7, 128.6, 128.4, 127.5, 126.5, 124.5, 122.3, 121.9, 117.7, 58.4, 56.9 (d, J = 127.1 Hz), 13.5. ³¹P NMR (162 MHz, CDCl₃) δ 18.7. HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for C₂₉H₂₆BrNO₃P ⁺: 546.0828, found 546.0834.



ethyl 3-oxo-3-(o-tolylamino)-2-(triphenyl-λ⁵-phosphaneylidene)propanoate (5j)

¹H NMR (400 MHz, CDCl₃) δ 10.86 (s, 1H), 8.05 (d, J = 8.1 Hz, 1H), 7.79 – 7.63 (m, 6H), 7.56 – 7.36 (m, 9H), 7.10 (d, J = 7.3 Hz, 1H), 7.01 (t, J = 7.7 Hz, 1H), 6.83 (td, J = 7.4, 0.9 Hz, 1H), 3.72 (q, J = 7.1 Hz, 2H), 2.40 (s, 3H), 0.51 (t, J = 7.1 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.3, 170.1, 168.1, 168.0, 138.5, 138.4, 133.2, 133.1, 131.5, 129.8, 128.4, 128.3, 127.9, 127.0, 126.4, 126.1, 121.7, 120.4, 58.2, 57.0 (d, J = 127.8 Hz), 18.5, 13.5. ³¹P NMR (162 MHz, CDCl₃) δ 18.5. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₃₀H₂₉NO₃P⁺: 482.1880, found 482.1888.



ethyl 3-((3,5-dichlorophenyl)amino)-3-oxo-2-(triphenyl- λ^5 -phosphaneylidene) -propanoate (5k)

¹H NMR (400 MHz, CDCl₃) δ 11.25 (s, 1H), 7.77 – 7.64 (m, 6H), 7.59 – 7.50 (m, 3H), 7.50 – 7.41 (m, 8H), 6.85 (t, *J* = 1.8 Hz, 1H), 3.69 (q, *J* = 7.1 Hz, 2H), 0.52 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.3, 170.2, 168.1, 168.0, 141.9, 134.5, 133.3, 133.2, 131.8, 128.6, 128.5, 127.2, 126.2, 121.3, 117.2, 58.5, 57.0 (d, *J* = 126.6 Hz), 13.5. ³¹P NMR (162 MHz, CDCl₃) δ 18.7. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₃₀H₂₉NO₃P⁺: 536.0944, found 536.0950.



ethyl 3-((2-fluoro-5-methylphenyl)amino)-3-oxo-2-(triphenyl- λ^5 -phosphane -ylidene) propanoate (5l)

¹H NMR (400 MHz, CDCl₃) δ 11.22 (s, 1H), 8.03 (dd, J = 7.7, 1.9 Hz, 1H), 7.85 – 7.64 (m, 6H), 7.60 – 7.38 (m, 9H), 6.89 (dd, J = 11.0, 8.3 Hz, 1H), 6.69 – 6.47 (m, 1H), 3.73

(q, J = 7.1 Hz, 2H), 2.15 (s, 3H), 0.53 (t, J = 7.1 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.0, 169.9, 168.0, 167.9, 151.7, 149.4, 133.3, 133.3, 133.2, 131.6, 131.6, 128.5, 128.4, 128.4, 128.1, 128.0, 127.6, 126.6, 121.7, 121.6, 121.3, 113.9, 113.7, 58.3, 56.9 (d, J = 127.4 Hz), 20.9, 13.5. ³¹P NMR (162 MHz, CDCl₃) δ 18.7. HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for C₃₀H₂₈FNO₃P⁺: 500.1785, found 500.1793.



ethyl 3-((3,5-dimethoxyphenyl)amino)-3-oxo-2-(triphenyl-λ⁵-phosphaneylidene) -propanoate (5m)

¹H NMR (400 MHz, CDCl₃) δ 10.95 (s, 1H), 7.73 – 7.53 (m, 6H), 7.51 – 7.39 (m, 3H), 7.39 – 7.29 (m, 6H), 6.67 (d, *J* = 2.3 Hz, 2H), 5.98 (t, *J* = 2.3 Hz, 1H), 3.77 – 3.42 (m, 8H), 0.43 (t, *J* = 7.1 Hz, 3H).³¹P NMR (162 MHz, CDCl₃) δ 18.62. ¹³C NMR (101 MHz, CDCl₃) δ 170.2, 170.0, 168.0, 167.9, 160.7, 141.7, 141.7, 133.3, 133.2, 131.6, 131.5, 128.4, 128.3, 127.6, 126.7, 97.4, 94.7, 58.2, 56.7 (d, *J* = 127.3 Hz), 55.2, 13.5. ³¹P NMR (162 MHz, CDCl₃) δ 18.6. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₃₁H₃₁NO₅P⁺: 528.1934, found 528.1940.



benzyl 3-oxo-3-(*p*-tolylamino)-2-(triphenyl-λ⁵-phosphaneylidene)propanoate (5n) ¹H NMR (400 MHz, CDCl₃) δ 10.73 (s, 1H), 7.63 – 7.50 (m, 6H), 7.42 – 7.21 (m, 11H), 7.12 – 7.01 (m, 3H), 6.89 (d, J = 8.3 Hz, 2H), 6.73 (dd, J = 7.5, 1.5 Hz, 2H), 4.59 (s, 2H), 2.13 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 169.6 (d, J = 12.6 Hz), 167.8 (d, J =9.5 Hz), 137.4, 137.3, 136.5, 133.1, 133.0, 131.5, 131.4, 131.0, 128.9, 128.4, 128.2, 128.0, 127.6, 127.4, 126. 7, 119.4, 64.4, 56.7 (d, J = 127.8 Hz), 20.7. ³¹P NMR (162 MHz, CDCl₃) δ 18.5. HRMS (ESI-TOF) *m*/*z*: [M+H]⁺ Calcd for C₃₅H₃₁NO₃P⁺: 544.2036, found 544.2047.



3-oxo-*N*-(*p*-tolyl)-2-(triphenyl-λ⁵-phosphaneylidene)propenamide (50)

¹H NMR (400 MHz, CDCl₃) δ 11.72 (s, 1H), 8.18 (d, *J* = 3.7 Hz, 1H), 7.74 – 7.63 (m, 6H), 7.63 – 7.56 (m, 3H), 7.56 – 7.46 (m, 8H), 7.03 (d, *J* = 8.3 Hz, 2H), 2.25 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 183.2, 182.9, 166.5, 166.4, 136.9, 133.8, 133.7 132.7, 132.6, 131.7, 129.1, 129.0, 124.5, 123.6, 119.8, 78.5, 20.7.³¹P NMR (162 MHz, CDCl₃) δ 22.1. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₂₈H₂₅NO₂P⁺: 438.1617, found 438.1625.



3-oxo-*N*-(*p*-tolyl)-2-(triphenyl- λ^5 -phosphaneylidene)butanamide (5p)

¹H NMR (400 MHz, CDCl₃) δ 12.22 (s, 1H), 7.84 – 7.56 (m, 6H), 7.56 – 7.34 (m, 9H), 7.29 (d, J = 8.4 Hz, 2H), 6.89 (d, J = 8.3 Hz, 2H), 2.13 (s, 3H), 1.30 (s, 4H). ¹³C NMR (101 MHz, CDCl₃) δ 191.4, 191.2, 168.1, 168.0, 137.1, 137.1, 133.3, 133.2, 132.0, 131.9, 131.3, 128.9, 128.8, 127.9, 126.9, 119.2, 75.5, 29.4, 29.4, 20.7. ³¹P NMR (162 MHz, CDCl₃) δ 17.1. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₂₉H₂₇NO₂P⁺: 452.1774, found 452.1780.



3-cyclopropyl-3-oxo-N-(p-tolyl)-2-(triphenyl- λ^5 -phosphaneylidene)propenamide (5q)

¹H NMR (400 MHz, CDCl₃) δ 11.94 (s, 1H), 7.86 – 7.53 (m, 6H), 7.51 – 7.33 (m, 9H), 7.28 (d, J = 8.4 Hz, 2H), 6.87 (d, J = 8.2 Hz, 2H), 2.12 (s, 3H), 0.77 (qd, J = 7.7, 3.8 Hz, 3H), -0.02 (dt, J = 32.0, 14.0 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 193.3 (d, J = 18.2 Hz), 167.5 (d, J = 9.6 Hz), 137.3, 133.3, 133.2, 131.8, 131.1, 128.9, 128.8, 128.7, 127.8, 126.8, 119.6, 75.2 (d, J = 120.2 Hz), 20.7, 20.7, 9.6. ³¹P NMR (162 MHz, CDCl₃) δ 17.5. HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for C₃₁H₂₉NO₂P⁺: 478.1930, found 478.1934.



methyl 3-((tert-butyldimethylsilyl)oxy)-5,7-dioxo-7-(p-tolylamino)-6-(triphenyl -λ⁵-phosphaneylidene)heptanoate (5r)

¹H NMR (400 MHz, CDCl₃) δ 12.15 (s, 1H), 7.82 – 7.70 (m, 6H), 7.55 (dd, J = 7.4, 1.4 Hz, 3H), 7.51 – 7.46 (m, 6H), 7.38 (d, J = 8.3 Hz, 2H), 6.99 (d, J = 8.3 Hz, 2H), 4.62 (dd, J = 6.8, 5.0 Hz, 1H), 3.59 (s, 3H), 2.37 (dd, J = 14.2, 4.7 Hz, 1H), 2.26 (d, J = 10.0 Hz, 3H), 2.22 – 2.11 (m, 1H), 1.74 (d, J = 55.8 Hz, 2H), 0.75 (s, 9H), -0.00 (s, 3H), -0.06 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 171.6, 137.2, 133.5, 133.4, 132.1, 131.6, 129.1, 129.0, 128.9, 128.6, 128.5, 127.9, 127.0, 119.9, 67.2, 51.4, 48.2, 42.8, 25.8, 20. 8, 17.9, 0.5 (d, J = 102.8 Hz), -4.9 (d, J = 53.0 Hz). ³¹P NMR (162 MHz, CDCl₃) δ 16.5. HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for C₃₉H₄₇NO₅PSi⁺: 668.2956, found 668.2965.



1-benzyl-5-isopropyl-3-phenylimidazolidine-2,4-dione (7a)

¹H NMR (400 MHz, CDCl₃) δ 7.45 – 7.20 (m, 10H), 5.07 (d, *J* = 15.2 Hz, 1H), 4.08 (d, *J* = 15.2 Hz, 1H), 3.72 (d, *J* = 3.1 Hz, 1H), 2.22 (ddd, *J* = 10.8, 7.0, 3.5 Hz, 1H), 1.08 (d, *J* = 7.1 Hz, 3H), 0.89 (d, *J* = 6.9 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.9, 155.9, 135.5, 131.7, 128.9, 128.2, 128.1, 128.0, 126.0, 62.9, 44.9, 28.6, 17.5, 16.0. HRMS (ESI-TOF) *m*/*z*: [M+H]⁺ Calcd for C₁₉H₂₁N₂O₂⁺: 309.1598, found 309.1608.



1-benzyl-5-isopropyl-3-(p-tolyl)imidazolidine-2,4-dione (7b)

¹H NMR (400 MHz, CDCl₃) δ 7.35 – 7.11 (m, 9H), 5.14 (d, *J* = 15.2 Hz, 1H), 4.15 (d, *J* = 15.3 Hz, 1H), 3.78 (d, *J* = 3.1 Hz, 1H), 2.36 (s, 3H), 2.29 (dtd, *J* = 14.0, 7.0, 3.2 Hz, 1H), 1.15 (d, *J* = 7.1 Hz, 3H), 0.96 (d, *J* = 6.9 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.9, 156.0, 138.0, 135.5, 129.5, 129.0, 128.9, 128.1, 128.0, 125.9, 62.9, 44.8, 28.5, 21.1, 17.4, 16.0. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₂₀H₂₃N₂O₂⁺: 323.1754, found 323. 1760.



1-benzyl-5-isopropyl-3-(4-methoxyphenyl)imidazolidine-2,4-dione (7c)

¹H NMR (400 MHz, CDCl₃) δ 7.50 – 7.21 (m, 7H), 7.10 – 6.72 (m, 2H), 5.14 (d, *J* = 15.2 Hz, 1H), 4.16 (d, *J* = 15.2 Hz, 1H), 3.90 – 3.67 (m, 4H), 2.30 (dtd, *J* = 14.0, 7.0, 3.1 Hz, 1H), 1.15 (d, *J* = 7.0 Hz, 3H), 0.97 (d, *J* = 6.9 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 171.12, 159.08, 156.18, 135.54, 128.89, 128.14, 128.03, 127.45, 124.33, 114.25, 62.90, 55.39, 44.85, 28.55, 17.46, 16.03. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₂₀H₂₃N₂O₃⁺: 339.1703, found 339.1709.



1-benzyl-5-isopropyl-3-(3-methoxyphenyl)imidazolidine-2,4-dione (7d)

¹H NMR (400 MHz, CDCl₃) δ 7.46 – 7.26 (m, 6H), 6.95 (dddd, J = 24.0, 8.3, 2.1, 0.8 Hz, 3H), 5.15 (d, J = 15.2 Hz, 1H), 4.16 (d, J = 15.2 Hz, 1H), 3.81 (s, 3H), 3.79 (d, J = 3.1 Hz, 1H), 2.30 (dtd, J = 14.0, 7.0, 3.1 Hz, 1H), 1.16 (d, J = 7.1 Hz, 3H), 0.97 (d, J = 6.9 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.8, 159.9, 155.8, 135.5, 132.7, 129.6, 128.9, 128.2, 128.1, 118.3, 114.0, 111.8, 62.9, 55.3, 44.9, 28.6, 17.5, 16.0. HRMS (ESITOF) m/z: [M+H]⁺ Calcd for C₂₀H₂₃N₂O₃⁺: 339.1703, found 339.1709.



1-benzyl-3-(4-chlorophenyl)-5-isopropylimidazolidine-2,4-dione (7e)

¹H NMR (400 MHz, CDCl₃) δ 7.47 – 7.27 (m, 9H), 5.15 (d, *J* = 15.2 Hz, 1H), 4.16 (d, *J* = 15.2 Hz, 1H), 3.80 (d, *J* = 3.1 Hz, 1H), 2.31 (dtd, *J* = 14.0, 7.0, 3.1 Hz, 1H), 1.16 (d, *J* = 7.1 Hz, 3H), 0.96 (d, *J* = 6.9 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.6, 155.5, 135.3, 133.6, 130.2, 129.1, 129.0, 128.2, 127.2, 62.9, 44.9, 28.6, 17.5, 16.0. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₁₉H₂₀ClN₂O₂⁺: 343.1208, found 343.1213.



1-benzyl-3-(4-bromophenyl)-5-isopropylimidazolidine-2,4-dione (7f)

¹H NMR (400 MHz, CDCl₃) δ 7.66 – 7.51 (m, 1H), 7.51 – 7.26 (m, 8H), 5.16 (dd, J = 15.2, 6.6 Hz, 1H), 4.16 (dd, J = 15.2, 4.3 Hz, 1H), 3.80 (t, J = 3.5 Hz, 1H), 2.31 (dtd, J = 10.9, 7.0, 4.2 Hz, 1H), 1.16 (d, J = 7.0 Hz, 3H), 0.96 (d, J = 7.0 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.6, 155.5, 135.3, 132.1, 130.8, 129.0, 129.0, 128.2, 128.1, 128.1, 127.4, 126.1, 121.7, 63.0, 44.9, 28.7, 28.6, 17.5, 16.1, 16.0. HRMS (ESI-TOF) m/z: [M+Na]⁺ Calcd for C₁₉H₁₉BrN₂NaO₂⁺: 409.0522, found 409.0528.



1-benzyl-3-(3-chlorophenyl)-5-isopropylimidazolidine-2,4-dione (7g)

¹H NMR (400 MHz, CDCl₃) δ 7.44 – 7.36 (m, 1H), 7.35 – 7.18 (m, 8H), 5.06 (d, *J* = 15.2 Hz, 1H), 4.07 (d, *J* = 15.2 Hz, 1H), 3.72 (d, *J* = 3.1 Hz, 1H), 2.22 (dtd, *J* = 14.0, 7.0, 3.2 Hz, 1H), 1.07 (d, *J* = 7.1 Hz, 3H), 0.88 (d, *J* = 6.9 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.5, 155.3, 135.3, 134.4, 132.8, 129.8, 129.0, 128.2, 128.1, 126.1, 124.0, 62.9, 44.8, 28.5, 17.5, 16.0. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₁₉H₂₀ClN₂O₂⁺: 343.1208, found 343.1213.



1-benzyl-3-(3-bromophenyl)-5-isopropylimidazolidine-2,4-dione (7h)

¹H NMR (400 MHz, CDCl₃) δ 7.63 (t, *J* = 1.9 Hz, 1H), 7.50 (ddd, *J* = 7.9, 1.8, 1.2 Hz, 1H), 7.44 – 7.38 (m, 2H), 7.38 – 7.32 (m, 3H), 7.32 – 7.28 (m, 2H), 5.16 (d, *J* = 15.2 Hz, 1H), 4.16 (d, *J* = 15.2 Hz, 1H), 3.80 (d, *J* = 3.1 Hz, 1H), 2.32 (dtd, *J* = 14.0, 7.0, 3.1 Hz, 1H), 1.16 (d, *J* = 7.1 Hz, 3H), 0.97 (d, *J* = 6.9 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.6, 155.3, 135.3, 132.9, 131.1, 130.2, 129.0, 128.9, 128.2, 124.6, 122.3, 62.9, 44.9, 28.6, 17.6, 16.1. HRMS (ESI-TOF) *m/z*: [M+Na]⁺ Calcd for C₁₉H₁₉BrN₂NaO₂⁺: 409.0522, found 409.0528.



1-benzyl-3-(3,5-dichlorophenyl)-5-isopropylimidazolidine-2,4-dione (7i)

¹H NMR (400 MHz, CDCl₃) δ 7.44 (d, *J* = 1.9 Hz, 2H), 7.37 (dddd, *J* = 6.7, 5.7, 3.8, 2.0 Hz, 4H), 7.28 (dd, *J* = 11.8, 5.4 Hz, 2H), 5.15 (d, *J* = 15.2 Hz, 1H), 4.15 (d, *J* = 15.2 Hz, 1H), 3.80 (d, *J* = 3.2 Hz, 1H), 2.31 (dtd, *J* = 14.0, 7.0, 3.2 Hz, 1H), 1.16 (d, *J* = 7.1 Hz, 3H), 0.96 (d, *J* = 6.9 Hz, 3H).¹³C NMR (101 MHz, CDCl₃) δ 170.2, 154.8, 135.1, 135.0, 133.5, 129.1, 128.3, 128.2, 128.0, 124.2, 62.9, 44.9, 28.6, 17.5, 16.0. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₁₉H₁₉Cl₂N₂O₂₃⁺: 377.0818, found 377.0821.



methyl-4-(3-benzyl-4-isopropyl-2,5-dioxoimidazolidin-1-yl)benzoate (7j)

¹H NMR (400 MHz, CDCl₃) δ 8.20 – 8.06 (m, 2H), 7.66 – 7.51 (m, 2H), 7.44 – 7.28 (m, 5H), 5.18 (d, *J* = 15.2 Hz, 1H), 4.17 (d, *J* = 15.2 Hz, 1H), 3.93 (s, 3H), 3.82 (d, *J* = 3.1 Hz, 1H), 2.33 (dtd, *J* = 14.0, 7.0, 3.2 Hz, 1H), 1.17 (d, *J* = 7.0 Hz, 3H), 0.98 (d, *J* = 6.9 Hz, 3H).¹³C NMR (101 MHz, CDCl₃) δ 170.5, 166.3, 155.3, 135.9, 135.3, 130.3, 129.2, 129.0, 128.3, 128.2, 125.4, 62.9, 52.3, 44.9, 28.6, 17.6, 16.1. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₂₁H₂₃N₂O₄⁺: 367.1652, found 367.1656.



4-(3-benzyl-4-isopropyl-2,5-dioxoimidazolidin-1-yl)benzonitrile (7k)

¹H NMR (400 MHz, CDCl₃) δ 7.72 – 7.56 (m, 4H), 7.35 – 7.20 (m, 5H), 5.08 (d, *J* = 15.2 Hz, 1H), 4.09 (d, *J* = 15.2 Hz, 1H), 3.75 (d, *J* = 3.1 Hz, 1H), 2.34 – 2.16 (m, 1H), 1.09 (d, *J* = 7.1 Hz, 3H), 0.88 (d, *J* = 6.9 Hz, 3H).¹³C NMR (101 MHz, CDCl₃) δ 170.2, 154.8, 135.8, 135.0, 133.2, 132.7, 129.1, 128.3, 128.2, 125.9, 118.2, 111.1, 62.9, 44.9, 28.6, 17.5, 15.9. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₂₀H₂₀N₃O₂⁺: 334.1550, found 334.1553.



1-benzyl-5-isopropyl-3-(naphthalen-2-yl)imidazolidine-2,4-dione (7l)

¹H NMR (400 MHz, CDCl₃) δ 7.87 – 7.80 (m, 2H), 7.76 (dd, *J* = 9.1, 5.0 Hz, 2H), 7.46 – 7.37 (m, 3H), 7.33 – 7.18 (m, 5H), 5.09 (d, *J* = 15.2 Hz, 1H), 4.09 (d, *J* = 15.2 Hz, 1H), 3.75 (d, *J* = 3.1 Hz, 1H), 2.24 (dtd, *J* = 14.0, 7.0, 3.1 Hz, 1H), 1.09 (d, *J* = 7.1 Hz, 3H), 0.92 (d, *J* = 6.9 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 171.0, 156.0, 135.5, 133.1, 132.5, 129.2, 128.9, 128.8, 128.2, 128.1, 128.1, 127.6, 126.5, 126.4, 124.9, 123.7, 63.0, 44.9 28.6, 17.5, 16.1. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₂₃H₂₃N₂O₂⁺: 359.1754, found 359.1766.



1-benzyl-3-(furan-3-yl)-5-isopropylimidazolidine-2,4-dione (7m)

¹H NMR (400 MHz, CDCl₃) δ 7.45 – 7.27 (m, 6H), 6.49 (dd, *J* = 3.4, 2.1 Hz, 1H), 6.40 (dd, *J* = 3.4, 0.9 Hz, 1H), 5.13 (d, *J* = 15.2 Hz, 1H), 4.14 (d, *J* = 15.2 Hz, 1H), 3.80 (d, *J* = 3.2 Hz, 1H), 2.29 (dtd, *J* = 14.0, 7.0, 3.2 Hz, 1H), 1.15 (d, *J* = 7.1 Hz, 3H), 0.97 (d, *J* = 6.9 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.1, 154.4, 141.5, 137.6, 135.1, 129.0, 128.2, 111.3, 106.5, 63.2, 44.9, 28.5, 17.5, 15.9. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₁₇H₁₉N₂O₃⁺: 299.1390, found 299.1397.



1-benzyl-5-methyl-3-(p-tolyl)imidazolidine-2,4-dione (7n)

¹H NMR (400 MHz, CDCl₃) δ 7.44 – 7.20 (m, 9H), 5.05 (d, *J* = 15.2 Hz, 1H), 4.22 (d, *J* = 15.2 Hz, 1H), 3.92 (q, *J* = 6.9 Hz, 1H), 2.37 (s, 3H), 1.45 (d, *J* = 7.0 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 172.5, 155.2, 138.0, 135.6, 129.6, 129.0, 128.9, 128.1, 128.1, 125.8, 54.5, 44.7, 21.1, 15.3. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₁₈H₁₉N₂O₂⁺: 295.1441, found 295.1453.



1-benzyl-5-phenyl-3-(*p*-tolyl)imidazolidine-2,4-dione (70)

¹H NMR (400 MHz, CDCl₃) δ 7.47 – 7.37 (m, 3H), 7.36 – 7.28 (m, 5H), 7.26 (dd, J = 11.5, 5.2 Hz, 4H), 7.21 – 7.13 (m, 2H), 5.21 (d, J = 14.9 Hz, 1H), 4.79 (s, 1H), 3.79 (d, J = 14.9 Hz, 1H), 2.35 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.1, 155.5, 138.0, 135.2, 132.5, 129.6, 129.3, 129.0, 128.9, 128.5, 128.1, 127.6, 125.8, 62.7, 44.6, 21.1. HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for C₂₃H₂₁N₂O₂⁺: 357.1598, found 357.1600.



1,5-dibenzyl-3-(p-tolyl)imidazolidine-2,4-dione (7p)

¹H NMR (400 MHz, CDCl₃) δ 7.41 – 7.24 (m, 6H), 7.24 – 7.08 (m, 6H), 6.94 (d, J = 8.0 Hz, 2H), 5.21 (d, J = 15.1 Hz, 1H), 4.15 (t, J = 4.4 Hz, 1H), 4.08 (d, J = 15.1 Hz, 1H), 3.29 – 3.12 (m, 2H), 2.33 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 171.2, 155.6, 138.2, 135.3, 134.2, 129.6, 129.0, 128.8, 128.6, 128.3, 128.2, 127.4, 126.0, 59.1, 45.0, 35.0, 21.1. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₂₄H₂₃N₂O₂⁺: 371.1754, found 371.1759.



1-benzyl-5-(4-(tert-butoxy)benzyl)-3-(p-tolyl)imidazolidine-2,4-dione (7q)

¹H NMR (400 MHz, CDCl₃) δ 7.34 (s, 3H), 7.29 – 7.12 (m, 4H), 7.12 – 7.00 (m, 2H), 7.00 – 6.71 (m, 4H), 5.23 (d, *J* = 15.0 Hz, 1H), 4.11 (dd, *J* = 14.0, 9.7 Hz, 2H), 3.31 – 2.91 (m, 2H), 2.33 (s, 3H), 1.34 (d, *J* = 3.2 Hz, 9H). ¹³C NMR (101 MHz, CDCl₃) δ 171.3, 155.6, 154.7, 138.2, 135.4, 130.1, 129.6, 129.0, 128.9, 128.8, 128.4, 128.2, 126.0, 124.2, 78.5, 59.2, 45.1, 34.3, 28.8, 21.1. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₂₈H₃₁N₂O₃⁺: 443.2329, found 443.2336.



1-benzyl-5-(sec-butyl)-3-(p-tolyl)imidazolidine-2,4-dione (7r)

¹H NMR (400 MHz, CDCl₃) δ 7.68 – 7.00 (m, 9H), 5.16 (d, *J* = 15.2 Hz, 1H), 4.10 (d, *J* = 15.2 Hz, 1H), 3.86 (d, *J* = 3.0 Hz, 1H), 2.36 (s, 3H), 2.12 – 1.92 (m, 1H), 1.79 – 1.45 (m, 2H), 0.91 (dd, *J* = 15.8, 7.2 Hz, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 170.9, 156.0, 138.0, 135.5, 129.5, 129.0, 128.9, 128.2, 128.1, 125.9, 61.4, 44.5, 35.1, 24.8, 21.1, 13.3, 12.0. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₂₁H₂₅N₂O₂⁺: 337.1911, found 337.1918.



ethyl-3-(3-benzyl-2,5-dioxo-1-(p-tolyl)imidazolidin-4-yl)propanoate (7s)

¹H NMR (400 MHz, CDCl₃) δ 7.45 – 7.17 (m, 9H), 5.07 (d, *J* = 15.2 Hz, 1H), 4.14 (dt, *J* = 14.3, 10.0 Hz, 3H), 3.99 (dd, *J* = 6.1, 3.0 Hz, 1H), 2.50 – 2.25 (m, 6H), 2.25 – 2.05 (m, 1H), 1.24 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 172.2, 171.3, 155.6, 138.2, 135.4, 129.6, 129.0, 128.9, 128.4, 128.2, 125.9, 60.8, 57.3, 44.7, 28.0, 23.6, 21.1, 14.1. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₂₂H₂₅N₂O₄⁺: 381.1809, found 381.1821.



1-benzyl-5-(2-(methylthio)ethyl)-3-(p-tolyl)imidazolidine-2,4-dione (7t)

¹H NMR (400 MHz, CDCl₃) δ 7.53 – 6.95 (m, 9H), 5.03 (d, *J* = 15.2 Hz, 1H), 4.22 (d, *J* = 15.2 Hz, 1H), 4.03 (dd, *J* = 6.2, 3.1 Hz, 1H), 2.59 – 2.42 (m, 2H), 2.37 (s, 3H), 2.34 – 2.17 (m, 1H), 2.08 (ddd, *J* = 14.5, 7.3, 4.6 Hz, 1H), 2.02 (s, 3H).¹³C NMR (101 MHz, CDCl₃) δ 171.4, 155.8, 138.1, 135.4, 129.6, 129.0, 128.3, 128.2, 125.8, 57.5, 45.2, 28.0, 27.7, 21.1, 15.2. HRMS (ESI-TOF) *m/z*: [M+H]⁺ Calcd for C₂₀H₂₃N₂O₂S ⁺: 355.1475, found 355.1483.

8. References

[1] (a) H. Wang, H. Jung, F. Song, S. Zhu, Z. Bai, D. Chen, G. He, S. Chang, G. Chen, Nat. Chem., 2021, 13, 378-385; (b) S. Lin, B. Lin, Z. Zhang, J. Chen, Y. Luo, Y. Xia, Org. Lett., 2022, 24, 3302-3306.

9. NMR Spectra of Products

¹H NMR (400 MHz, CDCl₃) of compound **3a**



¹³C NMR (101 MHz, CDCl₃) of compound **3a**



210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 f1 (ppm)

³¹P NMR (162 MHz, CDCl₃) of compound **3a**



 ^1H NMR (400 MHz, CDCl₃) of compound 3b







 ^{31}P NMR (162 MHz, CDCl₃) of compound 3b



-40 -60 f1 (ppm)





 ^{13}C NMR (101 MHz, CDCl₃) of compound 3c



³¹P NMR (162 MHz, CDCl₃) of compound **3c**



 ^1H NMR (400 MHz, CDCl₃) of compound 3d







 ^{31}P NMR (162 MHz, CDCl₃) of compound 3d



¹H NMR (400 MHz, CDCl₃) of compound 3e



 $^{13}\mathrm{C}$ NMR (101 MHz, CDCl₃) of compound 3e



³¹P NMR (162 MHz, CDCl₃) of compound **3**e



¹H NMR (400 MHz, DMSO) of compound $\mathbf{3f}$



 $^{13}\mathrm{C}$ NMR (101 MHz, DMSO) of compound 3f



 ^{31}P NMR (162 MHz, DMSO) of compound 3f



140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 $_{f1\,(ppm)}$

¹H NMR (400 MHz, CDCl₃) of compound 3g



 ^{13}C NMR (101 MHz, CDCl₃) of compound 3g



 ^{31}P NMR (162 MHz, CDCl₃) of compound 3g



 ^1H NMR (400 MHz, CDCl₃) of compound $\boldsymbol{3h}$



 $^{13}\mathrm{C}$ NMR (101 MHz, CDCl₃) of compound **3h**



 ^{31}P NMR (162 MHz, CDCl₃) of compound $\boldsymbol{3h}$



S32





 ^{13}C NMR (101 MHz, CDCl₃) of compound 3i







¹H NMR (400 MHz, CDCl₃) of compound **3**j



 $^{13}\mathrm{C}$ NMR (101 MHz, CDCl₃) of compound 3j



³¹P NMR (162 MHz, CDCl₃) of compound **3**j







 ^{13}C NMR (101 MHz, CDCl₃) of compound **3**l


³¹P NMR (162 MHz, CDCl₃) of compound **31**



 ^1H NMR (400 MHz, CDCl₃) of compound 3m



S37

 ^{13}C NMR (101 MHz, CDCl₃) of compound 3m



 ^{31}P NMR (162 MHz, CDCl₃) of compound 3m













¹H NMR (400 MHz, CDCl₃) of compound 30



 ^{13}C NMR (101 MHz, CDCl₃) of compound 30



 ^{31}P NMR (162 MHz, CDCl₃) of compound 30







 ^{13}C NMR (101 MHz, CDCl₃) of compound $\boldsymbol{3p}$



³¹P NMR (162 MHz, CDCl₃) of compound **3**p



 1 H NMR (400 MHz, CDCl₃) of compound **5a**



¹³C NMR (101 MHz, CDCl₃) of compound **5a**



 ^{31}P NMR (162 MHz, CDCl₃) of compound 5a







 ^{13}C NMR (101 MHz, CDCl₃) of compound 5b



^{210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10} f1(ppm)





 ^1H NMR (400 MHz, CDCl₃) of compound 5c



S46









S48





¹H NMR (400 MHz, CDCl₃) of compound **5**e



¹³C NMR (101 MHz, CDCl₃) of compound **5**e



 ^{31}P NMR (162 MHz, CDCl₃) of compound 5e







 ^{13}C NMR (101 MHz, CDCl₃) of compound $\mathbf{5f}$







 ^1H NMR (400 MHz, CDCl_3) of compound $\mathbf{5g}$



 ^{13}C NMR (101 MHz, CDCl₃) of compound 5g



 ^{31}P NMR (162 MHz, CDCl_3) of compound 5g



140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 f1 (ppm)

 ^1H NMR (400 MHz, CDCl₃) of compound 5h



 $^{13}\mathrm{C}$ NMR (101 MHz, CDCl_3) of compound $\mathbf{5h}$





¹H NMR (400 MHz, CDCl₃) of compound **5**i



¹³C NMR (101 MHz, CDCl₃) of compound **5**i



 ^{31}P NMR (162 MHz, CDCl_3) of compound 5i



¹H NMR (400 MHz, CDCl₃) of compound **5**j



 ^{13}C NMR (101 MHz, CDCl_3) of compound 5j



210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 f1(ppm)





 ^1H NMR (400 MHz, CDCl₃) of compound 5k



S58

 $^{13}\mathrm{C}$ NMR (101 MHz, CDCl₃) of compound 5k



 ^{31}P NMR (162 MHz, CDCl_3) of compound 5k



140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 $_{f1(ppm)}^{\rm res}$

 ^1H NMR (400 MHz, CDCl_3) of compound 5l



 ^{13}C NMR (101 MHz, CDCl₃) of compound **5**l



³¹P NMR (162 MHz, CDCl₃) of compound **5**l



 ^1H NMR (400 MHz, CDCl₃) of compound 5m







 ^{31}P NMR (162 MHz, CDCl₃) of compound 5m



140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 $f1\,(\text{ppm})$

 ^1H NMR (400 MHz, CDCl₃) of compound 5n



 ^{13}C NMR (101 MHz, CDCl₃) of compound 5n



³¹P NMR (162 MHz, CDCl₃) of compound **5n**



¹H NMR (400 MHz, CDCl₃) of compound **50**



S64

¹³C NMR (101 MHz, CDCl₃) of compound **50**



³¹P NMR (162 MHz, CDCl₃) of compound **50**



140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 $_{f1(ppm)}^{\rm res}$









 ^1H NMR (400 MHz, CDCl₃) of compound 5q



 ^{13}C NMR (101 MHz, CDCl₃) of compound **5**q



 ^{31}P NMR (162 MHz, CDCl₃) of compound 5q



140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 f1 (ppm)

 ^1H NMR (400 MHz, CDCl₃) of compound 5r



 ^{13}C NMR (101 MHz, CDCl₃) of compound 5r



I (ppm)





¹H NMR (400 MHz, CDCl₃) of compound 7a



 ^{13}C NMR (101 MHz, CDCl₃) of compound 7a



 ^1H NMR (400 MHz, CDCl₃) of compound 7b







 ^1H NMR (400 MHz, CDCl_3) of compound 7c


¹³C NMR (101 MHz, CDCl₃) of compound **7**c



¹H NMR (400 MHz, CDCl₃) of compound **7d**







 ^1H NMR (400 MHz, CDCl_3) of compound 7e



S74

 ^{13}C NMR (101 MHz, CDCl₃) of compound 7e



 ^1H NMR (400 MHz, CDCl₃) of compound 7f





 ^{13}C NMR (101 MHz, CDCl₃) of compound 7f



 ^1H NMR (400 MHz, CDCl₃) of compound 7g



 ^{13}C NMR (101 MHz, CDCl₃) of compound **7g**



 ^1H NMR (400 MHz, CDCl₃) of compound 7h



 $^{13}\mathrm{C}$ NMR (101 MHz, CDCl_3) of compound 7h



 ^1H NMR (400 MHz, CDCl_3) of compound 7i







 ^1H NMR (400 MHz, CDCl₃) of compound 7j



 ^{13}C NMR (101 MHz, CDCl₃) of compound 7j



 ^1H NMR (400 MHz, CDCl_3) of compound 7k



S80

 ^{13}C NMR (101 MHz, CDCl₃) of compound 7k



 ^1H NMR (400 MHz, CDCl₃) of compound 71



 $^{13}\mathrm{C}$ NMR (101 MHz, CDCl₃) of compound 71



 ^1H NMR (400 MHz, CDCl₃) of compound 7m







 $^1\mathrm{H}$ NMR (400 MHz, CDCl_3) of compound 7n







¹H NMR (400 MHz, CDCl₃) of compound **70**







 $^1\mathrm{H}$ NMR (400 MHz, CDCl_3) of compound 7p







 ^1H NMR (400 MHz, CDCl₃) of compound 7q



 ^{13}C NMR (101 MHz, CDCl₃) of compound 7q



 ^1H NMR (400 MHz, CDCl_3) of compound 7r







 1 H NMR (400 MHz, CDCl₃) of compound 7s







 $^1\mathrm{H}$ NMR (400 MHz, CDCl_3) of compound 7t



^{13}C NMR (101 MHz, CDCl₃) of compound 7t

