**Diversity-Oriented Synthesis of Chromone Inden-1-one fused Cyclopentadienylides and C-acylated Chromone Adducts via Allylic Phosphorus Ylides**

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I. General Information

a) Materials and reagents

All reactions were carried out under argon atmosphere in oven-dried glassware with magnetic stirring. All solvents and reagents were used as purchased from commercial suppliers without further purification. Triethylamine (Et₃N) was freshly distilled from calcium hydride under argon atmosphere and stored over 4 Å molecular sieves. Starting materials which were not commercially available were synthesized according to the previously reported methods.

b) Instrumentation

**Thin layer chromatography (TLC):** TLC analyses were performed on precoated aluminum-backed silica gel plate (Merck 60 F254, 0.2 mm thickness) which was visualized by florescence quenching.

**Flash Column Chromatography:** The crude products were purified on silica gel (Merck Kieselgel 60 230-400 mesh).

**NMR Spectroscopy:** ¹H, ¹³C{¹H}, ¹⁹F{¹H}, and ³¹P{¹H}-NMR spectra were recorded on an Oxford JEOL 400 MHz spectrometer, a Bruker Ascend 400 MHz spectrometer (400 MHz for ¹H, 100 MHz for ¹³C, 376 MHz for ¹⁹F, and 162 MHz for ³¹P). All NMR spectra were recorded at 299 K unless otherwise noted. Chemical shifts are reported in δ ppm referenced to an internal standard, such as TMS for ¹H-NMR (δ = 0.0 ppm), CDCl₃ for ¹³C-NMR (δ = 77.0 ppm), CD₂Cl₂ for ¹³C-NMR (δ = 53.5 ppm). The following abbreviations were used to explain the multiplicities: s (singlet), d (doublet), t (triplet), q (quartet), sep (septet), m (multiplet), brs (broad singlet), dd (doublet of doublet), td (triplet of doublet), dt (doublet of triplet of doublet) and p (pseudo). Coupling constants (J) are reported in Hertz (Hz).

**Single Crystal X-Ray Diffraction:** The X-ray diffraction measurements were carried out at 200 K or 224 K on either a Bruker D8 Venture or a Bruker KAPPA APEX II CCD area detector system equipped with a graphite monochromator, a Mo-Kα fine-focus sealed tube (k = 0.71073 Å) or a Cu-Kα fine-focus sealed tube (k = 1.54178 Å).

**Melting Point:** Melting points were measured on a hot stage melting point apparatus and were uncorrected.

**High-Resolution Mass Spectrometry (HRMS):** HRMS were recorded on Waters XeVo G2-S QTof using ESI (TOF analyzer) or JEOL JMS-700 using EI (double-focusing magnetic sector). UltrafleXtreme MALDI-TOF/TOF using MALDI (Bruker
II. Preliminary studies

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*a* Yield of the products 3a, 4 & 5 were determined by ¹H NMR analysis of the crude reaction mixture using triphenyl methane as an internal standard. †Reaction carried out at 60 °C.

III. Reaction conditions and plausible reaction mechanism for compound 6.
IV. Detailed optimization of compound 5aa.\textsuperscript{a}

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\textsuperscript{a}Unless otherwise specified, all reactions were carried out with 1a (0.3 mmol), PhCOCl 2a, Base and PR\textsubscript{3}, in the given anhydrous solvent (1.5 mL) under argon atmosphere at 30 °C. \textsuperscript{b}Yield of the products 5aa & 4, 1a
were determined by $^1$H NMR analysis of the crude reaction mixture using triphenyl methane as an internal standard. Reaction carried out at 60 °C.

V. a) Typical Procedure for the Preparation of compound 1 (TP-A)

Following the reported procedure$^1$, a round-bottomed flask equipped with a magnetic stir bar was charged with 3-Formylchromone$^2$ (5.0 mmol.), 1,3-indanedione (1.0 equiv.), and (±)-Camphor-10-Sulfonic Acid (CSA) (0.2 equiv.) in water: EtOH (1:1) at 80 °C in an oil bath for 4 h. After that, the resulting mixture was filtered under a vacuum and the residue was washed with methanol (2 times) then ethyl ether (2 times) to obtain product 1.

b) Typical Procedure for the Preparation of compound 3 (TP-B).

A dry and argon-flushed 10 mL Schlenk flask equipped with a magnetic stir bar was charged with 1 (0.3 mmol.), PBu$_3$ (1.2 equiv.), PhCOCl 2a (3.0 equiv.), and Et$_3$N (3.6 equiv.) in anhydrous CH$_2$Cl$_2$ (1.5 mL) at 30 °C. After completion of the reaction, the reaction mixture was diluted with CH$_2$Cl$_2$ (4 mL), and the organic phase was washed with sodium bicarbonate solution. The resulted organic layer was dried over sodium sulfate and filtered through filter paper then the organic solution was concentrated under vacuum. Further, the crude reaction mixture was purified by flash column chromatography on silica gel to obtain the desired products 3.

c) Typical Procedure for the Preparation of compound 5 (TP-C).
A dry and argon-flushed 10 mL Schlenk flask equipped with a magnetic stir bar was charged with compound 1 (0.3 mmol), PPhMe₂ (0.2 equiv.), R¹COCl (1.2 equiv.), and Et₃N (1.3 equiv.) in anhydrous THF (1.5 mL) at 30 °C. After completion of the reaction, the solvent was removed under vacuum and the crude residue was subjected to flash column chromatography on silica gel to obtain the desired products 5.

VI. Characterization of all Compounds

2-((4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1a.

Following the TP-A, 1a was obtained from 4-oxo-4H-chromene-3-carbaldehyde (0.87 g, 5.0 mmol), 1H-indene-1,3(2H)-dione (0.73 g, 1.0 equiv.), and CSA (0.23 g, 0.2 equiv.). The residue was filtered under vacuum and obtain 1a as a pale yellow solid (1.2 g, 80% yield). Rᵣ = 0.50 (Hexanes:EtOAc = 7:3) : mp.: 277.3-278.5 °C.

¹H NMR (400 MHz, CDCl₃) δ/ppm: 10.40 (s, 1H), 8.44 (s, 1H), 8.32 (dd, J = 8.1, 1.6 Hz, 1H), 8.08-7.98 (m, 2H), 7.89-7.80 (m, 2H), 7.75 (dd, J = 8.7, 7.2, 1.7 Hz, 1H), 7.57 (dd, J = 8.4, 0.5 Hz, 1H), 7.50 (dd, J = 9.1, 7.2, 0.9 Hz, 1H).

¹³C¹H]-NMR (100 MHz, CDCl₃) δ/ppm: 190.2, 189.0, 175.3, 163.4, 156.1, 142.1, 140.4, 136.5, 135.6, 135.4, 134.5, 129.4, 126.7, 126.4, 124.0, 123.6, 123.4, 118.6, 118.7.


2-(5-methyl-2-phenylbenzofuran-3-yl)-1-oxo-1H-inden-3-yl pivalate 1b.

Following the TP-A, 1b was obtained from 6-methyl-4-oxo-4H-chromene-3-carbaldehyde (0.94 g, 5.0 mmol), and 1H-indene-1,3(2H)-dione (0.73 g, 1.0 equiv.), CSA (0.23 g, 0.2 equiv.). The residue was filtered under vacuum and obtain 1b as a pale green solid (1.1 g, 70%). Rᵣ = 0.50 (Hexanes:EtOAc = 8:2) : mp.: 228.2-229.4 °C.

¹H NMR (400 MHz, CDCl₃) δ/ppm: 10.37 (s, 1H), 8.43 (pd, J = 0.6 Hz, 1H), 8.09 (pd, J = 1.1 Hz, 1H), 8.06-7.97 (m, 2H), 7.87-7.79 (d, 2H), 7.54 (d, J = 8.7, 2.2 Hz, 1H), 7.45 (pd, J = 8.5 Hz, 1H), 2.49 (s, 3H).

¹³C¹H]-NMR (100 MHz, CDCl₃) δ/ppm: 190.2, 189.1, 175.3, 163.4, 154.3, 142.1, 140.3, 136.8, 136.6, 135.6, 135.5, 135.3, 129.1, 126.0, 123.6, 123.5, 123.3, 118.4, 118.3,
20.9.
HRMS (EI) m/z: [M]+ calcd for C_{20}H_{12}O_{4}: 316.0736 found: 316.0737.

2-(5-methoxy-2-phenylbenzofuran-3-yl)-1-oxo-1H-inden-3-yl pivalate 1c.

Following the TP-A, 1c was obtained from 6-methoxy-4-oxo-4H-chromene-3-carbaldehyde (1.02 g, 5.0 mmol), and 1H-indene-1,3(2H)-dione (0.73 g, 1.0 equiv.), CSA (0.23 g, 0.2 equiv.). The residue was filtered under a vacuum and obtain 1c as a pale green solid (1.2 g, 72%). R_f = 0.38 (Hexanes:EtOAc = 7:3); mp: 266.7-267.7 °C.

1H NMR (400 MHz, CDCl_3) δ/ppm: 10.37 (s, 1H), 8.46 (s, 1H), 8.08 (dd, J = 3.1 Hz, 1H), 7.49 (dd, J = 9.1 Hz, 1H), 7.32 (dd, J = 9.1, 3.1 Hz, 1H), 3.93 (s, 3H).

13C{1H}-NMR (100 MHz, CDCl_3) δ/ppm: 190.2, 189.9, 175.5, 157.8, 150.8, 142.1, 140.3, 136.9, 135.5, 135.3, 129.1, 124.8, 124.3, 123.5, 123.4, 120.0, 117.8, 105.9, 50.1.

HRMS (EI) m/z: [M]+ calcd for C_{20}H_{12}O_{4}: 332.0685 found: 332.0677.

2-((6-fluoro-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1d.

Following the TP-A, 1d was obtained from 6-fluoro-4-oxo-4H-chromene-3-carbaldehyde (0.96 g, 5.0 mmol), and 1H-indene-1,3(2H)-dione (0.73 g, 1.0 equiv.), CSA (0.23 g, 0.2 equiv.). The residue was filtered under a vacuum and obtain 1d as a half-white solid (1.1 g, 68%). R_f = 0.40 (Hexanes:EtOAc = 8:2); mp: 265.8-266.9 °C

1H NMR (400 MHz, CDCl_3) δ/ppm: 10.39 (dd, J = 0.6 Hz, 1H), 8.39 (s, 1H), 8.06-8.00 (m, 2H), 7.96 (dd, J = 8.6, 3.1 Hz, 1H), 7.88-7.80 (m, 2H), 7.60 (dd, J = 9.1, 4.1 Hz, 1H), 7.51-7.43 (m, 1H).

13C{1H}-NMR (100 MHz, CDCl_3) δ/ppm: 190.1, 188.9, 174.1, 163.2, 154.8, 142.2, 140.4, 137.5, 135.7, 129.8, 129.3, 125.2, 123.6, 123.5, 120.5, 119.9, 118.7.

19F NMR (376 MHz, CDCl_3) δ/ppm: -112.9.

HRMS (EI) m/z: [M]+ calcd for C_{19}H_{9}FO_{4}: 320.0485 found: 320.0466.

2-((6-chloro-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1e.
Following the TP-A, 1e was obtained from 6-chloro-4-oxo-4H-chromene-3-carbaldehyde (1.04 g, 5.0 mmol), and 1H-indene-1,3(2H)-dione (0.73 g, 1.0 equiv.), CSA (0.23 g, 0.2 equiv.). The residue was filtered under a vacuum and obtain 1e as a half-white solid (1.0 g, 60%). Rf = 0.40 (Hexanes:EtOAc = 8:2); mp.: 271.2-272.3 °C. 

\[ ^1H \text{NMR} \ (400 \text{ MHz, CDCl}_3) \ \delta/\text{ppm}:\ 10.37 \ (s, \ 1H), 8.38 \ (s, \ 1H), 8.28 \ (d, J = 2.6 \text{ Hz}, 1H), 8.06-7.99 \ (m, 2H), 7.89-7.82 \ (m, 2H), 7.69 \ (dd, J = 8.9, 2.6 \text{ Hz}, 1H), 7.53 \ (d, J = 8.9 \text{ Hz}, 1H). \]

\[ ^{13}C\{^1H\}-\text{NMR} \ (100 \text{ MHz, CDCl}_3) \ \delta/\text{ppm}:\ 190.1, 188.9, 174.1, 163.2, 154.8, 142.2, 140.4, 137.5, 135.7, 129.8, 129.3, 125.2, 123.6, 123.5, 120.5, 119.9, 118.7. \]

HRMS (EI) m/z: [M]+ calcd for C_{19}H_{35}ClO_{4}: 336.0189 found: 336.0197.

HRMS (EI) m/z: [M]+ calcd for C_{19}H_{37}ClO_{4}: 338.0160 found: 338.0156.

### 2-((6-bromo-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1f.

Following the TP-A, 1f was obtained from 6-bromo-4-oxo-4H-chromene-3-carbaldehyde (1.25 g, 5.0 mmol), and 1H-indene-1,3(2H)-dione (0.73 g, 1.0 equiv.), CSA (0.23 g, 0.2 equiv.). The residue was filtered under a vacuum and obtain 1f as a pale green solid (1.0 g, 52%). Rf = 0.38 (Hexanes:EtOAc = 9:1); mp.: 264.4-265.6 °C. 

\[ ^1H \text{NMR} \ (400 \text{ MHz, CDCl}_3) \ \delta/\text{ppm}:\ 10.37 \ (s, \ 1H), 8.45 \ (d, J = 2.4 \text{ Hz}, 1H), 8.37 \ (s, 1H), 8.07-7.97 \ (m, 2H), 7.90-7.80 \ (m, 3H), 7.47 \ (d, J = 8.9 \text{ Hz}, 1H). \]

\[ ^{13}C\{^1H\}-\text{NMR} \ (100 \text{ MHz, CDCl}_3) \ \delta/\text{ppm}:\ 190.1, 188.9, 174.1, 163.2, 154.8, 142.2, 140.4, 137.5, 135.7, 135.5, 129.8, 129.3, 125.2, 123.6, 123.5, 120.5, 119.9, 118.7. \]

HRMS (EI) m/z: [M]+ calcd for C_{19}H_{79}BrO_{4}: 379.9684 found: 379.9700.

HRMS (EI) m/z: [M]+ calcd for C_{19}H_{81}BrO_{4}: 381.9664 found: 381.9627.

### 2-((6-nitro-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1g.

Following the TP-A, 1g was obtained from 6-nitro-4-oxo-4H-chromene-3-
carbaldehyde (1.09 g, 5.0 mmol), and 1H-indene-1,3(2H)-dione (0.73 g, 1.0 equiv.), CSA (0.23 g, 0.2 equiv.). The residue was filtered under a vacuum and obtain **1g** as a pale yellow solid (1.3 g, 76%). \( R_f = 0.45 \) (Hexanes:EtOAc = 6:4); mp.: 309.2-310.5 °C.

\(^1\)H NMR (400 MHz, CDCl₃) \( \delta /ppm: 10.39 \) (s, 1H), 9.19 (d, \( J = 2.7 \) Hz, 1H), 8.58 (dd, \( J = 9.7, 2.8 \) Hz, 1H), 8.34 (s, 1H), 8.08-8.02 (m, 2H), 7.90-7.84 (m, 2H), 7.74 (d, \( J = 9.1 \) Hz, 1H).

\(^13\)C\(^{\text{1H}}\)-NMR (100 MHz, CDCl₃) \( \delta /ppm: 190.1, 188.5, 174.0, 162.8, 158.8, 145.5, 142.2, 140.5, 135.9, 135.6, 134.4, 130.8, 128.7, 123.1, 123.8, 123.6, 123.3, 120.5, 119.3.

HRMS (EI) m/z: \([M]^+\) calcd for C₁₉H₉NO₆: 347.0430 found: 347.0425.

**2-((7-methyl-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1h.**

Following the **TP-B**, **1h** was obtained from 7-methyl-4-oxo-4H-chromene-3-carbaldehyde (0.94 g, 5.0 mmol), and 1H-indene-1,3(2H)-dione (0.73 g, 1.0 equiv.), CSA (0.23 g, 0.2 equiv.). The residue was filtered under a vacuum and obtain **1h** as a half-white solid (1.25 g, 80%). \( R_f = 0.45 \) (Hexanes:EtOAc = 8:2); mp.: 224.4-225.6 °C.

\(^1\)H NMR (400 MHz, CDCl₃) \( \delta /ppm: 10.34 \) (s, 1H), 8.40 (s, 1H), 8.17 (d, \( J = 8.2 \) Hz, 1H), 8.06-7.94 (m, 2H), 7.87-7.78 (m, 2H), 7.33 (s, 1H), 7.28 (d, \( J = 8.0 \) Hz, 1H), 2.51 (s, 3H).

\(^13\)C\(^{\text{1H}}\)-NMR (100 MHz, CDCl₃) \( \delta /ppm: 190.1, 189.1, 175.1, 163.3, 156.1, 146.1, 142.0, 136.6, 135.5, 135.3, 129.0, 127.8, 126.3, 123.4, 123.3, 121.6, 118.4, 118.3, 21.8.

HRMS (EI) m/z: \([M]^+\) calcd for C₂₀H₁₂O₄: 316.0736 found: 316.0752.

**2-((7-methoxy-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1i.**

Following the **TP-B**, **1i** was obtained from 7-methoxy-4-oxo-4H-chromene-3-carbaldehyde (1.02 g, 5.0 mmol), and 1H-indene-1,3(2H)-dione (0.73 g, 1.0 equiv.), CSA (0.23 g, 0.2 equiv.). The residue was filtered under a vacuum and obtain **1i** as a half-white solid (1.1 g, 68%). \( R_f = 0.45 \) (Hexanes:EtOAc =7:2); mp.: 268.8-269.3 °C.

\(^1\)H NMR (400 MHz, CDCl₃) \( \delta /ppm: 10.34 \) (s, 1H), 8.44 (s, 1H), 8.22 (d, \( J = 8.8 \) Hz, 1H) 8.09-7.97 (m, 2H), 7.89-7.77 (m, 2H), 7.04 (dd, \( J = 9.2, 2.5 \) Hz, 1H), 6.94 (d, \( J = 2.3 \) Hz, 1H), 3.64 (s, 3H).
\(^{13}\)C\(^{1}\)H\(\text{-NMR}\) (100 MHz, CDCl\(_3\)) \(\delta/\text{ppm}: 190.3, 189.1, 174.5, 164.7, 163.1, 157.8, 142.1, 140.4, 136.8, 135.5, 135.3, 129.2, 128.1, 123.5, 123.3, 118.6, 117.7, 115.4, 101.0, 55.9.

HRMS (EI) m/z: [M]\(^+\) calcd for C\(_{20}\)H\(_{12}\)O\(_5\): 332.0685 found: 332.0668.

2-((7-fluoro-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1j.

Following the TP-A, 1j was obtained from 7-fluoro-4-oxo-4H-chromene-3-carbaldehyde (0.96 g, 5.0 mmol), and 1H-indene-1,3(2H)-dione (0.73 g, 1.0 equiv.), CSA (0.23 g, 0.2 equiv.). The residue was filtered under a vacuum and obtain 1j as a half-white solid (1.1 g, 76%). \(R_f = 0.48\) (Hexanes:EtOAc =8:2) ; mp.: 284.3-285.5 \(^\circ\)C.

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta/\text{ppm}: 10.36\) (s, 1H), 8.39 (s, 1H), 8.34 (dd, \(J = 10, 6.2\) Hz, 1H), 8.08-7.99 (m, 2H), 7.87-7.82 (m, 2H), 7.33-7.16 (m, 3H).

\(^{13}\)C\(^{1}\)H\(\text{-NMR}\) (100 MHz, CDCl\(_3\)) \(\delta/\text{ppm}: 190.1, 188.9, 174.3, 167.3, 164.8, 163.2, 157.1 (d, \(J_{C-F} = 13.3\) Hz) 142.2, 140.4, 135.8, 135.7, 135.5, 129.8, 129.1, 123.6, 123.4, 120.8, 118.7, 115.1 (d, \(J_{C-F} = 22.8\) Hz), 105.4 (d, \(J_{C-F} = 24.5\) Hz).

\(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta/\text{ppm}: -100.9.

HRMS (EI) m/z: [M]\(^+\) calcd for C\(_{19}\)H\(_9\)FO\(_4\):320.0485 found: 320.0464.

2-((8-nitro-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1k.

Following the TP-A, 1k was obtained from 8-nitro-4-oxo-4H-chromene-3-carbaldehyde (1.1 g, 5.0 mmol), and 1H-indene-1,3(2H)-dione (0.73 g, 1.0 equiv.), CSA (0.23 g, 0.2 equiv.). The residue was filtered under a vacuum and obtain 1k as a brown solid (1.1 g, 76%). \(R_f = 0.50\) (Hexanes:EtOAc =7:3) ; mp.: 249.3-250.8 \(^\circ\)C.

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta/\text{ppm}: 10.40\) (s, 1H), 7.54 (d, \(J = 8.1, 1.7\) Hz, 1H), 7.54 (d, \(J = 8.2, 1.6\) Hz, 1H), 8.31 (s, 1H), 8.08-8.01 (m, 2H), 7.92-7.82 (d, 2H), 7.62 (t, \(J = 7.9\) Hz, 1H).

\(^{13}\)C\(^{1}\)H\(\text{-NMR}\) (100 MHz, CDCl\(_3\)) \(\delta/\text{ppm}: 189.7, 188.6, 173.5, 162.3, 148.3, 142.2, 140.5, 139.2, 135.9, 135.7, 133.9, 132.3, 130.9, 130.3, 125.6, 125.5, 123.7, 123.6, 119.3.

HRMS (EI) m/z: [M]\(^+\) calcd for C\(_{19}\)H\(_9\)NO\(_6\):347.0430 found: 347.0423.
2-((6,8-dimethyl-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 11.

Following the TP-A, 11 was obtained from 6,8-dimethyl-4-oxo-4H-chromene-3-carbaldehyde (1.0 g, 5.0 mmol), and 1H-indene-1,3(2H)-dione (0.73 g, 1.0 equiv.), CSA (0.23 g, 0.2 equiv.). The residue was filtered under a vacuum and obtain 11 as a pale green solid (1.05 g, 64%). Rf = 0.45 (Hexanes:EtOAc = 7:3); mp.: 296.6-297.8 °C.

1H NMR (400 MHz, CDCl3) δ/ppm: 10.41 (s, 1H), 8.46 (s, 1H), 8.06-7.98 (m, 2H), 7.94 (pd, J = 0.8 Hz, 1H), 7.87-7.79 (m, 2H), 7.39 (pd, J = 0.9 Hz, 1H), 2.51 (s, 3H), 2.44 (s, 3H).

13C{1H}-NMR (100 MHz, CDCl3) δ/ppm: 190.3, 189.2, 175.7, 163.1, 152.9, 142.1, 140.3, 137.1, 136.8, 135.9, 135.5, 135.3, 128.9, 127.8, 127.6, 123.6, 123.5, 123.3, 118.2, 20.9, 15.3.


2-((6,8-dibromo-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1m.

Following the TP-A, 1m was obtained from 6,8-dibromo-4-oxo-4H-chromene-3-carbaldehyde (1.65 mg, 5.0 mmol), and 1H-indene-1,3(2H)-dione (0.73 g, 1.0 equiv.), CSA (0.23 g, 0.2 equiv.). The residue was filtered under a vacuum and obtain 1m as a pale green solid (1.2 g, 55%). Rf = 0.50 (Hexanes:EtOAc = 7:3); mp: 302.5-303.6 °C.

1H NMR (400 MHz, CDCl3) δ/ppm: 10.41 (s, 1H), 8.39 (d, J = 2.5 Hz, 1H, 1H), 8.33 (s, 1H), 8.09 (d, J = 2.2 Hz, 1H, 1H), 8.07-8.00 (m, 2H), 7.90-7.82 (m, 2H).

13C{1H}-NMR (100 MHz, CDCl3) δ/ppm: 189.9, 188.7, 173.7, 162.8, 151.9, 142.2, 140.4, 140.2, 135.8, 135.6, 134.6, 130.4, 128.6, 128.6, 125.9, 123.7, 123.6, 119.8, 118.7, 133.4.


2-((1-oxo-1H-benzo[f]chromen-2-yl)methylene)-1H-indene-1,3(2H)-dione 1n.

Following the TP-A, 1n was obtained from 1-oxo-1H-benzo[f]chromene-2-carbaldehyde (1.1 g, 5.0 mmol), and 1H-indene-1,3(2H)-dione (0.73 g, 1.0 equiv.), CSA (0.23 g, 0.2 equiv.). The residue was filtered under a vacuum and obtain 1n as a half-white solid (1.3 g, 76%). \( R_f = 0.38 \) (Hexanes:EtOAc = 8:2) ; mp.: 297.4-298.5 °C.

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta/\text{ppm} \): 10.56 (s, 1H), 8.61-8.53 (m, 1H), 8.49 (s, 1H), 8.24 (d, \( J = 8.7 \) Hz, 1H), 8.09-8.01 (m, 2H), 8.00-7.93 (m, 1H), 7.89-7.82 (m, 3H), 7.78-7.69 (m, 2H).

\(^{13}\)C\(^{\{1\}H\}\)-NMR (100 MHz, CDCl\(_3\)) \( \delta/\text{ppm} \): 190.2, 188.9, 175.1, 162.3, 153.6, 142.2, 140.4, 136.9, 136.4, 135.6, 135.4, 131.1, 130.6, 129.8, 129.6, 127.3, 127.2, 123.6, 123.4, 120.7, 117.5.

HRMS (EI) m/z: [M]\(^+\) calcd for C\(_{23}\)H\(_{12}\)O\(_4\): 352.0736 found: 352.0741.

2-((4-oxo-4H-benzo[h]chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1o.

Following the TP-A, 1o was obtained from 4-oxo-4H-benzo[h]chromene-3-carbaldehyde (1.1 g, 5.0 mmol), and 1H-indene-1,3(2H)-dione (0.73 g, 1.0 equiv.), CSA (0.23 g, 0.2 equiv.). The residue was filtered under a vacuum and obtain 1o as a yellow solid (1.4 g, 80%). \( R_f = 0.47 \) (Hexanes:EtOAc = 8:2) ; mp.: 287.3-288.2 °C.

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta/\text{ppm} \): 10.56 (s, 1H), 8.61-8.53 (m, 1H), 8.58 (s, 1H), 8.25 (d, \( J = 8.7 \) Hz, 1H), 8.81-8.01 (m, 2H), 7.99-7.93 (m, 1H), 7.87-7.82 (m, 3H), 7.79-7.69 (m, 2H).

\(^{13}\)C\(^{\{1\}H\}\)-NMR (100 MHz, CDCl\(_3\)) \( \delta/\text{ppm} \): 190.2, 188.9, 175.1, 162.3, 153.6, 142.2, 140.4, 136.3, 136.2, 135.6, 135.4, 129.8, 129.7, 128.2, 127.6, 126.4, 123.9, 123.6, 123.4, 122.3, 121.3, 120.4, 119.7.

HRMS (EI) m/z: [M]\(^+\) calcd for C\(_{23}\)H\(_{12}\)O\(_4\): 352.0736 found: 352.0719.
2-((5-hydroxy-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1p.

Following the TP-A, 1p was obtained from 5-hydroxy-4-oxo-4H-chromene-3-carbaldehyde (0.95 g, 5.0 mmol), and 1H-indene-1,3(2H)-dione (0.73 g, 1.0 equiv.), CSA (0.23 g, 0.2 equiv.). The residue was filtered under a vacuum and obtain 1p as a yellow solid (1.2 g, 75%). Rf = 0.43 (Hexanes:EtOAc =7:3) ; mp.: 249.1-250.2 °C.

\(^1\)H NMR (400 MHz, CDCl\(_3\)) δ/ppm: 12.31 (s, 1H), 10.38 (s, 1H), 8.31 (s, 1H), 8.13-7.97 (m, 2H) 7.90-7.77 (m, 2H), 7.60 (t, J = 8.3 Hz, 1H), 7.01 (pd, J = 8.4, 0.6 Hz, 1H), 6.89 (pd, J = 8.4, 0.6 Hz, 1H).

\(^13\)C{\(^1\)H} NMR (100 MHz, CDCl\(_3\)) δ/ppm: 180.6, 172.7, 155.9, 138.4, 134.3, 129.6, 123.6, 123.5, 117.3, 112.9, 110.7, 107.7.

HRMS (EI) m/z: [M]⁺ calcd for C\(_{21}\)H\(_{10}\)O\(_5\) : 318.0528 found: 318.0517.

5,6-dimethoxy-2-((4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1q.

Following the TP-A, 1q was obtained from 4-oxo-4H-chromene-3-carbaldehyde (0.87 g, 5.0 mmol), and 5,6-dimethoxy-1H-indene-1,3(2H)-dione (1.03 g, 5.0 mmol.), CSA (0.23 g, 0.2 equiv.). The residue was filtered under a vacuum and obtain 1q as a yellow solid (1.2 g, 68%). Rf = 0.49 (Hexanes:EtOAc = 5:5); mp.: 271.7-272.8 °C.

\(^1\)H NMR (400 MHz, CDCl\(_3\)) δ/ppm: 10.28 (s, 1H), 8.33 (d, J = 7.8 Hz, 1H), 8.26 (s, 1H), 7.74 (t, J = 8.3 Hz, 1H), 7.56 (d, J = 8.5 Hz, 1H), 7.48 (t, J = 7.8 Hz, 1H), 7.04 (d, J = 11.4 Hz, 1H), 7.33 (s, 2H), 4.03 (s, 6H).

\(^13\)C{\(^1\)H} NMR (100 MHz, CDCl\(_3\)) δ/ppm: 196.6, 162.7, 155.9, 138.4, 134.3, 133.1, 126.6, 126.2, 118.6, 118.5, 103.9, 103.8, 103.2, 56.7, 44.7.

HRMS (EI) m/z: [M]⁺ calcd for C\(_{21}\)H\(_{14}\)O\(_6\) : 362.0790 found: 362.0775.
2-((4-oxo-4H-chromen-3-yl)methylene)-1,3-diphenylpropane-1,3-dione 6.

Following the reported procedure, a 50 mL round-bottomed flask equipped with a magnetic stir bar was charged with 4-oxo-4H-chromene-3-carbaldehyde (0.87 g, 5.0 mmol.), 1,3-diphenylpropane-1,3-dione (1.6 g, 1.5 equiv.), K$_2$CO$_3$ (0.69 g, 0.2 equiv.) and acetic anhydride (30.0 mL). The reaction mixture was stirred for 8 h at 80 °C. After completion of the reaction, the residue was purified by column chromatography (SiO$_2$, Hexanes:EtOAc = 65:35) to give 6 as a half-white solid (1.45 g, 76%). $R_f$ = 0.45 (Hexanes:EtOAc = 7:3); mp.: 177.0-178.1 °C.

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$/ppm: 8.26 (d, $J$ = 0.7 Hz, 1H), 8.17 (d, $J$ = 8.2, 1.8 Hz, 1H), 8.00-7.91 (m, 4H), 7.68 (d, $J$ = 0.9 Hz, 1H), 7.67-7.56 (m, 2H), 7.55-7.47 (m, 3H), 7.45-7.36 (m, 4H).

$^{13}$C($^1$H)-NMR (100 MHz, CDCl$_3$) $\delta$/ppm: 190.1, 188.8, 180.6, 164.2, 161.5, 156.1, 142.1, 140.4, 136.4, 135.7, 135.5, 134.3, 129.6, 123.6, 123.5, 117.3, 112.9, 110.7, 107.7. HRMS (EI) m/z: [M]$^+$ calcd for C$_{25}$H$_{16}$O$_4$: 380.1049 found: 380.1068.


Following the TP-B, 3a was obtained from 2-((4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1a (90.75 mg, 0.3 mmol), PBU$_3$ (88.8µL, 1.2 equiv.), benzoyl chloride 2a (104.5 µL, 3.0 equiv.) and Et$_3$N (150.5 µL, 3.6 equiv.) in anhydrous CH$_2$Cl$_2$ (1.5 mL) at 30 °C for 8 h. After workup, the residue was purified by column chromatography (SiO$_2$, Hexanes:EtOAc = 70:30) to give 3a as a dark red solid (135.0 mg, 92%). $R_f$ = 0.30 (Hexanes:EtOAc = 9:1); mp.: 184.2-185.1 °C.

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$/ppm: 8.23 (dd, $J$ = 7.9, 1.9 Hz 1H), 7.57 (dt, $J$ = 9.0, 7.0, 1.7 Hz 1H), 7.46-7.44 (m, 1H), 7.33 (d, $J$ = 7.2 Hz 1H), 7.29-7.25 (m, 1H), 6.94-6.88 (m, 1H), 2.73-2.54 (m, 6H), 1.48-1.44 (m, 12H), 0.93-0.89 (t, $J$ = 8.0 Hz, 9H).

$^{13}$C($^1$H)-NMR (100 MHz, CDCl$_3$) $\delta$/ppm: 189.7, 174.2, 156.3, 146.1 (d, $^3$J$_{C-P}$ = 12.4 Hz), 141.3, 139.3, 134.4, (d, $^3$J$_{C-P}$ = 9.4 Hz), 133.8, 132.3, 126.1, 124.7, 123.3, 122.8, 122.6 (d, $^3$J$_{C-P}$ = 10.5 Hz), 122.5, 122.3, (d, $^3$J$_{C-P}$ = 12.7 Hz), 118.9, 117.5, 82.8, (d, $^1$J$_{C-P}$ = 102.9 Hz), 24.1, (d, $^3$J$_{C-P}$ = 4.0 Hz), 23.8, (d, $^2$J$_{C-P}$ = 16.0 Hz), 21.3, (d, $^1$J$_{C-P}$ = 52.3
$31^P$ NMR (162 MHz, CDCl$_3$) δ/ppm: 22.0.

HRMS (EI) m/z: [M]$^+$ calcd for C$_{31}$H$_{35}$O$_3$P: 486.2324 found: 486.2310.


Following the TP-B, 3b was obtained from 2-((6-methyl-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1b (94.89 mg, 0.3 mmol), PBu$_3$ (88.8 µL, 1.2 equiv.), 2a (104.5 µL, 3.0 equiv.), and Et$_3$N (150.5 µL, 3.6 equiv.) in anhydrous CH$_2$Cl$_2$ (1.5 mL) at 30 °C for 48 h. After workup, the residue was purified by column chromatography (SiO$_2$, Hexanes:EtOAc = 70:30) to give 3b as a dark red solid (138.0 mg, 90%). $R_f$ = 0.45 (Hexanes:EtOAc = 8:2); mp.: 179.9-181.1 °C.

$^1$H NMR (400 MHz, CDCl$_3$) δ/ppm: 8.03 (s, 1H), 7.40-7.30 (m, 3H), 7.28-7.20 (m, 2H), 6.95-6.85 (m, 1H), 2.70-2.58 (m, 6H), 2.43 (s, 3H), 1.53-1.39 (m, 12H), 0.92 (t, $J$ = 6.7 Hz, 9H).

$^{13}$C{$_1$H}-NMR (100 MHz, CDCl$_3$) δ/ppm: 189.6, 174.3, 154.5, 146.3 (d, $^2$J$_{C-P}$ = 11.9 Hz), 141.3, 139.2, 139.1, 135.4 (d, $^3$J$_{C-P}$ = 9.7 Hz), 133.8, 133.4, 132.0, 125.6, 124.5, 123.2, 122.4 (d, $^3$J$_{C-P}$ = 10.6 Hz), 122.3 (d, $^2$J$_{C-P}$ = 11.2 Hz), 121.2, 118.9, 117.2, 82.0 (d, $^1$J$_{C-P}$ = 104.2 Hz), 24.1 (d, $^3$J$_{C-P}$ = 3.9 Hz), 23.7 (d, $^2$J$_{C-P}$ = 15.7 Hz), 21.5 (d, $^1$J$_{C-P}$ = 52.7 Hz), 20.7, 13.5.

$31^P$ NMR (162 MHz, CDCl$_3$) δ/ppm: 22.0.

HRMS (EI) m/z: [M]$^+$ calcd for C$_{32}$H$_{37}$O$_3$P: 500.2480 found: 500.2462.


Following the TP-B, 3c was obtained from 2-((6-methoxy-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1c (99.69 mg, 0.3 mmol), PBu$_3$ (88.8 µL, 1.2 equiv.), benzoyl chloride 2a (104.5 µL, 3.0 equiv.), and Et$_3$N (150.5 µL, 3.6 equiv.) in anhydrous CH$_2$Cl$_2$ (1.5 mL) at 30 °C for 24 h. After workup, the residue was purified by column chromatography (SiO$_2$, Hexanes:EtOAc = 70:30) to give 3c as a dark red
solid (114.5 mg, 73%). $R_f = 0.45$ (Hexanes:EtOAc = 8:2); mp.: 115.3-116.2 °C.

$^1H$ NMR (400 MHz, CDCl$_3$) $\delta$/ppm: 7.65 (d, $J = 3.2$ Hz, 1H), 7.38 (d, $J = 9.1$, 1H), 7.31 (d, $J = 7.3$ Hz, 1H), 7.28-7.21 (m, 1H), 7.18 (dd, $J = 9.1$, 3.2 Hz, 1H), 6.94-6.86 (m, 1H), 2.75-2.53 (m, 6H), 1.55-1.37 (m, 12H), 0.91 (t, $J = 6.7$ Hz, 9H).

$^{13}C$($^1H$)-NMR (100 MHz, CDCl$_3$) $\delta$/ppm: 189.7, 173.9, 155.2, 151.1, 146.5 (d, $^2J_{C-P} = 12.1$ Hz), 141.4, 139.2, 134.6 (d, $^3J_{C-P} = 9.9$ Hz), 133.8, 124.6, 123.2, 122.9, 122.4 (d, $^3J_{C-P} = 10.4$ Hz), 122.1 (d, $^2J_{C-P} = 12.3$ Hz), 121.9, 118.9, 118.7, 105.9, 81.6 (d, $^1J_{C-P} = 103.3$ Hz), 55.8, 24.2 (d, $^3J_{C-P} = 3.8$ Hz), 23.8 (d, $^2J_{C-P} = 15.8$ Hz), 21.5 (d, $^1J_{C-P} = 52.8$ Hz), 13.5.

$^{31}P$ NMR (162 MHz, CDCl$_3$) $\delta$/ppm: 21.9.

HRMS (EI) m/z: [M]$^+$ calcd for C$_{32}$H$_{37}$O$_4$P: 516.2429 found: 516.2454.


Following the TP-B, 3d was obtained from 2-((6-fluoro-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1d (96.01 mg, 0.3 mmol), PbBu$_3$ (88.8µL, 1.2 equiv.), benzoyl chloride 2a (104.5 µL, 3.0 equiv.), and Et$_3$N (150.5 µL, 3.6 equiv.) in anhydrous CH$_2$Cl$_2$ (1.5 mL) at 30 °C for 8 h. After workup, the residue was purified by column chromatography (SiO$_2$, Hexanes:EtOAc = 70:30) to give 3d as a dark red solid (135.0 mg, 89%). $R_f = 0.46$ (Hexanes:EtOAc = 9:1); mp.: 169.7-170.5 °C

$^1H$ NMR (400 MHz, CDCl$_3$) $\delta$/ppm: 7.87 (dd, $J = 9.0$, 3.1 Hz, 1H), 7.43 (dd, $J = 9.1$, 4.3 Hz, 1H), 7.34 (d, $J = 7.4$ Hz, 1H), 7.3-7.28 (m, 1H), 7.3-7.2 (m, 1H), 6.94-6.90 (td, $J = 6.9$, 2.0 Hz, 1H), 2.80-2.52 (m, 6H), 1.55-1.37 (m, 12H), 0.92 (t, $J = 6.9$ Hz, 9H).

$^{13}C$($^1H$)-NMR (100 MHz, CDCl$_3$) $\delta$/ppm: 189.7, 173.2, 159.5, 157.1, 152.4, 146.2, (d, $^2J_{C-P} = 12.0$ Hz), 141.2, 139.1, 134.9, (d, $^3J_{C-P} = 9.6$ Hz), 133.9, 124.8, 123.7, (d, $^1J_{C-F} = 6.7$ Hz), 123.4, 122.3, (d, $^3J_{C-P} = 10.6$ Hz), 121.5, (d, $^2J_{C-P} = 12.2$ Hz), 120.0 (d, $^1J_{C-F} = 25.2$ Hz), 119.8, 119.0, 118.9, 111.0 (d, $^1J_{C-F} = 23.7$ Hz), 81.1 (d, $^1J_{C-P} = 103.0$ Hz), 24.1 (d, $^3J_{C-P} = 3.9$ Hz), 23.8 (d, $^2J_{C-P} = 15.8$ Hz), 21.4 (d, $^1J_{C-P} = 52.5$ Hz), 13.5.

$^{31}P$ NMR (162 MHz, CDCl$_3$) $\delta$/ppm: 22.1.

$^{19}F$ NMR (376 MHz, CDCl$_3$) $\delta$/ppm: -119.9.

HRMS (EI) m/z: [M]$^+$ calcd for C$_{31}$H$_{34}$O$_3$PF: 504.2230 found: 504.2198.

Following the TP-B, 3e was obtained from 2-((6-chloro-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1e (101.02 mg, 0.3 mmol), PBu₃ (88.8µL, 1.2 equiv.), benzoyl chloride 2a (104.5 µL, 3.0 equiv.), and Et₃N (150.5 µL, 3.6 equiv.) in anhydrous CH₂Cl₂ (1.5 mL) at 30 °C for 8 h. After workup, the residue was purified by column chromatography (SiO₂, Hexanes:EtOAc = 70:30) to give 3e as a dark red solid (141.2 mg, 90%). Rₜ = 0.55 (Hexanes:EtOAc = 9:1); mp.: 161.7-162.8 °C.

¹H NMR (400 MHz, CDCl₃) δ/ppm: 8.20 (d, J = 2.6 Hz, 1H), 7.50 (dd, J = 8.9, 2.6 Hz, 1H), 7.4 (d, J = 8.8 Hz, 1H), 7.33 (d, J = 7.2 Hz, 1H), 6.90 (td, J = 7.2, 1.7 Hz, 1H), 2.70-2.55 (m, 6H), 1.54-1.37 (m, 12H), 0.92 (t, J = 6.9 Hz, 9H).

¹³C{¹H}-NMR (100 MHz, CDCl₃) δ/ppm: 189.6, 172.8, 154.6, 146.1 (d, ²J_C-P = 12.7 Hz), 141.2, 139.1, 135.0 (d, ²J_C-P = 9.6 Hz), 133.9, 132.1, 128.1, 125.7, 124.8, 123.9, 123.4, 122.4 (d, ²J_C-P = 10.8 Hz), 121.7 (d, ²J_C-P = 12.3 Hz), 119.1, 119.0, 82.5 (d, ¹J_C-P = 102.7 Hz), 24.1 (d, ²J_C-P = 4.1 Hz), 23.7 (d, ²J_C-P = 15.7 Hz), 21.7 (d, ¹J_C-P = 52.8 Hz), 13.5.

³¹P NMR (162 MHz, CDCl₃) δ/ppm: 22.2.


Following the TP-B, 3f was obtained from 2-((6-bromo-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1f (114.4 mg, 0.3 mmol), PBu₃ (88.8µL, 1.2 equiv.), benzoyl chloride 2a (104.5 µL, 3.0 equiv.), and Et₃N (150.5 µL, 3.6 equiv.) in anhydrous CH₂Cl₂ (1.5 mL) at 30 °C for 8 h. After workup, the residue was purified by column chromatography (SiO₂, Hexanes:EtOAc = 70:30) to give 3f as a dark red solid (148.2 mg, 87%). Rₜ = 0.50 (Hexanes:EtOAc = 8:2); mp.: 170.3-171.2 °C.

¹H NMR (400 MHz, CDCl₃) δ/ppm: 8.36 (d, J = 2.5 Hz, 1H), 7.63 (dd, J = 8.8, 2.6 Hz, 1H), 7.35-7.34 (m, 1H), 7.33 (s, 1H), 6.94-6.90 (td, J = 14.1, 7.1 Hz, 1H), 2.71-2.53 (m,
6H), 1.54-1.36 (m, 12H), 0.92 (t, J = 6.9 Hz, 9H).

$^{13}$C($^1$H)-NMR (100 MHz, CDCl$_3$) δ/ppm: 189.6, 172.7, 155.0, 145.9 (d, $^2$J$_{C-P}$ = 11.6 Hz), 141.1, 139.1, 135.0 (d, $^3$J$_{C-P}$ = 9.4 Hz), 134.9, 133.9, 128.8, 124.3, 124.3, 123.4, 122.4 (d, $^2$J$_{C-P}$ = 10.5 Hz), 121.6 (d, $^3$J$_{C-P}$ = 11.93 Hz), 119.3, 119.1, 115.5, 83.1 (d, $^1$J$_{C-P}$ = 101.8 Hz), 24.1(d, $^3$J$_{C-P}$ = 3.8 Hz), 23.8 (d, $^2$J$_{C-P}$ = 15.4 Hz), 21.4 (d, $^1$J$_{C-P}$ = 52.7 Hz), 13.5.

$^{31}$P NMR (162 MHz, CDCl$_3$) δ/ppm: 22.2.

HRMS (EI) m/z: [M]$^+$ calcd for C$_{31}$H$_{34}$O$_3$P$^{79}$Br: 564.1429 found: 564.1411.

HRMS (EI) m/z: [M]$^+$ calcd for C$_{31}$H$_{34}$O$_3$P$^{81}$Br: 566.1408 found: 566.1397.

8-nitro-11-(tributyl-5-phosphanylidene)-4c,10a,111a-tetrahydro-10H-benzo[5,6]pentaleno[1,2-b]chromene-10,12(4bH)-dione 3g.

Following the TP-B, 3g was obtained from 2-((6-nitro-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1g (104.18 mg, 0.3 mmol), PBu$_3$ (88.8 µL, 1.2 equiv.), benzoyl chloride 2a (104.5 µL, 3.0 equiv.), and Et$_3$N (150.5 µL, 3.6 equiv.) in anhydrous CH$_2$Cl$_2$ (1.5 mL) at 30 °C for 8 h. After workup, the residue was purified by column chromatography (SiO$_2$, Hexanes: EtOAc = 70:30) to give 3g as a dark red solid (128.0 mg, 80%). R$_f$ = 0.50 (Hexanes: EtOAc = 8:2); mp.: 212.5-213.6 °C.

$^1$H NMR (400 MHz, CDCl$_3$) δ/ppm: 9.15 (d, J = 2.8 Hz, 1H), 8.40 (dd, J = 9.1 2.9 Hz, 1H), 7.54 (d, J = 9.2 Hz, 1H), 7.36 (d, J = 7.4 Hz, 1H), 7.34-7.24 (m, 1H), 6.97 (td, J = 7.3, 1.2 Hz, 1H), 2.76-2.56 (m, 6H), 1.57-1.39 (m, 12H), 0.93 (t, J = 6.8 Hz, 9H).

$^{13}$C($^1$H)-NMR (100 MHz, CDCl$_3$) δ/ppm: 189.5, 172.0, 159.4, 146.3 (d, $^2$J$_{C-P}$ = 11.7 Hz), 143.1, 140.7, 139.1, 135.3 (d, $^3$J$_{C-P}$ = 9.4 Hz), 134.1, 126.6, 125.2, 123.6, 123.3, 122.9, 122.7 (d, $^3$J$_{C-P}$ = 10.4 Hz), 121.2 (d, $^2$J$_{C-P}$ = 12.3 Hz), 119.3, 118.6, 83.5 (d, $^1$J$_{C-P}$ = 102.2 Hz), 24.1 (d, $^3$J$_{C-P}$ = 4.0 Hz), 23.3 (d, $^2$J$_{C-P}$ = 15.8 Hz), 21.7 (d, $^3$J$_{C-P}$ = 52.3 Hz), 13.5.

$^{31}$P NMR (162 MHz, CDCl$_3$) δ/ppm: 22.7.

HRMS (EI) m/z: [M]$^+$ calcd for C$_{31}$H$_{34}$NO$_3$P: 531.2175 found: 531.2171.

Following the TP-B, 3h was obtained from 2-((7-methyl-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1h (94.89 mg, 0.3 mmol), PBu₃ (88.8μL, 1.2 equiv.), benzoyl chloride 2a (104.5 μL, 3.0 equiv.), and Et₃N (150.5 μL, 3.6 equiv.) in anhydrous CH₂Cl₂ (1.5 mL) at 30 °C for 48 h. After workup, the residue was purified by column chromatography (SiO₂, Hexanes:EtOAc = 70:30) to give 3h as a dark red solid (113.5 mg, 75%). Rf = 0.48 (Hexanes:EtOAc =8:2); mp.: 157.2-158.2 °C.

1H NMR (400 MHz, CDCl₃) δ/ppm: 8.11 (d, J = 8.1 Hz, 1H), 7.32 (d, J = 7.2 Hz 1H), 7.28-7.20 (m, 3H), 7.09 (d, J = 8.1 Hz, 1H), 6.96-6.85 (m, 1H), 2.74-2.55 (m, 6H), 2.47 (s, 3H), 1.55-1.36 (m, 12H), 0.91 (t, J = 6.8 Hz, 9H).

13C{¹H}-NMR (100 MHz, CDCl₃) δ/ppm: 189.6, 174.3, 156.4, 146.1 (d, 3JC-P = 12.2 Hz), 143.3, 141.4, 141.3, 139.3, 134.0 (d, ²JC-P = 9.9 Hz), 133.7, 125.8, 124.6, 123.9, 123.2, 122.6 (d, ²JC-P = 10.3 Hz), 122.3 (d, ³JC-P = 12.2 Hz), 120.4 118.9, 117.4, 82.2 (d, ⁴JC-P = 103.7 Hz), 24.1 (d, ⁵JC-P = 3.8 Hz), 23.8 (d, ⁶JC-P = 15.6 Hz), 21.7 (d, ⁷JC-P = 52.9 Hz), 21.2, 13.5.

31P NMR (162 MHz, CDCl₃) δ/ppm: 21.9.


Following the TP-B, 3i was obtained from 2-((7-methoxy-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1i (99.69 mg, 0.3 mmol), PBu₃ (88.8μL, 1.2 equiv.), benzoyl chloride 2a (104.5 μL, 3.0 equiv.), and Et₃N (150.5 μL, 3.6 equiv.) in anhydrous CH₂Cl₂ (1.5 mL) at 30 °C for 24 h. After workup, the residue was purified by column chromatography (SiO₂, Hexanes:EtOAc = 75:25) to give 3i as a dark red solid (109.0 mg, 70%). Rf = 0.38 (Hexanes:EtOAc =8:2); mp.: 152.9-153.8 °C.

1H NMR (400 MHz, CDCl₃) δ/ppm: 8.13 (d, J = 8.8 Hz, 1H), 7.32 (d, J = 7.1 Hz 1H), 7.26-7.21 (m, 3H), 6.96-6.80 (m, 3H), 3.91 (s, 3H), 2.7-2.55 (m, 6H), 1.53-1.37 (m, 12H), 0.91 (t, J = 6.7 Hz, 9H).

13C{¹H}-NMR (100 MHz, CDCl₃) δ/ppm: 189.5, 174.1, 163.3, 158.0, 146.1 (d, ³JC-P = 12.1 Hz), 141.3, 139.4, 133.6, 133.4 (d, ⁴JC-P = 9.8 Hz), 127.4, 124.6, 123.2, 122.6 (d,..
Following the TP-B, 3j was obtained from 2-((7-fluoro-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1j (96.08 mg, 0.3 mmol), PBu₃ (88.8µL, 1.2 equiv.), benzoyl chloride 2a (104.5 µL, 3.0 equiv.), and Et₃N (150.5 µL, 3.6 equiv.) in anhydrous CH₂Cl₂ (1.5 mL) at 30 °C for 24 h. After workup, the residue was purified by column chromatography (SiO₂, Hexanes:EtOAc = 70:30) to give 3j as a dark red solid (132.1 mg, 87%). R₉ = 0.50 (Hexanes:EtOAc = 8:2); mp.: 150.9-151.8 °C.

1H NMR (400 MHz, CDCl₃) δ/ppm: 8.23 (d, J = 8.9, 6.6 Hz, 1H), 7.33 (d, J = 7.3 Hz 1H), 7.30-7.20 (m, 2H), 6.96 (dd, J = 9.6, 2.4 Hz, 1H), 7.99 (td, J = 8.2, 2.4 Hz, 1H), 6.92 (td, J = 7.1, 1.6 Hz, 1H), 2.76-2.53 (m, 6H), 1.58-1.36 (m, 12H), 0.92 (t, J = 6.8 Hz, 9H).

13C{¹H}-NMR (100 MHz, CDCl₃) δ/ppm: 189.5, 173.4, 166.3, 163.8, 157.3 (d, J_C-F = 13.2 Hz), 146.1 (d, J_C-P = 11.8 Hz) 141.1, 139.2, 134.2 (d, J_C-P = 9.7 Hz), 133.8, 128.3 (d, J_C-F = 10.6 Hz), 124.8, 123.3, 122.5 (d, J_C-P = 10.8 Hz 121.7 (d, J_C-P = 12.2 Hz) 119.6, 119.0, 110.9 (d, J_C-P = 22.2 Hz), 103.9 (d, J_C-P = 25.2 Hz), 82.5 (d, J_C-P = 103.2 Hz), 24.1 (d, J_C-P = 3.8 Hz), 23.8 (d, J_C-P = 15.7 Hz), 21.4 (d, J_C-P = 52.6 Hz), 13.5.

31P NMR (162 MHz, CDCl₃) δ/ppm: 22.3.

19F NMR (376 MHz, CDCl₃) δ/ppm: -106.4.

HRMS (EI) m/z: [M]^+ caleed for C₃₁H₃₄O₂PF: 504.2230 found: 504.2215.
Following the TP-B, 3k was obtained from 2-((8-nitro-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1k (104.18 mg, 0.3 mmol), PBu₃ (88.8µL, 1.2 equiv.), benzoxy chloride 2a (104.5 µL, 3.0 equiv.) and Et₃N (150.5 µL, 3.6 equiv.) in anhydrous CH₂Cl₂ (1.5 mL) at 30 °C for 3 h. After workup, the residue was purified by column chromatography (SiO₂, Hexanes:EtOAc= 70:30) to give 3k as a dark red solid (138.0 mg, 86%). R_f = 0.33 (Hexanes:EtOAc = 8:2); mp:179.7-180.8 °C.

³¹P NMR (162 MHz, CDCl₃) δ/ppm: 22.6.

HRMS (EI) m/z: [M]+ calcd for C₃₁H₅₄N₂O₅P: 531.2175 found: 531.2159.


Following the TP-B, 3l was obtained from 2-((6,8-dimethyl-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1l (330.3 mg, 0.3 mmol), PBu₃ (88.8µL, 1.2 equiv.), benzoxy chloride 2a (104.5 µL, 3.0 equiv.) and Et₃N (150.5 µL, 3.6 equiv.) in anhydrous CH₂Cl₂ (1.5 mL) at 30 °C for 24 h. After workup, the residue was purified by column chromatography (SiO₂, Hexanes:EtOAc= 70:30) to give 3l as a dark red solid (116.0 mg, 75%). R_f = 0.50 (Hexanes:EtOAc = 8:2); mp: 211.1-212.3 °C.

¹³C{¹H}-NMR (100 MHz, CDCl₃) δ/ppm: 190.6, 171.7, 148.8, 144.5 (d, ³JC-P = 11.8 Hz), 140.6, 138.9, 138.5, 135.6 (d, ²JC-P = 9.6 Hz), 134.3, 132.1, 128.4, 125.3, 125.2, 123.4, 123.1 (d, ³JC-P = 10.5 Hz), 121.3, 120.8 (d, ²JC-P = 12.4 Hz), 119.6, 81.1 (d, ¹JC-P = 102.6 Hz), 24.1 (d, ³JC-P = 3.9 Hz), 23.5 (d, ²JC-P = 15.8 Hz), 21.2 (d, ¹JC-P = 52.6 Hz), 13.5.

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**3¹P NMR** (162 MHz, CDCl₃) δ/ppm: 21.9.


**6,8-dibromo-11-(tributyl-λ₅-phosphanylidene)-10H-benzo[5,6]pentaleno[1,2-b]chromene-10,12(11H)-dione 3m.**

Following the TP-B, 3m was obtained from 2-((6,8-dibromo-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1m (138.02 mg, 0.3 mmol), PBu₃ (88.8 µL, 1.2 equiv.), benzoyl chloride 2a (104.5 µL, 3.0 equiv.), and Et₃N (150.5 µL, 3.6 equiv.) in anhydrous CH₂Cl₂ (1.5 mL) at 30 °C for 8 h. After workup, the residue was purified by column chromatography (SiO₂, Hexane:EtOAc = 75:25) to give 3m as a dark red solid (165.0 mg, 85%). Rₚ = 0.40 (Hexanes:EtOAc = 9:1); mp.: 214.2-215.5 °C.

**¹H NMR** (400 MHz, CDCl₃) δ/ppm: 8.30 (d, J = 8.3 Hz, 1H), 7.91 (d, J = 2.3 Hz, 1H), 7.35 (d, J = 7.4 Hz, 1H), 7.32-7.27 (m, 2H), 6.99-6.90 (m, 1H), 2.73-2.49 (m, 6H), 1.58-1.35 (m, 12H), 0.91 (t, J = 6.7 Hz, 9H).

**¹³C{¹H}-NMR** (100 MHz, CDCl₃) δ/ppm: 189.7, 171.8, 151.6, 145.3 (d, J_C-P = 11.8 Hz), 140.9, 140.8, 139.0, 137.5, 135.4 (d, J_C-P = 9.6 Hz), 134.1, 128.3, 125.1, 125.0, 123.5, 122.8 (d, J_C-P = 10.5 Hz), 120.8 (d, J_C-P = 12.4 Hz), 119.4, 115.1, 112.0, 82.8 (d, J_C-P = 102.8 Hz), 24.1 (d, J_C-P = 4.1 Hz), 23.8 (d, J_C-P = 15.8 Hz), 21.3 (d, J_C-P = 52.8 Hz), 13.5.

**3¹P NMR** (162 MHz, CDCl₃) δ/ppm: 22.4.

**HRMS** (EI) m/z: [M]+ calcd for C₃₁H₃₉⁷⁹Br₇⁹BrO₃P: 642.0534 found: 642.0519.

**HRMS** (EI) m/z: [M]+ calcd for C₃₁H₃₃⁷⁹Br₈¹BrO₄P: 644.0514 found: 644.0522.

**HRMS** (EI) m/z: [M]+ calcd for C₃₁H₃₃⁸¹Br₈¹BrO₄P: 646.0493 found: 646.0487.

**13-(tributyl-λ₅-phosphanylidene)-12H-benzo[f]benzo[5,6]pentaleno[1,2-b]chromene-12,14(13H)-dione 3n.**

Following the TP-B, 3n was obtained from 2-((1-oxo-1H-benzo[f]chromen-2-yl)methylene)-1H-indene-1,3(2H)-dione 1n (105.7 mg, 0.3 mmol), PBu₃ (88.8 µL, 1.2 equiv.), benzoyl chloride 2a (104.5 µL, 3.0 equiv.), and Et₃N (150.5 µL, 3.6 equiv.) in
anhydrous CH₂Cl₂ (1.5 mL) at 30 °C for 48 h. After workup, the residue was purified by column chromatography (SiO₂, Hexanes:EtOAc = 70:30) to give 3o as a dark red solid (85.1 mg, 52%). Rₚ = 0.53 (Hexanes:EtOAc = 7:3); mp.: 189.4-190.3 °C.

**1H NMR** (400 MHz, CDCl₃) δ/ppm: 10.17 (d, J = 8.7 Hz, 1H), 7.98 (d, J = 9.1 Hz 1H), 7.87 (d, J = 8.0 Hz, 1H), 7.69 (t, J = 7.9 Hz, 1H), 7.57 (d, J = 9.1 Hz, 1H), 7.52 (t, J = 7.5 Hz, 1H), 7.32 (d, J = 7.3 Hz, 1H), 7.29-7.20 (m, 2H), 6.95-6.85 (m, 1H), 2.79-2.59 (m, 6H), 1.55-1.40 (m, 12H), 0.92 (t, J = 6.7 Hz, 9H).

**13C{1H}-NMR** (100 MHz, CDCl₃) δ/ppm: 189.6, 177.2, 157.3, 144.7 (d, 3J_C-P = 12.0 Hz), 141.3, 141.2, 139.4, 133.8, 133.7, 133.4 (d, 2J_C-P = 10.0 Hz), 132.2, 130.2, 128.1, 126.9, 125.2 (d, 2J_C-P = 11.4 Hz), 124.9, 124.6, 123.2, 122.7, (d, 3J_C-P = 10.6 Hz), 118.7, 118.6, 114.7, 81.2 (d, 1J_C-P = 104.5 Hz), 24.3 (d, 3J_C-P = 3.9 Hz), 23.8 (d, 2J_C-P = 15.7 Hz), 21.8 (d, 1J_C-P = 53.7 Hz), 13.6.

**31P NMR** (162 MHz, CDCl₃) δ/ppm: 21.9.

**HRMS (EI) m/z: [M]+ calcd for C₃₅H₂₇O₅P: 536.2480 found: 536.2464.


Following the **TP-B**, 3o was obtained from 2-(4-oxo-4H-benzo[h]chromen-3-yl)methylene-1H-indene-1,3(2H)-dione 1o (105.72 mg, 0.3 mmol), PBu₃ (88.8μL, 1.2 equiv.), benzyol chloride 2a (104.5 μL, 3.0 equiv.), and Et₃N (150.5 μL, 3.6 equiv.) in anhydrous CH₂Cl₂ (1.5 mL) at 30 °C for 24 h. After workup, the residue was purified by column chromatography (SiO₂, Hexanes:EtOAc =75:25) to give 3o as a dark red solid (118.0 mg, 73%). Rₚ = 0.50 (Hexanes:EtOAc = 8:2); mp.: 191.3-192.1 °C.

**1H NMR** (400 MHz, CDCl₃) δ/ppm: 8.69-8.61 (m, 1H), 8.23 (d, J = 8.7 Hz, 1H), 7.94-7.84 (m, 1H), 7.73-7.60 (m, 3H), 7.42-7.29 (m, 3H), 6.94 (td, J = 7.4, 1.0 Hz, 1H), 2.79-2.56 (m, 6H), 1.62-1.37 (m, 12H), 0.91 (t, J = 6.9 Hz, 9H).

**13C{1H}-NMR** (100 MHz, CDCl₃) δ/ppm: 189.7, 174.4, 153.1, 145.9 (d, 3J_C-P = 12.2 Hz), 141.3, 139.6, 135.6, 133.9 (d, 2J_C-P = 9.9 Hz), 133.8, 128.3, 127.8, 126.3, 124.8, 124.4, 123.3, 123.1 (d, 2J_C-P = 10.3 Hz), 122.9 (d, 3J_C-P = 12.3 Hz), 122.7, 122.4, 121.9, 118.9, 117.9, 81.7 (d, 1J_C-P = 104.0 Hz), 24.2 (d, 3J_C-P = 3.9 Hz), 23.8 (d, 3J_C-P = 15.7 Hz), 21.6 (d, 3J_C-P = 53.3 Hz), 13.5.

**31P NMR** (162 MHz, CDCl₃) δ/ppm: 22.1.

**HRMS (EI) m/z: [M]+ calcd for C₃₅H₂₇O₅P: 536.2480 found: 536.2489.

Following the TP-B, 3p was obtained from 2-((5-hydroxy-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1p (95.40 mg, 0.3 mmol), PBu3 (88.8 µL, 1.2 equiv.), benzoyl chloride 2a (156.83 µL, 4.5 equiv.) and Et3N (192.30 µL, 4.6 equiv.) in anhydrous CH2Cl2 (1.5 mL) at 30 °C for 24 h. After workup, the residue was purified by column chromatography (SiO2, Hexanes:EtOAc = 80:20) to give 3p as a dark red solid (128.0 mg, 70%). Rf = 0.40 (Hexanes:EtOAc = 9:1); mp.: 190.8-191.5 °C.

1H NMR (400 MHz, CDCl3) δ/ppm: 8.26 (d, J = 7.3 Hz, 2H), 7.63 (t, J = 7.4 Hz, 1H), 7.57-7.47 (m, 3H), 7.39 (d, J = 8.5 Hz, 1H), 7.30 (d, J = 7.3 Hz, 1H), 7.27-7.28 (m, 2H), 6.98 (d, J = 7.6 Hz, 1H), 7.39 (dd, J = 7.1, 1.6 Hz, 1H), 2.51-2.34 (m, 6H), 1.58-1.28 (m, 12H), 0.86 (t, J = 6.8 Hz, 9H).

13C{1H}-NMR (100 MHz, CDCl3) δ/ppm: 189.5, 173.4, 165.4, 157.7, 150.2, 144.9 (d, 3JCP = 11.8 Hz), 141.3, 141.2, 139.3, 133.8 (d, 3JCP = 11.8 Hz), 133.7, 132.8, 131.6, 130.8, 130.3, 128.2, 124.6, 123.3 (d, 3JCP = 12.3 Hz), 123.2, 122.3 (d, 3JCP = 12.2 Hz), 118.9, 116.9, 115.9, 82.4 (d, 3JCP = 103.5 Hz), 24.1 (d, 3JCP = 3.9 Hz), 23.8 (d, 3JCP = 15.9 Hz), 21.5 (d, 3JCP = 53.0 Hz), 13.5.

31P NMR (162 MHz, CDCl3) δ/ppm: 21.6.


Following the TP-B, 3q was obtained from 5,6-dimethoxy-2-((4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1q (108.7 mg, 0.3 mmol), PBu3 (88.8 µL, 1.2 equiv.), benzoyl chloride 2a (104.5 µL, 3.0 equiv.) and Et3N (150.5 µL, 3.6 equiv.) in anhydrous CH2Cl2 (1.5 mL) at 30 °C for 3 h. After workup, the residue was purified by column chromatography (SiO2, Hexanes:EtOAc =72:25) to give 3q as a reddish brown solid (90.0 mg, 55%). Rf = 0.38 (Hexanes:EtOAc = 8:2); mp.: 160.7-161.7 °C.

1H NMR (400 MHz, CDCl3) δ/ppm: 8.23 (dd, J = 7.9, 1.6 Hz, 1H), 7.56 (dt, J = 8.9,
7.5, 1.7 Hz, 1H), 7.27 (dd, J = 7.5, 0.7 Hz, 1H), 6.98 (s, 1H), 6.83 (s, 1H), 4.03 (s, 3H), 3.86 (s, 3H), 2.71-2.55 (m, 6H), 1.57-1.38 (m, 12H), 0.92 (t, J = 6.7 Hz, 9H).

$^{13}$C$^{1}$H]-NMR (100 MHz, CDCl$_3$) δ/ppm: 189.2, 173.6, 156.2, 153.9, 146.4, 145.6 (d, $^2$J$_{C-P}$ = 11.9 Hz), 136.7, 135.3 (d, $^3$J$_{C-P}$ = 9.6 Hz), 132.0, 131.2, 126.1, 122.8, 122.5, 121.4 (d, $^2$J$_{C-P}$ = 12.1 Hz), 120.5 (d, $^3$J$_{C-P}$ = 10.7 Hz), 117.3, 107.9, 103.4, 82.9 (d, $^1$J$_{C-P}$ = 103.2 Hz), 56.3, 56.2, 24.2 (d, $^3$J$_{C-P}$ = 3.8 Hz), 23.8 (d, $^2$J$_{C-P}$ = 15.7 Hz), 21.4 (d, $^1$J$_{C-P}$ = 52.8 Hz), 13.5.

$^{31}$P NMR (162 MHz, CDCl$_3$) δ/ppm: 22.0.

HRMS (EI) m/z: [M]$^+$ calcd for C$_{33}$H$_{39}$O$_3$P: 546.2535. found: 546.2538.

3-(4-benzoyl-2,5-diphenylfuran-3-yl)-4H-chromen-4-one 6.

Following the TP-B, 6 was obtained 2-((4-oxo-4H-chromen-3-yl)methylene)-1,3-diphenylpropane-1,3-dione 1r (114.1 mg, 0.3 mmol), PBu$_3$ (88.8 µL, 1.2 equiv.), benzoyl chloride 2a (104.5 µL, 3.0 equiv.) and Et$_3$N (150.5 µL, 3.6 equiv.) in anhydrous CH$_2$Cl$_2$ (1.5 mL) at 30 °C for 3 h. After workup, the residue was purified by column chromatography (SiO$_2$, Hexanes:EtOAc = 7:30) to give 6 as a half-white solid (99.0 mg, 75%). R$_f$ = 0.38 (Hexanes:EtOAc = 8:2); mp.: 115.8-116.8 °C.

$^1$H NMR (400 MHz, CDCl$_3$) δ/ppm: 8.16 (dd, J = 7.9, 1.6 Hz, 1H), 8.02 (s, 1H), 7.86 (dd, J = 8.3, 1.4 Hz, 1H), 7.73-7.62 (m, 3H), 7.55-7.48 (m, 2H), 7.44 (d, J = 8.4, Hz, 1H), 7.40-7.28 (m, 5H), 7.27-7.18 (m, 5H).

$^{13}$C$^{1}$H]-NMR (100 MHz, CDCl$_3$) δ/ppm: 192.4, 175.6, 156.2, 154.9, 152.6, 150.5, 137.2, 133.6, 132.9, 129.8, 129.7, 129.2, 128.6, 128.6, 128.2, 128.1, 127.1, 126.1, 126.1, 125.1, 123.8, 123.8, 118.0, 117.4, 113.8.

HRMS (EI) m/z: [M]$^+$ calcd for C$_{33}$H$_{39}$O$_3$P: 468.1362. found: 468.1352.

2-(2-oxo-1-(4-oxo-4H-chromen-3-yl)-2-phenylethylidene)-1H-indene-1,3(2H)-dione 5aa.

Following the TP-C, 5aa was obtained from 2-((4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1a (90.75 mg, 0.3 mmol), Me$_2$PhP (8.54 µL, 0.2 equiv.),
benzoyl chloride 2a (41.82 µL, 1.2 equiv.), and Et3N (54.36 µL, 1.3 equiv.) in anhydrous THF (1.5 mL) at 30 °C for 0.5 h. After completion of the reaction, the residue was purified by column chromatography (SiO2, Hexanes:EtOAc= 70:30) to give 5aa as a pale yellow solid (110.0 mg, 90%). Rf = 0.41 (Hexanes:EtOAc = 7:3); mp.: 248.2-248.5 °C.

1H NMR (400 MHz, CDCl3) δ/ppm: 8.30 (s, 1H), 8.29 (dd, J = 7.9, 1.5 Hz 1H), 8.11 (dd, J = 7.3, 1.2, Hz 1H), 7.96-7.92 (m, 1H), 7.89-7.84 (m, 1H), 7.84-7.74 (m, 2H), 7.70 (dtd, J = 9.0 7.3, 1.6, Hz, 1H), 7.57 (t, J = 7.4 Hz 1H), 7.50-7.41 (m, 4H).

13C{1H}-NMR (100 MHz, CDCl3) δ/ppm: 195.4, 187.6, 187.3, 173.0, 155.9, 155.4, 146.2, 142.6, 140.7, 135.8, 132.7, 134.9, 134.3, 134.2, 131.6, 129.0, 128.9, 126.6, 126.0, 123.7, 123.6, 123.5, 118.4, 118.2.

HRMS (ESI) m/z: [M+H]+ calced for C26H18O5: 407.0919 found: 407.0916.

2-(1-(6-methyl-4-oxo-4H-chromen-3-yl)-2-oxo-2-phenylethylidene)-1H-indene-1,3(2H)-dione 5ba.

Following the TP-C, 5ba was obtained from 2-((6-methyl-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1b (94.89 mg, 0.3 mmol), Me2PhP (8.54µL, 0.2 equiv.), benzoyl chloride 2a (41.82 µL, 1.2 equiv.), and Et3N (54.36 µL, 1.3 equiv.) in anhydrous THF (1.5 mL) at 30 °C for 3 h. After completion of the reaction, the residue was purified by column chromatography (SiO2, Hexanes:EtOAc =70:30) to give 5ba as a pale yellow solid (101.0 mg, 80%). Rf = 0.45 (Hexanes:EtOAc = 7:3); mp.: 290.3-291.2 °C.

1H NMR (400 MHz, CDCl3) δ/ppm: 8.27 (s, 1H), 8.14-8.07 (m, 3H), 7.95 (dd, J = 6.1, 1.5 Hz, 1H), 7.90-7.85 (m, 1H), 7.83-7.75 (m, 2H), 7.56 (t, J = 7.5 Hz, 1H), 7.52 (dd, J = 8.6, 2.1 Hz, 1H), 7.46 (t, J = 7.8 Hz, 2H), 7.38 (d, J = 8.6 Hz, 1H), 2.50 (s, 3H).

13C{1H}-NMR (100 MHz, CDCl3) δ/ppm: 195.4, 187.8, 187.4, 173.1, 155.3, 154.2, 146.5, 142.7, 140.8, 136.2, 135.8, 135.6, 135.5, 135.1, 134.2, 131.5, 129.1, 128.9, 126.0, 123.7, 123.6, 123.4, 118.2, 117.9, 20.9.

HRMS (ESI) m/z: [M+H]+ calced for C27H16O5: 420.0998 found: 420.0989.

2-(1-(6-chloro-4-oxo-4H-chromen-3-yl)-2-oxo-2-phenylethylidene)-1H-indene-1,3(2H)-dione 5ea.
Following the TP-C, 5ea was obtained from 2-((6-chloro-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1e (101.02 mg, 0.3 mmol), Me2PhP (8.54µL, 0.2 equiv.), benzoyl chloride 2a (41.82 µL, 1.2 equiv.), and Et3N (54.36 µL, 1.3 equiv.) in anhydrous THF (1.5 mL) at 30 °C for 0.5 h. After completion of the reaction, the residue was purified by column chromatography (SiO2, Hexanes:EtOAc= 70:30) to give 5ea as a pale yellow solid (95.0 mg, 72%). Rf = 0.45 (Hexanes:EtOAc = 7:3); mp: 270.1-271.2 °C.

1H NMR (400 MHz, CDCl3) δ/ppm: 8.29 (s, 1H), 8.27 (d, J = 2.6 Hz, 1H), 8.12-8.07 (m, 2H), 7.99-7.95 (m, 1H), 7.91-7.87 (m, 1H), 7.85-7.77 (m, 2H), 7.70 (dd, J = 9.4, 2.7 Hz, 1H), 7.62-7.56 (m, 1H), 7.51-7.43 (m, 3H).

13C{1H}-NMR (100 MHz, CDCl3) δ/ppm: 195.3, 187.6, 187.3, 171.9, 155.2, 154.3, 145.4, 142.6, 140.8, 135.9, 135.7, 134.9, 134.5, 134.4, 132.1, 131.8, 129.1, 128.9, 126.1, 124.6, 123.7, 123.6, 119.9, 118.4.

HRMS (EI) m/z: [M]+ calcld for C26H1335ClO5: 440.0452 found: 440.0434.

HRMS (EI) m/z: [M]+ calcld for C26H1337ClO5: 442.0422 found: 442.0436.

2-((7-methoxy-4-oxo-4H-chromen-3-yl)-2-oxo-2-phenylethylidene)-1H-indene-1,3(2H)-dione 5ia.

Following the TP-C, 5ia was obtained from 2-((7-methoxy-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1i (99.69 mg, 0.3 mmol), Me2PhP (8.54µL, 0.2 equiv.), benzoyl chloride 2a (41.82 µL, 1.2 equiv.), and Et3N (54.36 µL, 1.3 equiv.) in anhydrous THF (1.5 mL) at 30 °C for 2 h. After completion of the reaction, the residue was purified by column chromatography (SiO2, Hexanes:EtOAc= 70:30) to give 5ia as a pale yellow solid (82.0 mg, 62%). Rf = 0.40 (Hexanes:EtOAc = 7:3); mp: 268.8-269.3 °C.

1H NMR (400 MHz, CDCl3) δ/ppm: 8.23 (s, 1H), 8.19 (d, J = 8.9 Hz, 1H), 8.14-8.07 (m, 2H), 7.99-7.95 (m, 1H), 7.90-7.86 (m, 1H), 7.85-7.74 (m, 2H), 7.62-7.54 (m, 1H), 7.50-7.43 (m, 2H), 7.02 (dd, J = 9.1, 2.3 Hz, 1H), 6.87 (d, J = 2.4 Hz, 1H), 3.91 (s, 3H).

13C{1H}-NMR (100 MHz, CDCl3) δ/ppm: 195.5, 187.8, 187.4, 172.3, 164.6, 157.7, 155.1, 146.6, 142.7, 140.8, 135.8, 135.6, 135.1, 134.1, 131.6, 129.1, 128.9, 128.1, 123.7,
123.6, 118.5, 117.6, 115.2, 100.5, 55.9.

**HRMS (EI) m/z:** [M]+ calcd for C_{27}H_{16}O_{6}: 436.0947 found: 436.0966.

**2-(1-(7-fluoro-4-oxo-4H-chromen-3-yl)-2-oxo-2-phenylethylidene)-1H-indene-1,3(2H)-dione 5ja.**

Following the **TP-C, 5ja** was obtained from 2-((7-fluoro-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1j (96.08 mg, 0.3 mmol), Me_{2}PhP (8.54 µL, 0.2 equiv.), benzoyl chloride 2a (41.82 µL, 1.2 equiv.), and Et_{3}N (54.36 µL, 1.3 equiv.) in anhydrous THF (1.5 mL) at 30 °C for 0.5 h. After completion of the reaction, the residue was purified by column chromatography (SiO_{2}, Hexanes:EtOAc = 70:30) to give 5ja as a pale yellow solid (95.4 mg, 75%). R_f = 0.43 (Hexanes:EtOAc = 7:3); mp.: 223.5-224.5 °C.

**^{1}H NMR (400 MHz, CDCl_{3}) δ/ppm:** 8.3 (dd, J = 8.7, 6.3 Hz, 1H), 8.29 (s, 1H), 8.11 (d, J = 7.8 Hz, 2H), 7.96 (dd, J = 6.4, 1.3 Hz, 1H), 7.93-7.85 (m, 1H), 7.85-7.75 (m, 2H), 7.58 (t, J = 7.5 Hz, 1H), 7.47 (t, J = 7.7 Hz, 2H), 7.24-7.15 (m, 2H).

**^{13}C{^{1}H}-NMR (100 MHz, CDCl_{3}) δ/ppm:** 195.3, 187.7, 187.3, 172.2, 167.2, 164.6, 157.9 (d, J_{C, F} = 13.3 Hz), 155.2, 145.6, 142.6, 140.8, 135.9, 135.8, 134.9, 134.3, 131.8, 129.1 (d, J_{C, F} = 10.9 Hz), 129.1, 128.9, 123.8, 123.7, 120.7, 120.6, 118.7, 114.8 (d, J_{C, F} = 22.8 Hz), 105.0 (d, J_{C, F} = 25.6 Hz).

**^{19}F NMR (376 MHz, CDCl_{3}) δ/ppm:** -101.3.

**HRMS (EI) m/z:** [M]+ calcd for C_{28}H_{13}O_{5}F: 424.0747 found: 424.0745.

**2-(1-(6,8-dimethyl-4-oxo-4H-chromen-3-yl)-2-oxo-2-phenylethylidene)-1H-indene-1,3(2H)-dione 5la.**

Following the **TP-C, 5la** was obtained from 2-((6,8-dimethyl-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1l (99.10 mg, 0.3 mmol), Me_{2}PhP (8.54 µL, 0.2 equiv.), benzoyl chloride 2a (41.82 µL, 1.2 equiv.), and Et_{3}N (54.36 µL, 1.3 equiv.) in anhydrous THF (1.5 mL) at 30 °C for 0.5 h. After completion of the reaction, the residue was purified by column chromatography (SiO_{2}, Hexanes:EtOAc = 70:30) to give 5la as a pale yellow solid (105.1 mg, 80%). R_f = 0.53 (Hexanes:EtOAc = 7:3); mp.: 228.
304.1-305.2 °C.

$^1$H NMR (400 MHz, CDCl$_3$) δ/ppm: 8.32 (s, 1H), 8.10 (dd, $J = 7.3, 1.4$ Hz, 1H), 7.99-7.93 (m, 1H), 7.92 (d, $J = 0.8$ Hz, 1H), 7.89-7.85 (m, 1H), 7.83-7.75 (m, 2H), 7.60-7.53 (m, 1H), 7.50-7.42 (m, 2H), 7.36 (s, 1H).

$^{13}$C$^1$H-NMR (100 MHz, CDCl$_3$) δ/ ppm: 195.4, 187.9, 187.4, 173.4, 155.2, 152.8, 146.8, 142.7, 140.8, 136.5, 135.8, 135.6, 135.5, 135.1, 134.1, 129.1, 128.9, 127.4, 123.7, 123.6, 123.5, 123.4, 117.9, 20.9, 15.3.

HRMS (EI) m/z: [M]$^+$ calcd for C$_{28}$H$_{18}$O$_5$: 434.1154 found: 434.1163.

2-(6,8-dibromo-4-oxo-4H-chromen-3-yl)-2-oxo-2-phenylethylidene)-1H-indene-1,3(2H)-dione 5ma.

Following the TP-C, 5ma was obtained from 2-((6,8-dibromo-4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1m (138.04 mg, 0.3 mmol), Me$_2$PhP (8.54µL, 0.2 equiv.), benzoyl chloride 2a (41.82 µL, 1.2 equiv.), and Et$_3$N (54.36 µL, 1.3 equiv.) in anhydrous THF (1.5 mL) at 30 °C for 0.5 h. After completion of the reaction, the residue was purified by column chromatography (SiO$_2$, Hexanes:EtOAc = 70:30) to give 5ma as a pale yellow solid (85.0 mg, 50%). R$_f$ = 0.30 (Hexanes:EtOAc = 7:3); mp.: 269.1-270.5 °C.

$^1$H NMR (400 MHz, CDCl$_3$) δ/ppm: 8.41-8.34 (m, 2H), 8.12-8.04 (m, 3H), 7.98-7.94 (m, 1H), 7.92-7.87 (m, 1Hz), 7.86-7.77 (m, 2H), 7.59 (t, $J = 7.4$ Hz, 1H), 7.47 (t, $J = 7.8$ Hz, 2H).

$^{13}$C$^1$H-NMR (100 MHz, CDCl$_3$) δ/ ppm: 194.9, 187.4, 187.3, 171.4, 154.8, 151.6, 144.6, 142.6, 140.9, 140.0, 136.0, 135.9, 134.8, 134.4, 132.1, 129.1, 129.0, 128.7, 125.7, 123.8, 119.4, 118.6, 113.1.

HRMS (EI) m/z: [M]$^+$ calcd for C$_{26}$H$_{12}^{79}$Br$_{79}$BrO$_5$: 561.9051 found: 561.9042.

HRMS (EI) m/z: [M]$^+$ calcd for C$_{26}$H$_{12}^{79}$Br$_{81}$BrO$_5$: 563.9031 found: 563.9021.

HRMS (EI) m/z: [M]$^+$ calcd for C$_{26}$H$_{12}^{81}$Br$_{81}$BrO$_5$: 565.9011 found: 565.9024.

2-(2-oxo-1(1-oxo-1H-benzo[f]chromen-2-yl)-2-phenylethylidene)-1H-indene-1,3(2H)-dione 5na.
Following the TP-C, 5na was obtained from 2-((1-oxo-1H-benzo[f]chromen-2-yl)methylene)-1H-indene-1,3(2H)-dione 1n (105.70 mg, 0.3 mmol), Me2PhP (8.54µL, 0.2 equiv.), benzoyl chloride 2a (41.82 µL, 1.2 equiv.), and Et3N (54.36 µL, 1.3 equiv.) in anhydrous THF (1.5 mL) at 30 °C for 2 h. After completion of the reaction, the residue was purified by column chromatography (SiO2, Hexanes:EtOAc = 65:35) to give 5na as a pale yellow solid (97.0 mg, 74%). Rf = 0.45 (Hexanes:EtOAc = 7:3); mp.: 264.5-265.6 °C.

1H NMR (400 MHz, CDCl3) δ/ppm: 9.94 (d, J = 8.7 Hz, 1H), 8.32 (s, 1H), 8.23-8.20 (m, 2H), 8.12 (d, J = 9.1 Hz, 1H), 7.97-7.94 (m, 1H), 7.92 (d, J = 7.4 Hz, 1H), 7.90-7.86 (m, 1H), 7.84-7.72 (m, 3H), 7.63 (dd, J = 8.78, 7.2, 1.2 Hz, 1H), 7.60-7.55 (m, 1H), 7.53-7.44 (m, 3H).

13C{1H}-NMR (100 MHz, CDCl3) δ/ppm: 195.5, 187.9, 187.3, 174.7, 157.2, 152.9, 146.8, 142.7, 140.8, 136.1, 135.8, 135.6, 135.2, 134.2, 134.3, 131.6, 131.0, 130.6, 129.6, 129.2, 128.9, 128.3, 127.2, 126.9, 123.7, 123.6, 120.9, 117.4, 117.3.


2-(2-oxo-1-(4-oxo-4H-benzo[h]chromen-3-yl)-2-phenylethylidene)-1H-indene-1,3(2H)-dione 5oa.

Following the TP-C, 5oa was obtained from 2-((1-oxo-1H-benzo[f]chromen-2-yl)methylene)-1H-indene-1,3(2H)-dione 1o (105.70 mg, 0.3 mmol), Me2PhP (8.54µL, 0.2 equiv.), benzoyl chloride 2a (41.82 µL, 1.2 equiv.), and Et3N (54.36 µL, 1.3 equiv.) in anhydrous THF (1.5 mL) at 30 °C for 0.5 h. After completion of the reaction, the residue was purified by column chromatography (SiO2, Hexanes:EtOAc = 75:25) to give 5oa as a pale yellow solid (98.0 mg, 75%). Rf = 0.40 (Hexanes:EtOAc = 7:3); mp.: 180.2-181.5 °C.

1H NMR (400 MHz, CDCl3) δ/ppm: 8.50 (s, 1H), 8.45 (d, J = 8.2 Hz, 1H), 8.21 (d, J = 8.7 Hz, 1H), 8.18-8.12 (m, 2H), 8.0-7.95 (m, 1H), 7.93 (d, J = 7.7 Hz, 1H), 7.91-7.87 (m, 1H), 7.85-7.76 (m, 3H), 7.75-7.64 (m, 2H), 7.80 (t, J = 7.4 Hz, 1H), 7.48 (t, J = 7.8 Hz, 2H).

13C{1H}-NMR (100 MHz, CDCl3) δ/ppm: 195.5, 187.8, 187.3, 172.8, 154.4, 153.5, 146.2, 142.7, 140.8, 136.0, 135.8, 135.7, 135.0, 134.2, 131.8, 129.7, 129.1, 128.9, 128.1, 127.1, 127.5, 126.1, 123.8, 123.7, 123.7, 122.2, 121.1, 120.2, 119.7.

2-(2-oxo-1-(4-oxo-4H-chromen-3-yl)-2-(p-tolyl)ethylidene)-1H-indene-1,3(2H)-dione 5ab.

Following the TP-C, 5ab was obtained from 2-((4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1a (90.75 mg, 0.3 mmol), Me₂PhP (8.54 µL, 1.2 equiv.), 4-methylbenzoyl chloride 2b (52.35 µL, 1.2 equiv.), and Et₃N (54.36 µL, 1.3 equiv.) in anhydrous THF (1.5 mL) at 30 °C for 0.5 h. After completion of the reaction, the residue was purified by column chromatography (SiO₂, Hexanes:EtOAc = 80:20) to give 5ab as a pale yellow solid (109.0 mg, 86%). Rf = 0.40 (Hexanes:EtOAc = 90:10); mp.: 284.1-285.0 °C.

1H NMR (400 MHz, CDCl₃) δ/ppm: 8.35-8.25 (m, 2H), 8.00 (d, J = 8.2 Hz, 2H), 7.96 (d, J = 6.6 Hz, 1H), 7.91-7.85 (m, 1H), 7.84-7.75 (m, 2H), 7.71 (td, J = 6.7, 1.0 Hz, 1H), 7.54-7.41 (m, 2H), 7.30-7.22 (m, 2H), 2.37 (s, 3H).

13C{1H}-NMR (100 MHz, CDCl₃) δ/ppm: 194.9 187.8, 187.4, 172.9, 155.9, 155.2, 146.4, 145.3, 142.6, 140.8, 135.8, 135.6, 134.2, 132.6, 131.4, 129.7, 129.2, 126.7, 125.9, 123.8, 123.7, 123.6, 118.6, 118.2, 21.8.

HRMS (EI) m/z: [M]+ calcd for C₂₇H₁₆O₅: 420.0998 found: 420.0989.

2-(2-(4-methoxyphenyl)-2-oxo-1-(4-oxo-4H-chromen-3-yl)ethylidene)-1H-indene-1,3(2H)-dione 5ac.

Following the TP-C, 5ac was obtained from 2-((4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1a (90.75 mg, 0.3 mmol), Me₂PhP (8.54 µL, 0.2 equiv.), 4-methoxy benzoyl chloride 2c (52.8 µL, 1.2 equiv.), and Et₃N (54.36 µL, 1.3 equiv.) in anhydrous THF (1.5 mL) at 30 °C for 3 h. After completion of the reaction, the residue was purified by column chromatography (SiO₂, Hexanes:EtOAc = 70:30) to give 5ac as a pale yellow solid (115.0 mg, 88%). Rf = 0.36 (Hexanes:EtOAc = 7:3); mp.: 251.9-252.8 °C.

1H NMR (400 MHz, CDCl₃) δ/ppm: 8.33-8.27 (m, 1H), 8.16-8.05 (m, 2H), 7.99-7.93 (m, 1H), 7.92-7.87 (m, 1H), 7.84-7.77 (m, 2H), 7.71 (dtd, J = 9.4, 7.7, 1.7 Hz, 1H),
Following the TP-C, 5ad was obtained from 2-((4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1a (90.75 mg, 0.3 mmol), Me_2PhP (8.54 µL, 0.2 equiv.), 4-fluorobenzoyl chloride 2d (42.59 µL, 1.2 equiv.), and Et_3N (54.36 µL, 1.3 equiv.) in anhydrous THF (1.5 mL) at 30 °C for 0.5 h. After completion of the reaction, the residue was purified by column chromatography (SiO_2, Hexanes:EtOAc = 70:30) to give 5ad as a pale yellow solid (110.0 mg, 86%). R_f = 0.45 (Hexanes:EtOAc = 7:3); mp: 267.4-268.5 °C.

^1H NMR (400 MHz, CDCl_3) δ/ppm: 8.30 (s, 1H), 8.29 (dd, J = 8.0, 1.6 Hz, 1H), 8.18-8.11 (m, 2H), 7.99-7.94 (m, 1H), 7.86-7.78 (m, 2H), 7.73 (dt, J = 9.5, 6.9, 1.7 Hz, 1H), 7.58 (t, J = 7.5 Hz, 1H), 7.75-7.45 (m, 2H), 7.19-7.10 (m, 2H).

^13C^1H-NMR (100 MHz, CDCl_3) δ/ppm: 193.8, 187.8, 187.2, 173.3, 167.6, 167.6, 165.1, 155.9, 155.5, 145.9, 142.6, 140.7, 135.9 (d, J_C-F = 17.2 Hz), 134.3, 131.8, 131.7, 131.6, 126.6, 126.1, 123.7, 123.6, 118.3, 118.3, 116.2 (d, J_C-F = 23.2 Hz).

^19F NMR (376 MHz, CDCl_3) δ/ppm: -102.8.

HRMS (EI) m/z: [M]^+ calcd for C_{26}H_{13}O_5F: 424.0747 found: 424.0764.

Following the TP-C, 5ae was obtained from 2-((4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1a (90.75 mg, 0.3 mmol), Me_2PhP (8.54 µL, 0.2 equiv.), 4-
chlorobenzoyl chloride 2e (41.82 µL, 1.2 equiv.), and Et3N (54.36 µL, 1.3 equiv.) in anhydrous THF (1.5 mL) at 30 °C for 2 h. After completion of the reaction, the residue was purified by column chromatography (SiO2, Hexanes:EtOAc = 70:30) to give 5ae as a pale yellow solid (121.1 mg, 92%). Rf = 0.53 (Hexanes:EtOAc = 7:3); mp.: 196.4-197.5 °C.

1H NMR (400 MHz, CDCl3) δ/ppm: 8.32 (s, 1H), 8.27 (dd, J = 8.1, 1.5 Hz, 1H), 8.17-8.03 (m, 2H), 7.97-7.95 (m, 1H), 7.91-7.87 (m, 1H), 7.87-7.78 (m, 2H), 7.76-7.69 (m, 1H), 7.49 (t, J = 8.6 Hz, 2H), 7.46-7.41 (m, 2H).

13C{1H}-NMR (100 MHz, CDCl3) δ/ppm: 194.3, 187.8, 187.2, 173.3, 155.9, 155.8, 145.9, 142.6, 140.7, 140.6, 135.9, 135.8, 134.4, 133.6, 131.6, 131.5, 130.3, 129.3, 128.8, 126.6, 126.1, 123.8, 123.7, 123.6, 118.3, 118.2.

HRMS (EI) m/z: [M]+ calcd for C26H1335ClO5: 440.0452 found: 440.0431.

HRMS (EI) m/z: [M]+ calcd for C26H1337ClO5: 442.0422 found: 442.0396.

2-(2-(4-bromophenyl)-2-oxo-1-(4-oxo-4H-chromen-3-yl)ethylidene)-1H-indene-1,3(2H)-dione 5af.

Following the TP-C, 5af was obtained from 2-((4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1a (90.75 mg, 0.3 mmol), Me2PhP (8.54µL, 0.2 equiv.), 4-bromobenzoyl chloride 2f (49.09 mg, 1.2 equiv.), and Et3N (54.36 µL, 1.3 equiv.) in anhydrous THF (1.5 mL) at 30 °C for 0.5 h. After completion of the reaction, the residue was purified by column chromatography (SiO2, Hexanes:EtOAc = 70:30) to give 5af as a pale yellow solid (110.0 mg, 75%). Rf = 0.50 (Hexanes:EtOAc = 65: 35); mp.: 236.5-237.8 °C.

1H NMR (400 MHz, CDCl3) δ/ppm: 8.32 (s, 1H), 8.28 (dd, J = 7.9, 1.3 Hz, 1H), 7.97 (d, J = 8.6 Hz, 3H), 7.92-7.76 (m, 1H), 7.86-7.77 (m, 2H), 7.73 (ddt, J = 8.7, 6.7, 1.5 Hz, 1H), 7.61 (d, J = 8.6 Hz, 2H), 7.53-7.44 (m, 2H).

13C{1H}-NMR (100 MHz, CDCl3) δ/ppm: 194.5, 187.8, 187.2, 173.3, 155.9, 155.9, 145.9, 142.7, 140.7, 135.9, 135.8, 134.4, 134.1, 132.3, 131.7, 130.4, 129.5, 126.6, 126.1, 123.8, 123.6, 123.7, 118.3, 118.2.

HRMS (EI) m/z: [M]+ calcd for C26H1379BrO5: 483.9946 found: 483.9971.

HRMS (EI) m/z: [M]+ calcd for C26H1381BrO5: 485.9926 found: 485.9979.
2-(2-(3-bromophenyl)-2-oxo-1-(4-oxo-4H-chromen-3-yl)ethylidene)-1H-indene-1,3(2H)-dione 5ag.

Following the TP-C, 5ag was obtained from 2-((4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1a (90.75 mg, 0.3 mmol), Me₂PhP (8.54µL, 0.2 equiv.), 3-bromobenzoyl chloride 2g (47.53 µL, 1.2 equiv.), and Et₃N (54.36 µL, 1.3 equiv.) in anhydrous THF (1.5 mL) at 30 °C for 0.5 h. After completion of the reaction, the residue was purified by column chromatography (SiO₂, Hexanes:EtOAc = 70:30) to give 5ag as a pale yellow solid (120.0 mg, 82%). Rᵣ = 0.48 (Hexanes:EtOAc = 7:3); mp.: 250.1-251.3 °C.

¹H NMR (400 MHz, CDCl₃) δ/ppm: 8.32 (s, 1H), 8.31-8.34 (m, 2H), 8.01-7.95 (m, 2H), 7.98-7.86 (m, 1H), 7.85-7.77 (m, 2H), 7.72 (dt, J = 9.2, 7.7, 1.7 Hz, 1H), 7.78-7.66 (m, 1H), 7.50 (d, J = 8.5 Hz, 1H), 7.56-7.44 (m, 1H), 7.32 (t, J = 7.9 Hz, 1H).

¹³C{¹H}-NMR (100 MHz, CDCl₃) δ/ppm: 194.2, 187.8, 187.1, 173.3, 156.2, 155.9, 145.9, 142.7, 140.6, 136.9, 136.8, 135.9, 135.8, 134.4, 131.7, 131.3, 130.4, 127.9, 126.6, 126.1, 123.8, 123.7, 123.2, 118.3, 118.1.

HRMS (EI) m/z: [M]+ calcd for C₂₆H₁₅⁷⁹BrO₅: 483.9946 found: 483.9931.

2-(2-(2-bromophenyl)-2-oxo-1-(4-oxo-4H-chromen-3-yl)ethylidene)-1H-indene-1,3(2H)-dione 5ah.

Following the TP-C, 5ah was obtained from 2-((4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1a (90.75 mg, 0.3 mmol), Me₂PhP (8.54µL, 0.2 equiv.), 2-bromobenzoyl chloride 2h (59.49 µL, 1.2 equiv.), and Et₃N (54.36 µL, 1.3 equiv.) in anhydrous THF (1.5 mL) at 30 °C for 2 h. After completion of the reaction, the residue was purified by column chromatography (SiO₂, Hexanes:EtOAc = 70:30) to give 5ah as a pale yellow solid (113.0 mg, 77%). Rᵣ = 0.38 (Hexanes:EtOAc = 7:3); mp.: 242.2-243.3 °C.

¹H NMR (400 MHz, CDCl₃) δ/ppm: 8.48 (s, 1H), 8.26 (dd, J = 8.0, 1.4 Hz, 1H), 8.08
(dd, J = 7.5, 1.9 Hz, 1H), 7.99-7.92 (m, 1H), 7.91-7.86 (m, 1H), 7.85-7.78 (m, 2H), 7.75-7.70 (m, 1H), 7.53 (d, J = 8.3 Hz, 1H), 7.47 (t, J = 7.9 Hz, 1H), 7.42-7.31 (m, 2H).

$^1$H NMR (100 MHz, CDCl$_3$) δ/ppm: 193.7, 188.0, 187.5, 173.6, 157.2, 155.9, 147.0, 142.5, 140.5, 135.8, 135.7, 135.4, 134.8, 134.4, 133.6, 133.3, 130.8, 127.4, 126.6, 126.1, 123.8, 123.7 122.2, 118.4, 118.3.

HRMS (EI) m/z: [M$^+$] calcd for C$_{26}$H$_{13}$BrO$_5$: 483.9946 found: 483.9935.

HRMS (EI) m/z: [M$^+$] calcd for C$_{26}$H$_{13}$81BrO$_5$: 485.9926 found: 485.9921.

4-(2-(1,3-dioxo-1,3-dihydro-2H-inden-2-ylidene)-2-(4-oxo-4H-chromen-3-yl)acetyl)benzonitrile 5ai.

Following the TP-C, 5ai was obtained from 2-((4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1a (90.75 mg, 0.3 mmol), Me$_2$PhP (8.54µL, 0.2 equiv.), 4-cyanobenzoyl chloride 2i (59.0 mg, 1.2 equiv.), and Et$_3$N (54.36 µL, 1.3 equiv.) in anhydrous THF (1.5 mL) at 30 °C for 2 h. After completion of the reaction, the residue was purified by column chromatography (SiO$_2$, Hexanes:EtOAc = 65:35) to give 5ai as a yellow solid (84.0 mg, 65%). R$_f$ = 0.50 (Hexanes:EtOAc = 5:5); mp.: 265.1-266.1 °C.

$^1$H NMR (400 MHz, CDCl$_3$) δ/ppm: 8.42 (s, 1H), 8.27-8.16 (m, 3H), 7.99 (d, J = 7.1 Hz, 1H), 7.90-7.80 (m, 3H), 7.79-7.71 (m, 3H), 7.54 (d, J = 8.5 Hz, 1H), 7.47 (t, J = 7.6 Hz, 1H).

$^{13}$C{H$^1$}-NMR (100 MHz, CDCl$_3$) δ/ppm: 194.3, 187.9, 187.0, 173.9, 157.4, 155.9, 146.1, 142.6, 140.4, 138.6, 136.2, 136.0, 134.6, 132.6, 131.7, 129.1, 126.5, 126.3, 123.9, 123.7, 118.3, 117.9, 116.7.

HRMS (MALDI) m/z: [M+H$^+$] calcd for C$_{27}$H$_{14}$NO$_5$: 432.0872 found: 432.0867.

2-(2-(naphthalen-1-yl)-2-oxo-1-(4-oxo-4H-chromen-3-yl)ethylidene)-1H-indene-1,3(2H)-dione 5aj.
Following the TP-C, 5aj was obtained from 2-((4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1a (90.75 mg, 0.3 mmol), Me₂PhP (8.54 µL, 0.2 equiv.), 1-naphthoyl chloride 2j (54.24 µL, 1.2 equiv.), and Et₃N (54.36 µL, 1.3 equiv.) in anhydrous THF (1.5 mL) at 30 °C for 2 h. After completion of the reaction, the residue was purified by column chromatography (SiO₂, Hexanes:EtOAc = 75:25) to give 5aj as a pale yellow solid (117.0 mg, 85%). Rf = 0.43 (Hexanes:EtOAc = 7:3); mp.: 190.3-191.5 °C.

1H NMR (400 MHz, CDCl₃) δ/ppm: 9.43 (d, J = 6.8 Hz, 1H), 8.41 (s, 1H), 8.34 (dd, J = 8.5, 1.1 Hz, 1H), 8.26 (d, J = 7.1 Hz, 1H), 8.03 (d, J = 8.2 Hz, 1H), 7.94 (d, J = 7.4 Hz, 1H), 7.89 (d, J = 7.9 Hz, 1H), 7.85-7.67 (m, 5H), 7.60 (t, J = 7.8 Hz, 1H), 7.51-7.41 (m, 3H).

13C{¹H}-NMR (100 MHz, CDCl₃) δ/ppm: 197.1, 188.1, 187.7, 173.1, 156.0, 154.7, 146.9, 142.7, 140.8, 135.7, 135.6, 135.2, 134.2, 134.0, 133.7, 131.0, 130.9, 130.7, 129.2, 128.6, 126.8, 126.7, 126.2, 125.9, 124.7, 123.8, 123.7, 123.6, 119.2, 118.2. HRMS (EI) m/z: [M]⁺ calcd for C₂₇H₁₆O₅: 456.0998 found: 456.0984.

2-(3-methyl-2-oxo-1-(4-oxo-4H-chromen-3-yl)butylidene)-1H-indene-1,3(2H)-dione 5ak.

Following the TP-C, 5ak was obtained from 2-((4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1a (90.75 mg, 0.3 mmol), Me₂PhP (8.54 µL, 0.2 equiv.), isobutyryl chloride 2k (37.7 µL, 1.2 equiv.), and Et₃N (54.36 µL, 1.3 equiv.) in anhydrous THF (1.5 mL) at 30 °C for 0.5 h. After completion of the reaction, the residue was purified by column chromatography (SiO₂, Hexanes:EtOAc =70:30) to give 5ak as a pale yellow solid (62.0 mg, 55%). Rf = 0.48 (Hexanes:EtOAc = 7:3); mp.: 200.1-201.2 °C.

1H NMR (400 MHz, CDCl₃) δ/ppm: 8.30 (dd, J = 8.0, 1.5 Hz, 1H), 8.17 (s, 1H), 7.99-7.95 (m, 1H), 7.93-7.89 (m, 1H), 7.85-7.78 (m, 2H), 7.85-7.75 (m, 2H), 7.73 (td, J = 8.8, 7.4, 1.6 Hz, 1H), 7.53-7.45 (m, 2H), 3.13 (sep, J = 7.01 Hz, 1H), 1.26 (brs, 3H), 1.24 (brs, 3H).

13C{¹H}-NMR (100 MHz, CDCl₃) δ/ppm: 209.8, 188.5, 187.3, 173.1, 155.9, 154.4, 147.8, 142.5, 140.7, 135.9, 135.7, 134.2, 130.4, 126.7, 126.0, 123.7, 123.6, 123.5, 118.6, 118.2, 41.1, 17.6. HRMS (EI) m/z: [M]⁺ calcd for C₂₃H₁₆O₅: 372.0998 found: 372.0981.
1-oxo-2-((4-oxo-4H-chromen-3-yl)(tributylphosphonio)methyl)-1H-inden-3-olate 4

A 50 mL round-bottomed flask equipped with a magnetic stir bar was charged with 2-((4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 1a (90.75 mg, 0.3 mmol), PBu₃ (88.8 µL, 1.2 equiv.) in anhydrous CH₂Cl₂ (15 mL). The reaction mixture was stirred for 30 minutes at 30 °C. After completion of the reaction, the residue was purified by column chromatography (SiO₂, DCM:MeOH = 90:10) to give 4 as a pale yellow solid (138.0 mg, 92%). Rf = 0.45 (DCM:MeOH = 9.5:0.5); m.p.: 227.1-228.4 °C.

¹H NMR (400 MHz, CDCl₃) δ/ppm: 8.86 (d, J = 3.8 Hz, 1H), 8.21 (dtd, J = 8.6, 7.0, 1.6 Hz, 1H), 7.47 (d, J = 8.4 Hz, 1H), 7.43 (t, J = 7.4 Hz, 1H), 7.40-7.36 (m, 2H), 7.35-7.23 (m, 2H), 5.35 (d, J = 18.5 Hz, 1H), 2.38-2.13 (m, 6H), 1.66-1.34 (m, 12H), 0.87 (t, J = 7.3 Hz, 9H).

¹³C{¹H}-NMR (100 MHz, CDCl₃) δ/ppm: 190.7, 190.6, 177.0, 159.2 (d, ²J_C-P = 6.2 Hz), 156.2, 139.3, 133.9, 130.0, 125.7 (d, ²J_C-P = 29.7 Hz), 123.1, 120.4, 118.3, 118.1, 96.5(d, ³J_C-P = 3.3 Hz), 27.6 (d, ¹J_C-P = 64.7 Hz), 25.2 (d, ²J_C-P = 46.9 Hz), 24.8, 24.1, 23.9, 20.4 (d, ³J_C-P = 43.9 Hz), 13.3.

³¹P NMR (162 MHz, CDCl₃) δ/ppm: 36.9.

HRMS (MALDI) m/z: [M]+ calcd for C₃₁H₃₇O₄P: 504.2429 found: 504.2424.


A 50 mL round-bottomed flask equipped with a magnetic stir bar was charged with 11-(tributyl-2$_5$-phosphanylidene)-10H-benzo[5,6]pentaleno[1,2-b]chromene-10,12(11H)-dione 3a (90.75 mg, 0.15 mmol), Trifluoromethanesulfonic acid (1.2 equiv.) in acetonitrile (1.5 mL). The reaction mixture was stirred for 30 minutes at 30 °C and monitored by TLC. After completion of the reaction, the solvent was removed in vacuo
and the crude compound was without purification to give 7 as a purple solid (85.0 mg, 90%). R_f = 0.35 (Hexanes:EtOAc = 9:1); mp.: 170.9-171.8 °C.

_H NMR (400 MHz, CDCl3) δ/ppm: 8.61 (d, J = 8.1 Hz, 1H), 7.86 (d, J = 7.6 Hz, 1H), 7.69 (d, J = 8.3 Hz, 1H), 7.65-7.43 (m, 4H), 7.22-7.09 (m, 1H), 2.87-2.54 (m, 6H), 1.71-1.35 (m, 12H), 0.94 (t, J = 6.8 Hz, 9H).

_C{13}[H]-NMR (100 MHz, CDCl3) δ/ppm: 189.0, 164.2, 155.8, 147.8 (d, J_C,P = 7.6 Hz), 146.8, 140.1, 138.4, 135.7, 135.3, 126.6, 125.9, 125.3, 125.1, 124.9, 120.9, 120.3 (q, J_C,F = 320.3 Hz) 119.6 (d, J_C,F = 9.9 Hz), 118.4 (d, J_C,F = 12.1 Hz), 117.9, 114.9, 86.5 (d, J_C,F = 98.2 Hz), 24.2 (d, J_C,F = 4.2 Hz), 23.6 (d, J_C,F = 15.9 Hz), 21.5, (d, J_C,F = 51.3 Hz), 13.4.

_P NMR (162 MHz, CDCl3) δ/ppm: 24.9.

_F NMR (376 MHz, CDCl3) δ/ppm: -78.2.

HRMS (MALDI) m/z: [M+Na] calecd for C_{32}H_{36}F_{3}O_{6}PSNa: 659.1820 found: 659.1814.


A 50 mL round-bottomed flask equipped with a magnetic stir bar was charged with LAH (11.0 mg, 2.0 equiv.) in anhydrous THF (0.5 mL) at 0 °C and 11-(tributyl-5-phosphanylidene)-10H-benzo[5,6]pentaleno[1,2-b]chromene-10,12(11H)-dione 3a (73.0 mg, 0.15 mmol) dissolved in anhydrous THF (0.5 mL) adding to reaction mixture. The reaction mixture was stirred for 30 minutes at 0 °C and monitored by TLC. After completion of the reaction, the residue was purified by column chromatography (SiO2, Hexanes:EtOAc = 80:20) to give 8 as a yellow solid (59.0 mg, 80%). R_f = 0.38 (Hexanes:EtOAc =7:3); mp.: 172.9-173.8 °C.

_H NMR (400 MHz, DMSO) δ/ppm: 8.18 (dd, J = 7.9, 1.2 Hz, 1H), 7.65-7.59 (m, 1H), 7.58-7.54 (m, 1H), 7.43-7.36 (m, 2H), 7.31 (t, J = 7.2 Hz, 1H), 7.23 (t, J = 7.4 Hz, 1H), 6.98 (t, J = 7.4 Hz, 1H), 5.60 (d, J = 8.8 Hz, 1H), 5.38 (d, J = 8.8 Hz, 1H), 2.68-2.50 (m, 6H), 1.54-1.26 (m, 12H), 0.84 (t, J = 6.9 Hz, 1H).

_C{13}[H]-NMR (100 MHz, DMSO) δ/ppm: 169.7, 154.8, 148.8, 148.6 (d, J_C,P = 9.6 Hz), 144.8 (d, J_C,P = 11.5 Hz), 137.8, 131.1, 128.1, 125.6, 124.7, 122.9, 122.6, 122.2, 117.4, 117.0, 116.4 (d, J_C,P = 13.4 Hz), 115.7 (d, J_C,P = 12.5 Hz), 74.8 (d, J_C,P = 107.4 Hz), 70.6, 23.3 (d, J_C,P = 4.4 Hz), 23.2 (d, J_C,P = 7.6 Hz), 19.7 (d, J_C,P = 53.4 Hz), 13.3.
$^{31}$P NMR (162 MHz, CDCl$_3$) δ/ppm: 22.1.

HRMS (MALDI) m/z: [M]$^+$ calcd for C$_{31}$H$_{37}$O$_3$P: 488.2480 found: 488.2475.

2-(1-benzyl-4-oxo-2-phenyl-1,4-dihydrochromeno[2,3-b]pyrrol-3-yl)-1H-indene-1,3(2H)-dione 9.

A 50 mL round-bottomed flask equipped with a magnetic stir bar was charged with 2-((4-oxo-4H-chromen-3-yl)methylene)-1H-indene-1,3(2H)-dione 5aa (61.0 mg, 0.15 mmol), and benzylamine (25.0 µL, 1.5 equiv) in DMSO (0.75 mL). The reaction mixture was stirred for 30 minutes at 30 °C. After completion of the reaction, the residue was purified by column chromatography (SiO$_2$, Hexanes:EtOAc = 65:35) to give 9 as an Off-white solid (56.0 mg, 75%). R$_f$ = 0.40 (Hexanes:EtOAc = 7:3); mp.: 211.0-212.2 °C.

$^1$H NMR (400 MHz, CDCl$_3$) δ/ppm: 8.11 (dd, $J$ = 8.0, 1.5 Hz, 1H), 8.09-8.03 (m, 2H), 7.98-7.83 (m, 2H), 7.55 (td, $J$ = 8.9, 6.8, 1.5 Hz, 1H), 7.52-7.46 (m, 2H), 7.45-7.37 (m, 4H), 7.34-7.24 (m, 4H), 7.07 (d, $J$ = 6.8 Hz, 1H), 5.26 (s, 2H), 4.28 (s, 1H).

$^{13}$C{$^1$H}-NMR (100 MHz, CDCl$_3$) δ/ppm: 198.1, 172.9, 154.2, 149.8, 142.3, 136.1, 135.2, 133.5, 132.4, 131.3, 129.2, 128.9, 128.8, 128.6, 127.9, 126.8, 126.7, 124.2, 123.3, 123.2, 117.2, 107.1, 104.3, 53.8, 46.8.

HRMS (EI) m/z: [M]$^+$ calcd for C$_{33}$H$_{21}$NO$_4$: 495.1471 found: 495.1457.

References

**VII. X-ray crystallographic data for selected compounds**

**a) Crystal Data and Structure Refinement for compound 3a (CCDC no. 2360174):**

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<tr>
<td>F(000)</td>
<td>1040</td>
</tr>
<tr>
<td>Crystal size</td>
<td>0.24 x 0.21 x 0.02 mm³</td>
</tr>
<tr>
<td>Theta range for data collection</td>
<td>2.53 to 25.06°</td>
</tr>
<tr>
<td>Index ranges</td>
<td>-14&lt;=h&lt;=14, -25&lt;=k&lt;=25, -12&lt;=l&lt;=12</td>
</tr>
<tr>
<td>Reflections collected</td>
<td>25869</td>
</tr>
<tr>
<td>Independent reflections</td>
<td>4800 [R(int) = 0.0956]</td>
</tr>
<tr>
<td>Completeness to theta = 25.06°</td>
<td>99.8 % Absorption correction multi-scan</td>
</tr>
</tbody>
</table>

Max. and min. transmission        0.9974 and 0.9693
Refinement method                 Full-matrix least-squares on F²
Data / restraints / parameters     4800 / 0 / 319
Goodness-of-fit on F²              1.017
Final R indices [I>2σ(I)]          R1 = 0.0534, wR2 = 0.1240
R indices (all data)               R1 = 0.0872, wR2 = 0.1428
Largest diff. peak and hole        0.214 and -0.345 e.Å⁻³

The purified compound 3a was dissolved in a minimal amount of CH₂Cl₂ in a glass vial. Hexanes were then added until the solution became turbid. The solution was allowed to slowly evaporate. After a few days, a dark red color crystals were obtained.
b) Crystal Data and Structure Refinement for compound 5aa (CCDC no. 2360188):

The purified compound 5aa was dissolved in a minimal amount of CH₂Cl₂ in a glass vial. Hexanes were then added until the solution became turbid. The solution was allowed to slowly evaporate. After a few days, a pale yellow color crystals were obtained.

Empirical formula: C₂₆H₁₄O₅

Formula weight: 406.37

Temperature: 200(2) K

Wavelength: 0.71073 Å

Crystal system: Monoclinic

Space group: P 2₁/c

Unit cell dimensions:
- a = 19.4485(7) Å, α = 90°
- b = 6.3637(2) Å, β = 105.2800(10)°
- c = 15.7718(5) Å, γ = 90°

Volume: 1882.98(11) Å³

Z: 4

Density (calculated): 1.433 Mg/m³

Absorption coefficient: 0.100 mm⁻¹

F(000): 840

Crystal size: 0.41 x 0.20 x 0.04 mm³

Theta range for data collection: 2.68 to 25.10°

Index ranges:
- -23 ≤ h ≤ 23
- -7 ≤ k ≤ 7
- -18 ≤ l ≤ 18

Reflections collected: 19261

Independent reflections: 3344 [R(int) = 0.0838]

Completeness to theta = 25.10°: 99.3%

Absorption correction: multi-scan

Max. and min. transmission: 0.9960 and 0.9602

Refinement method: Full-matrix least-squares on F²

Data / restraints / parameters: 3344 / 0 / 280

Goodness-of-fit on F²: 1.023

Final R indices [I>2sigma(I)]: R1 = 0.0443, wR2 = 0.1107

R indices (all data): R1 = 0.0584, wR2 = 0.1239

Largest diff. peak and hole: 0.219 and -0.184 e Å⁻³
c) Crystal Data and Structure Refinement for compound 4 (CCDC no. 2360233):

<table>
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<tr>
<th>Property</th>
<th>Value</th>
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</thead>
<tbody>
<tr>
<td>Empirical formula</td>
<td>C_{31}H_{36}O_4P</td>
</tr>
<tr>
<td>Formula weight</td>
<td>503.57</td>
</tr>
<tr>
<td>Temperature</td>
<td>200(2) K</td>
</tr>
<tr>
<td>Wavelength</td>
<td>0.71073 Å</td>
</tr>
<tr>
<td>Crystal system</td>
<td>Monoclinic</td>
</tr>
<tr>
<td>Space group</td>
<td>P 21/n</td>
</tr>
<tr>
<td>Unit cell dimensions</td>
<td>a = 9.6734(4) Å, α = 90°</td>
</tr>
<tr>
<td></td>
<td>b = 14.6130(6) Å, β = 101.356(2)°</td>
</tr>
<tr>
<td></td>
<td>c = 19.9002(9) Å, γ = 90°</td>
</tr>
<tr>
<td>Volume</td>
<td>2758.0(2) Å³</td>
</tr>
<tr>
<td>Z</td>
<td>4</td>
</tr>
<tr>
<td>Density (calculated)</td>
<td>1.213 Mg/m³</td>
</tr>
<tr>
<td>Absorption coefficient</td>
<td>0.133 mm⁻¹</td>
</tr>
<tr>
<td>F(000)</td>
<td>1076</td>
</tr>
<tr>
<td>Crystal size</td>
<td>0.13 x 0.10 x 0.03 mm³</td>
</tr>
<tr>
<td>Theta range for data collection</td>
<td>2.20 to 25.38°</td>
</tr>
<tr>
<td>Index ranges</td>
<td>-11&lt;=h&lt;=11, -17&lt;=k&lt;=17, -23&lt;=l&lt;=23</td>
</tr>
<tr>
<td>Reflections collected</td>
<td>31956</td>
</tr>
<tr>
<td>Independent reflections</td>
<td>5064 [R(int) = 0.0799]</td>
</tr>
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<td>Completeness to theta = 25.38°</td>
<td>99.8 %</td>
</tr>
<tr>
<td>Absorption correction</td>
<td>multi-scan</td>
</tr>
<tr>
<td>Max. and min. transmission</td>
<td>0.9960 and 0.9829</td>
</tr>
<tr>
<td>Refinement method</td>
<td>Full-matrix least-squares on F²</td>
</tr>
<tr>
<td>Data / restraints / parameters</td>
<td>5064 / 0 / 327</td>
</tr>
<tr>
<td>Goodness-of-fit on F²</td>
<td>1.054</td>
</tr>
<tr>
<td>Final R indices [I&gt;2sigma(I)]</td>
<td>R1 = 0.0684, wR2 = 0.1967</td>
</tr>
<tr>
<td>R indices (all data)</td>
<td>R1 = 0.1036, wR2 = 0.2344</td>
</tr>
<tr>
<td>Largest diff. peak and hole</td>
<td>1.277 and -0.440 e.Å⁻³</td>
</tr>
</tbody>
</table>

The purified compound 4 was dissolved in a minimal amount of CH₂Cl₂ in a glass vial. Hexanes were then added until the solution became turbid. The solution was allowed to slowly evaporate. After a few days, a pale yellow color crystals were obtained.
d) Crystal Data and Structure Refinement for compound 8 (CCDC no. 2363295):

The purified compound 4 was dissolved in a minimal amount of CH₂Cl₂ in a glass vial. Hexanes were then added until the solution became turbid. The solution was allowed to slowly evaporate. After a few days, a pale yellow color crystals were obtained.

**Empirical formula**: C₃₁H₃₇O₃P

**Formula weight**: 488.58

**Temperature**: 200(2) K

**Wavelength**: 0.71073 Å

**Crystal system**: Monoclinic

**Space group**: P 2₁/n

**Unit cell dimensions**:

- \(a = 11.3575(5) \text{ Å}\), \(\alpha = 90^\circ\)
- \(b = 11.1326(5) \text{ Å}\), \(\beta = 97.976(2)^\circ\)
- \(c = 21.2159(11) \text{ Å}\), \(\gamma = 90^\circ\)

**Volume**: 2656.6(2) Å³

**Z**: 4

**Density (calculated)**: 1.222 Mg/m³

**Absorption coefficient**: 0.134 mm⁻¹

**F(000)**: 1048

**Crystal size**: 0.22 x 0.09 x 0.03 mm³

**Theta range for data collection**: 2.07 to 25.05°.

**Index ranges**: -13 ≤ h ≤ 13, -13 ≤ k ≤ 13, -25 ≤ l ≤ 23

**Reflections collected**: 24725

**Independent reflections**: 4697 [R(int) = 0.0721]

**Completeness to theta = 25.05°**: 99.9 %

**Absorption correction**: None

**Max. and min. transmission**: 0.9960 and 0.9712

**Refinement method**: Full-matrix least-squares on F²

**Data / restraints / parameters**: 4697 / 0 / 319

**Goodness-of-fit on F²**: 1.029

**Final R indices [I>2sigma(I)]**: R₁ = 0.0442, wR₂ = 0.1230

**R indices (all data)**: R₁ = 0.0590, wR₂ = 0.1386

**Largest diff. peak and hole**: 0.199 and -0.331 e.Å⁻³
VIII. $^1$H, $^{13}$C NMR, $^{19}$F NMR and $^{31}$P NMR spectra for all compounds

$^1$H NMR spectrum of compound 1a (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 1a (CDCl$_3$, 100 MHz)
$^1$H NMR spectrum of compound 1b (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 1b (CDCl$_3$, 100 MHz)
$^1$H NMR spectrum of compound 1c (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 1c (CDCl$_3$, 100 MHz)
$^1$H NMR spectrum of compound 1d (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 1d (CDCl$_3$, 100 MHz)
$^{19}$F NMR spectrum of compound 1d (CDCl$_3$, 376 MHz)

1H NMR spectrum of compound 1e (CDCl$_3$, 400 MHz)
$^{13}$C NMR spectrum of compound 1e (CDCl$_3$, 100 MHz)

$^1$H NMR spectrum of compound 1f (CDCl$_3$, 400 MHz)
$^{13}$C NMR spectrum of compound 1f (CDCl$_3$, 100 MHz)

$^1$H NMR spectrum of compound 1g (CDCl$_3$, 400 MHz)
$^{13}$C NMR spectrum of compound 1g (CDCl$_3$, 100 MHz)

$^1$H NMR spectrum of compound 1h (CDCl$_3$, 400 MHz)
$^{13}$C NMR spectrum of compound 1h (CDCl$_3$, 100 MHz)

$^1$H NMR spectrum of compound 1i (CDCl$_3$, 400 MHz)
$^{13}$C NMR spectrum of compound 1i (CDCl$_3$, 100 MHz)

$^1$H NMR spectrum of compound 1j (CDCl$_3$, 400 MHz)
$^{13}$C NMR spectrum of compound 1j (CDCl$_3$, 100 MHz)

$^{19}$F NMR spectrum of compound 1j (CDCl$_3$, 376 MHz)
$^1$H NMR spectrum of compound 1k (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 1k (CDCl$_3$, 100 MHz)
$^1$H NMR spectrum of compound 11 (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 11 (CDCl$_3$, 100 MHz)
$^1$H NMR spectrum of compound 1m (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 1m (CDCl$_3$, 100 MHz)
$^1$H NMR spectrum of compound 1n (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 1n (CDCl$_3$, 100 MHz)
$^1$H NMR spectrum of compound 1o (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 1o (CDCl$_3$, 100 MHz)
$^1$H NMR spectrum of compound 1p (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 1p (CDCl$_3$, 100 MHz)
$^1$H NMR spectrum of compound 1q (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 1q (CDCl$_3$, 100 MHz)
$^1$H NMR spectrum of compound 1r (CDCl₃, 400 MHz)

$^{13}$C NMR spectrum of compound 1r (CDCl₃, 100 MHz)
$^1$H NMR spectrum of compound 3a (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 3a (CDCl$_3$, 100 MHz)
$^3$P NMR spectrum of compound 3a (CDCl$_3$, 162 MHz)

$^1$H NMR spectrum of compound 3b (CDCl$_3$, 400 MHz)
$^{13}$C NMR spectrum of compound 3b (CDCl$_3$, 100 MHz)

$^{31}$P NMR spectrum of compound 3b (CDCl$_3$, 162 MHz)
$^1$H NMR spectrum of compound 3c (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 3c (CDCl$_3$, 100 MHz)
$^{31}$P NMR spectrum of compound 3c (CDCl$_3$, 162 MHz)

$^1$H NMR spectrum of compound 3d (CDCl$_3$, 400 MHz)
$^{13}$C NMR spectrum of compound 3d (CDCl$_3$, 100 MHz)

$^{31}$P NMR spectrum of compound 3d (CDCl$_3$, 162 MHz)
$^{19}$F NMR spectrum of compound 3d (CDCl$_3$, 376 MHz)

$^1$H NMR spectrum of compound 3e (CDCl$_3$, 400 MHz)
$^{13}$C NMR spectrum of compound 3e (CDCl$_3$, 100 MHz)

$^{31}$P NMR spectrum of compound 3e (CDCl$_3$, 162 MHz)
$^1$H NMR spectrum of compound 3f (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 3f (CDCl$_3$, 100 MHz)
$^{31}\text{P} \text{ NMR spectrum of compound } 3f (\text{CDCl}_3, 162 \text{ MHz})$

$^{1}\text{H} \text{ NMR spectrum of compound } 3g (\text{CDCl}_3, 400 \text{ MHz})$
$^{13}$C NMR spectrum of compound $3g$ (CDCl$_3$, 100 MHz)

$^{31}$P NMR spectrum of compound $3g$ (CDCl$_3$, 162 MHz)
$^1$H NMR spectrum of compound 3h (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 3h (CDCl$_3$, 100 MHz)
$^{13}$P NMR spectrum of compound 3h (CDCl$_3$, 162 MHz)

$^1$H NMR spectrum of compound 3i (CDCl$_3$, 400 MHz)
$^{13}$C NMR spectrum of compound 3i (CDCl$_3$, 100 MHz)

$^{31}$P NMR spectrum of compound 3i (CDCl$_3$, 162 MHz)
$^1$H NMR spectrum of compound 3j (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 3j (CDCl$_3$, 100 MHz)
$^{31}$P NMR spectrum of compound 3j (CDCl$_3$, 162 MHz)

$^{19}$F NMR spectrum of compound 3j (CDCl$_3$, 376 MHz)
$^1$H NMR spectrum of compound 3k (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 3k (CDCl$_3$, 100 MHz)
$^3$P NMR spectrum of compound 3k (CDCl$_3$, 162 MHz)

$^1$H NMR spectrum of compound 3l (CDCl$_3$, 400 MHz)
$^{13}$C NMR spectrum of compound 3I (CDCl$_3$, 100 MHz)

$^{31}$P NMR spectrum of compound 3I (CDCl$_3$, 162 MHz)
$^1$H NMR spectrum of compound $3m$ (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound $3m$ (CDCl$_3$, 100 MHz)
$^{31}$P NMR spectrum of compound 3m (CDCl$_3$, 162 MHz)

$^1$H NMR spectrum of compound 3n (CDCl$_3$, 400 MHz)
$^{13}$C NMR spectrum of compound 3n (CDCl$_3$, 100 MHz)

$^{31}$P NMR spectrum of compound 3n (CDCl$_3$, 162 MHz)
$^1$H NMR spectrum of compound $3\alpha$ (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound $3\alpha$ (CDCl$_3$, 100 MHz)
$^3$P NMR spectrum of compound 3o (CDCl$_3$, 162 MHz)

$^1$H NMR spectrum of compound 3p (CDCl$_3$, 400 MHz)
$^{13}$C NMR spectrum of compound 3p (CDCl$_3$, 100 MHz)

$^{31}$P NMR spectrum of compound 3p (CDCl$_3$, 162 MHz)
$^1$H NMR spectrum of compound 3q (CDCl$_3$, 400 MHz)

13C NMR spectrum of compound 3q (CDCl$_3$, 100 MHz)
$^{31}$P NMR spectrum of compound 3q (CDCl$_3$, 162 MHz)

$^1$H NMR spectrum of compound 6 (CDCl$_3$, 400 MHz)
$^{13}$C NMR spectrum of compound 6 (CDCl$_3$, 100 MHz)
$^1$H NMR spectrum of compound 5aa (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 5aa (CDCl$_3$, 100 MHz)
$^1$H NMR spectrum of compound 5ba (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 5ba (CDCl$_3$, 100 MHz)
$^1$H NMR spectrum of compound 5ea (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 5ea (CDCl$_3$, 100 MHz)
$^1$H NMR spectrum of compound 5ia (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 5ia (CDCl$_3$, 100 MHz)
$^1$H NMR spectrum of compound 5ja (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 5ja (CDCl$_3$, 100 MHz)
$^{19}$F NMR spectrum of compound 5ja (CDCl$_3$, 376 MHz)
$^1$H NMR spectrum of compound 5la (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 5la (CDCl$_3$, 100 MHz)
H NMR spectrum of compound 5ma (CDCl₃, 400 MHz)

13C NMR spectrum of compound 5ma (CDCl₃, 100 MHz)
$^1$H NMR spectrum of compound 5na (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 5na (CDCl$_3$, 100 MHz)
$^1$H NMR spectrum of compound 5oa (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 5oa (CDCl$_3$, 100 MHz)
$^1$H NMR spectrum of compound 5ab (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 5ab (CDCl$_3$, 100 MHz)
$^{13}$C NMR spectrum of compound 5ac (CDCl$_3$, 400 MHz)

![C NMR Spectrum](image)

$^{13}$C NMR spectrum of compound 5ac (CDCl$_3$, 100 MHz)

![C NMR Spectrum](image)
$^1$H NMR spectrum of compound 5ad (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 5ad (CDCl$_3$, 100 MHz)
$^{19}$F NMR spectrum of compound $5ad$ (CDCl$_3$, 376 MHz)
$^1$H NMR spectrum of compound 5ae (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 5ae (CDCl$_3$, 100 MHz)
\[ ^{1}\text{H} \text{NMR spectrum of compound } 5\text{af (CDCl}_3, 400 \text{ MHz)} \]

\[ ^{13}\text{C} \text{NMR spectrum of compound } 5\text{af (CDCl}_3, 100 \text{ MHz)} \]
$^1$H NMR spectrum of compound 5ag (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 5ag (CDCl$_3$, 100 MHz)
$^{1}$H NMR spectrum of compound 5ah (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 5ah (CDCl$_3$, 100 MHz)
$^1$H NMR spectrum of compound 5ai (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 5ai (CDCl$_3$, 100 MHz)
$^1$H NMR spectrum of compound 5aj (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 5aj (CDCl$_3$, 100 MHz)
$^1$H NMR spectrum of compound 5ak (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 5ak (CDCl$_3$, 100 MHz)
$^1$H NMR spectrum of compound 4 (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 4 (CDCl$_3$, 100 MHz)
$^{31}$P NMR spectrum of compound 4 (CDCl$_3$, 162 MHz)
**1H NMR spectrum of compound 7 (CDCl₃, 400 MHz)**

![1H NMR spectrum of compound 7 (CDCl₃, 400 MHz)](image1)

**13C NMR spectrum of compound 7 (CDCl₃, 100 MHz)**

![13C NMR spectrum of compound 7 (CDCl₃, 100 MHz)](image2)
$^{31}$P NMR spectrum of compound 7 (CDCl$_3$, 162 MHz)

$^{19}$F NMR spectrum of compound 7 (CDCl$_3$, 376 MHz)
$^1$H NMR spectrum of compound 8 (DMSO, 400 MHz)

$^{13}$C NMR spectrum of compound 8 (DMSO, 100 MHz)
$^{31}$P NMR spectrum of compound 8 (DMSO, 162 MHz)
$^1$H NMR spectrum of compound 9 (CDCl$_3$, 400 MHz)

$^{13}$C NMR spectrum of compound 9 (CDCl$_3$, 100 MHz)