Supporting Information
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Efficient Synthesis of Substituted Polyarylphthalimides via Cycloaddition of Cyclopentadienones with 2-Bromomaleimide.

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Supporting Information

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General

Reactions were monitored by thin layer chromatography (TLC) using commercial aluminium-backed silica gel plates. TLC spots were viewed under ultraviolet light and by heating the plate after treatment with phosphomolybdic acid or potassium permanganate. Unless otherwise noted, all reagent-grade chemicals and solvents were obtained from commercial suppliers and were used as received. Chromatography purifications were performed by column chromatography using Silica Gel 60 (40-60 mesh). Melting points were obtained using a heating rate of 5°C/min and are uncorrected. Infrared spectra (IR): a fourier transform infrared spectrometer was used (using ATR (Attenuated Total Reflexion) for solid compounds) and the data are reported in reciprocal centimetres (cm⁻¹). ¹H NMR Spectra (300 MHz or 400 MHz), ¹⁹F NMR and ¹³C NMR spectra (75 MHz or 100 MHz) were recorded on a Bruker Advance 300 spectrometer and a Bruker Advance 400 spectrometer. Fluorobenzene was used as internal reference in ¹⁹F NMR spectroscopy. Low Resolution Mass Spectra (LRMS) were recorded either on an ion-trap spectrometer (ESI or DCI). HRMS were recorded on an Orbitrap apparatus (ESI). Elemental analyses were performed by the analytical service of the DCM.
General procedure: Synthesis of substituted dibenzylketones

Dibenzylketones were obtained according to a procedure developed by Van Leusen and coll.\textsuperscript{1} using TosMIC (toluenesulfonylmethyl isocyanide) and substituted benzylbromides in phase transfer catalysis conditions.

TosMIC, benzyl bromide and TBAI (tetrabutylammonium iodide) were stirred vigorously in CH\textsubscript{2}Cl\textsubscript{2} for 10 min before aqueous NaOH (40 wt %) was added. Vigorous stirring was carried on for the indicated time. The mixture was then neutralized with HCl (10 wt %) and the organic layer was collected. The aqueous layer was extracted 3 times with CH\textsubscript{2}Cl\textsubscript{2} and the organic layers were dried over anhydrous Na\textsubscript{2}SO\textsubscript{4}. Evaporation of the solvent gave the crude dialkylated isocyanide which was eventually dissolved in CH\textsubscript{2}Cl\textsubscript{2} and HCl (37 wt %) and vigorously stirred for the indicated time at room temperature. The mixture was neutralized with saturated NaHCO\textsubscript{3}. The organic layer was collected, the aqueous layer was extracted three times with CH\textsubscript{2}Cl\textsubscript{2} and the organic layers were dried over Na\textsubscript{2}SO\textsubscript{4}; removal of the solvent under vacuum gave a crude product which was purified by silica gel chromatography using the indicated solvents to yield the desired dibenzylketone.

1,3-Bis(4-bromophenyl)propan-2-one was synthesized using the general procedure starting from 0.980 g of TosMIC (5 mmol), 2.499 g of 4-bromobenzylbromide (10 mmol), 0.420 g (20 mole %) of TBAI in 120 mL of CH\textsubscript{2}Cl\textsubscript{2} and 50 mL of NaOH (40 wt %) vigorously stirred during 4h. Hydrolysis of the dialkylated isocyanide was carried out in 50 mL of CH\textsubscript{2}Cl\textsubscript{2} and 5 mL of HCl (37 wt %) stirred during 4h. The crude red oil was purified by silica gel chromatography using CH\textsubscript{2}Cl\textsubscript{2} to give 1.130 g of an oily white solid (61%).

\[ ^1\text{H NMR (300 MHz, CDCl}_3\text{) } \delta \text{ (ppm) : 7.45 (d, } J = 8.4 \text{ Hz, 4H), 7.01 (d, } J = 8.5 \text{ Hz, 4H), 3.68 (s, 4H).} \textsuperscript{2} \]

1,3-Di-p-tolylpropan-2-one was synthesized using another procedure developed by Van Leusen and coll.\textsuperscript{1} To a stirring suspension of 0.440 g of NaH (60 wt %, 10 mmol) in 2 mL of dry DMSO and 8 mL of dry Et\textsubscript{2}O, under argon, was added dropwise 0.980 g of TosMIC (5 mmol) in 8 mL of dry Et\textsubscript{2}O. After 10 min, 1.851 g of 4-methylbenzylbromide (10 mmol) in 8
mL of dry Et$_2$O was added. After 4h, stirring was stopped and the organic layer was washed six times with water, brine and dried over anhydrous Na$_2$SO$_4$. Removal of the solvents under vacuum gave the dialkylated isocyanide which was dissolved in 50 mL of CH$_2$Cl$_2$ and 5 mL of HCl (37 % wt); the solution was then vigorously stirred at room temperature. After 2.5h, the mixture was neutralized with saturated NaHCO$_3$ and the organic layer was collected. The aqueous layer was extracted three times with CH$_2$Cl$_2$ and the organic layers were dried over Na$_2$SO$_4$. Removal of the solvents under vacuum gave a solid that was purified by silica gel chromatography using pentane and CH$_2$Cl$_2$ to give 0.660 g of the desired product as an oily solid (60%).

\[\begin{align*}
\text{1H NMR (300 MHz, CDCl}_3\text{)} & \text{ } \delta (\text{ppm}) : 7.13 (d, J = 8.2 \\
& \text{ } \text{Hz, 4H), 7.04 (d, J = 8.0 Hz, 4H), 3.66 (s, 4H), 2.33 (s, 6H).}\text{ }^2
\end{align*}\]

1-(Bromomethyl)-4-methoxybenzene was synthesized using a procedure described by Maier and coll.$^3$ To a solution of 254 µL of PBr$_3$ (731 mg, 2.7 mmol) in 5 mL of Et$_2$O was added dropwise a solution of 690 mg of (4-methoxyphenyl)methanol (5 mmol) in 1 mL of Et$_2$O. The mixture was stirred at room temperature for 2h. The solution was poured on ice. The organic layer was then collected and treated with saturated NaHCO$_3$, washed with water and brine before being dried over Na$_2$SO$_4$. Evaporation of solvent gave 940 mg of the desired product as pale yellow oil (95%) used directly without further purification for the following reaction.

\[\begin{align*}
\text{1H NMR (300 MHz, CDCl}_3\text{)} & \text{ } \delta (\text{ppm}) : 7.33 (d, J = 8.7 Hz, 2H), 6.87 \\
& \text{ } \text{(d, J = 8.7 Hz, 2H), 4.51 (s, 2H), 3.81 (s, 3H).}\text{ }^3
\end{align*}\]

1,3-Bis(4-methoxyphenyl)propan-2-one was synthesized using the general procedure starting from 0.980 g of TosMIC (5 mmol), 2.200 g of 1-(bromomethyl)-4-methoxybenzene (10.1 mmol.) and 0.220 g of TBAI (12 mole %) vigorously stirred during 1h. Hydrolysis of the dialkylated isocyanide was carried out in 50 mL of CH$_2$Cl$_2$ and 5 mL of HCl (37 wt %) stirred during 15 min. The crude red solid was purified by silica gel chromatography using CH$_2$Cl$_2$ and pentane to give 0.811 g of the desired product as a white solid (60%).
1,3-Bis(4-chlorophenyl)propan-2-one was obtained using the general procedure starting from 0.760 g of TosMIC (4 mmol), 1.640 g of 4-chlorobenzylbromide (8 mmol) and 0.325 g of TBAI (22 mole %) in 100 mL of CH₂Cl₂ and 30 mL of aqueous NaOH (40 wt %) vigorously stirred during 1h. Hydrolysis of the dialkylated isocyanide was carried out in 40 mL of CH₂Cl₂ and 4 mL of HCl (37 wt %). The red crude solid was purified by silica gel chromatography to give 485 mg of the desired product as a white oily solid (43%).

\[
\begin{align*}
&\text{H NMR (300 MHz, CDCl₃)} \delta (\text{ppm}): 7.06 (d, J = 8.7 \text{ Hz}, 4\text{H}), 6.85 (d, J = 8.7 \text{ Hz}, 4\text{H}), 3.80 (s, 6\text{H}), 3.64 (s, 4\text{H}).^2 \\
&\text{1,3-Bis(4-chlorophenyl)propan-2-one was obtained using the general procedure starting from 0.760 g of TosMIC (4 mmol), 1.640 g of 4-chlorobenzylbromide (8 mmol) and 0.325 g of TBAI (22 mole %) in 100 mL of CH₂Cl₂ and 30 mL of aqueous NaOH (40 wt %) vigorously stirred during 1h. Hydrolysis of the dialkylated isocyanide was carried out in 40 mL of CH₂Cl₂ and 4 mL of HCl (37 wt %). The red crude solid was purified by silica gel chromatography to give 485 mg of the desired product as a white oily solid (43%).}
\end{align*}
\]

\[
\begin{align*}
&\text{H NMR (400 MHz, CDCl₃)} \delta (\text{ppm}): 7.29 (d, J = 8.41 \text{ Hz}, 4\text{H}), 7.07 (d, J = 8.4 \text{ Hz}, 4\text{H}), 3.69 (s, 4\text{H}).^4
\end{align*}
\]
General procedure for the transformation of dibenzylketones to tetracyclones and acecyclones.

Tetracyclones and acecyclones were synthesized using typical Knoevenagel condensation between substituted dibenzylketones and substituted 1,2-diaryldiketones or acenaphthenequinone in an absolute ethanolic solution of KOH refluxed for indicated time. The solution was then cooled to room temperature. If the product precipitates from the mixture it was eventually filtered and washed with absolute ethanol. Otherwise, ethanol was removed under vacuum before water and CH$_2$Cl$_2$ were added. The organic layer was collected while the aqueous phase was extracted with CH$_2$Cl$_2$. The combined organic layers were then dried over Na$_2$SO$_4$ and solvents were removed under vacuum to give a crude product which was purified by silica gel chromatography using the indicated solvents to yield the desired tetracyclone or acecyclone.

3,4-Bis(4-methoxyphenyl)-2,5-diphenylcyclopenta-2,4-dienone (3b) was synthesized using the general procedure starting from 1.050 g of dibenzylketone (5 mmol), 1.350 g of 1,2-bis(4-methoxyphenyl)ethane-1,2-dione (5 mmol) and 0.280 g of KOH (5 mmol) in 25 mL of refluxing ethanol during 3h. After filtration, 3b was isolated as a dark purple solid (1.801 g, 81%).

$^1$H NMR (CDCl$_3$, 400 MHz) δ (ppm) : 7.25-7.20 (m, 10H), 6.86 (d, $J = 8.8$ Hz, 4H), 6.72 (d, $J = 8.7$ Hz, 4H), 3.79 (s, 6H).$^5$

m.p. = 238.0-238.1°C (litt.$^6$ 226.8-227.2°C).

2,5-Diphenyl-3,4-di-p-tolylcyclopenta-2,4-dienone (3c) was synthesized using the general procedure starting from 1.051 g of dibenzylketone (5 mmol), 1.191 g of 1,2-di-p-tolyethylene-1,2-dione (5 mmol ) and 0.280 g of KOH (5 mmol) in 25 mL of refluxing ethanol during 2.5h. After filtration, 3c was isolated as a dark purple solid (1.600 g, 78%).
3,4-Bis(4-fluorophenyl)-2,5-diphenylcyclopenta-2,4-dienone (3d) was synthesized using the general procedure starting from 420 mg of dibenzylketone (2 mmol), 492 mg of 1,2-bis(4-fluorophenyl)ethane-1,2-dione (2 mmol) and 112 mg of KOH (2 mmol) in 10 mL of refluxing ethanol during 4h. After filtration, 3d was isolated as a dark purple solid (246 mg, 29%).

3,4-Bis(4-bromophenyl)-2,5-diphenylcyclopenta-2,4-dienone (3e) was synthesized using the general procedure starting from 1.051 g of dibenzylketone (5 mmol), 1.840 g of 1,2-bis(4-bromophenyl)ethane-1,2-dione (5 mmol) and 0.280 g of KOH (5 mmol) in 13 mL of refluxing ethanol for 3h. After filtration, 3e was isolated as a dark purple solid (2.193 g, 81%).

2,5-Bis(4-methoxyphenyl)-3,4-diphenylcyclopenta-2,4-dienone (3f) was synthesized using the general procedure starting from 270 mg of 1,3-bis(4-methoxyphenyl)propan-2-one (1...
mmol), 231 mg of benzil (1.1 mmol) and 56 mg of KOH (1 mmol) in 5 mL of refluxing ethanol for 2h. After filtration and purification over silica gel of the filtrate using CH₂Cl₂ and pentane, 3f was isolated as a dark purple solid (214 mg, 48%).

\[ \text{\textsuperscript{1}H NMR (CDCl₃, 300.13 MHz) \( \delta \) (ppm):} \] 7.25-7.16 (m, 10H), 6.95 (d, \( J = 8.3 \) Hz, 4H), 6.80 (d, \( J = 8.9 \) Hz, 4H).

\[ \text{m.p. = 182.1-182.2°C (litt.\textsuperscript{11} 192-193°C).} \]

3,4-Diphenyl-2,5-di-p-tolylcyclopenta-2,4-dienone (3g) was synthesized using the general procedure starting from 220 mg of 1,3-di-p-tolylpropan-2-one (1 mmol), 592 mg of benzil (2.8 mmol) and 61 mg of KOH (1.1 mmol) in 3 mL of refluxing ethanol during 2.5h. After removal of the ethanol and work up followed by silica gel chromatography using CH₂Cl₂ and pentane, a dark purple solid was isolated (106 mg of a 47:53 mixture of 3g:residual benzil, the yield was calculated by \textsuperscript{1}H NMR: 17%).

\[ \text{\textsuperscript{1}H NMR (CDCl₃, 300.13 MHz) \( \delta \) (ppm):} \] 7.25-7.09 (m, 10H), 7.04 (d, \( J = 8.5 \) Hz, 4H), 6.93 (d, \( J = 8.9 \) Hz, 4H), 2.31 (s, 6H).

\[ \text{m.p. = 235.1-235.2°C (litt.\textsuperscript{11} 239-240°C).} \]

2,5-Bis(4-chlorophenyl)-3,4-diphenylcyclopenta-2,4-dienone (3h) was synthesized using the general procedure starting from 222 mg of 1,3-bis(4-chlorophenyl)propan-2-one (0.8 mmol), 168 mg of benzil (0.8 mmol) and 45 mg of KOH (0.8 mmol) in 4 mL of refluxing ethanol during 1.5h. After filtration, 3h was isolated as a dark purple solid (195 mg, 54%).

\[ \text{\textsuperscript{1}H NMR (CDCl₃, 300.13 MHz) \( \delta \) (ppm):} \] 7.29-7.17 (m, 14H), 6.92 (d, \( J = 7.2 \) Hz, 4H).

\[ \text{m.p. = 235.1-235.2°C (litt.\textsuperscript{11} 239-240°C).} \]
2,5-Bis(4-bromophenyl)-3,4-diphenylcyclopenta-2,4-dienone (3i) was synthesized using the general procedure starting from 368 mg of 1,3-bis(4-bromophenyl)propan-2-one (1 mmol), 210 mg of benzil (1 mmol) and 56 mg of KOH (1 mmol) in 5 mL of refluxing ethanol during 0.5h. After filtration, 3i was isolated as a dark purple solid (293 mg, 54%).

\[ \text{m.p.} = 246.8-246.9^\circ\text{C} \] (litt.\(^6\) 249.5-250.0\(^\circ\text{C}).

\[ \text{\textsuperscript{1}H NMR (CDCl}_3, 300 \text{ MHz} \) \delta (ppm) : 7.37 (d, } J = 8.6 \text{ Hz, } 4\text{H}), 7.27-7.25 (m, } 2\text{H}), 7.22-7.17 (m, } 4\text{H}), 7.10 (d, } J = 8.6 \text{ Hz, } 4\text{H}), 6.90 (d, } J = 7.0 \text{ Hz, } 4\text{H}). \]

2,3,4,5-Tetrakis(4-methoxyphenyl)cyclopenta-2,4-dienone (3j) was synthesized using the general procedure starting from 270 mg of 1,3-bis(4-methoxyphenyl)propan-2-one (1 mmol), 297 mg of 1,2-bis(4-methoxyphenyl)ethane-1,2-dione (1.1 mmol) and 56 mg of KOH (1 mmol) in 5 mL of refluxing ethanol during 1h. After filtration, a dark purple solid were isolated (381 mg of a 90:10 mixture of 3j:residual 1,2-bis(4-methoxyphenyl)ethane-1,2-dione, the yield was calculated by \textsuperscript{1}H NMR : 75%).

\[ \text{\textsuperscript{1}H NMR (CDCl}_3, 400 \text{ MHz} \) \delta (ppm) : 7.21 (d, } J = 8.8 \text{ Hz, } 4\text{H}), 6.88 (d, } J = 8.7 \text{ Hz, } 4\text{H}), 6.81 (d, } J = 8.9 \text{ Hz, } 4\text{H}), 6.74 (d, } J = 8.7 \text{ Hz, } 4\text{H}), 3.81 (s, } 12\text{H}). \]

2,3,4,5-Tetra-p-tolycyclopenta-2,4-dienone (3k) was synthesized using the general procedure starting from 220 mg of 1,3-di-p-tolylpropan-2-one (1 mmol), 477 mg of 1,2-di-p-tolyethane-1,2-dione (2 mmol) and 61 mg of KOH (1.1 mmol) in 3 mL of refluxing ethanol during 4h. After removal of the ethanol and work up followed by purification by silica gel chromatography using CH\(_2\)Cl\(_2\) and pentane, 3k was isolated as a dark purple solid (163 mg, 39%).
$^1$H NMR (CDCl$_3$, 300.13 MHz) $\delta$ (ppm): 7.13 (d, $J = 8.2$ Hz, 4H), 7.04 (d, $J = 8.0$ Hz, 4H), 6.97 (d, $J = 7.9$ Hz, 4H), 6.81 (d, $J = 8.1$ Hz, 4H), 2.31 (s, 12H).$^{14}$

**m.p.** = 246.1-246.2°C (lit.$^{14}$ 250-251°C).

2,3,4,5-Tetrakis(4-bromophenyl)cyclopenta-2,4-dienone (3l) was synthesized using the general procedure starting from 391 mg of 1,3-bis(4-bromophenyl)propan-2-one (1.1 mmol), 391 mg of 1,2-bis(4-bromophenyl)ethane-1,2-dione (1.1 mmol ) and 60 mg of KOH (1.1 mmol ) in 3 mL of refluxing ethanol during 1h. After filtration, 3l was isolated as a dark purple solid (418 mg, 56%).

$^1$H NMR (CDCl$_3$, 300.13 MHz) $\delta$ (ppm): 7.40 (d, $J = 8.7$ Hz, 4H), 7.37 (d, $J = 8.7$ Hz, 4H), 7.08 (d, $J = 8.4$ Hz, 4H), 6.77 (d, $J = 8.3$ Hz, 4H).$^{15}$

**m.p.** = 311.5-312.2°C (litt.$^6$ 312.5-313.5°C).

7,9-Diphenyl-8H-cyclopenta[a]acenaphthylen-8-one (6a) was synthesized using the general procedure starting from 0.911 g of acenaphthenequinone (5 mmol), 1.050 g of dibenzylketone (5 mmol) and 0.280 g of KOH (5 mmol) in 25 mL of refluxing ethanol during 3h. After filtration, 6a was isolated as a dark blue solid (1.580 g, 89%).

$^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ (ppm): 8.02-7.99 (m, 2H), 7.81-7.79 (m, 2H), 7.77-7.75 (m, 4H), 7.54-7.50 (m, 2H), 7.57-7.44 (m, 4H), 7.36-7.32 (m, 2H).$^{16}$

**m.p.** = 288.9-295.3°C (litt.$^{17}$ 289.0°C).

7,9-Bis(4-methoxyphenyl)-8H-cyclopenta[a]acenaphthylen-8-one (6b) was synthesized using the general procedure starting from 200 mg of acenaphthenequinone (1.1 mmol), 270 mg of 1,3-bis(4-methoxyphenyl)propan-2-one (1 mmol) and 56 mg of KOH (1 mmol) in 7
mL of refluxing ethanol during 1h. After filtration, 6b was isolated as a dark green solid (396 mg, 95%).

\[ \text{\textsuperscript{1}H NMR (CDCl}_3, \text{ 400 MHz} \delta (ppm) : \] 8.04-8.02 (m, 2H), 7.85-7.83 (m, 2H), 7.79 (d, \( J = 8.8 \text{ Hz}, 4H \)), 7.60-7.56 (m, 2H), 7.06 (d, \( J = 8.8 \text{ Hz}, 4H \)), 3.90 (s, 6H).

\[ \text{\textsuperscript{13}C NMR (CDCl}_3, \text{ 100 MHz} \delta (ppm) : \] 203.09, 160.01, 153.10, 144.95, 132.56, 132.25, 130.77, 128.71, 127.63, 124.44, 121.57, 120.80, 114.50, 55.73.

MS (DCI, Methane) \( m/z \) (\%) : 444.88 (M+C\textsubscript{2}H\textsubscript{5}\textsuperscript{+}, 14), 416.99 (M+H\textsuperscript{+}, 100).

IR (ATR) \( \nu \) (cm\textsuperscript{-1}) : 3036, 2993, 2829, 2359, 1702, 1609, 1510, 1301, 1250, 1172, 1122, 1110, 1036, 821, 773.

m.p. = 108.1-108.2°C

HRMS (M+Na\textsuperscript{+}, ESI) \( m/z \) calcd for C\textsubscript{29}H\textsubscript{20}O\textsubscript{3} : 439.1305 ; found : 439.1304.

7,9-Di-\textit{p}-tolyl-8H-cyclopenta[\textit{a}]acenaphthylene-8-one (6c) was synthesized using the general procedure starting from 273 mg of acenaphthenequinone (1.5 mmol), 238 mg of 1,3-di-\textit{p}-tolylpropan-2-one (1 mmol) and 56 mg of KOH (1 mmol) in 5 mL of refluxing ethanol during 2h. After filtration, 6c was isolated as a dark blue solid (263 mg, 61%).

\[ \text{\textsuperscript{1}H NMR (CDCl}_3, \text{ 300.13 MHz} \delta (ppm) : \] 8.05 (d, \( J = 7.1 \text{ Hz}, 2H \)), 7.85 (d, \( J = 8.3 \text{ Hz}, 2H \)), 7.73 (d, \( J = 8.0 \text{ Hz}, 4H \)), 7.58 (dd, \( J = 8.1, 7.3 \text{ Hz}, 2H \)), 7.33 (d, \( J = 7.9 \text{ Hz}, 4H \)), 2.44 (s, 6H).

m.p. = 322.4-322.5°C (litt.\textsuperscript{18}225.0°C).

7,9-Bis(4-chlorophenyl)-8H-cyclopenta[\textit{a}]acenaphthylene-8-one (6d) was synthesized using the general procedure starting from 146 mg of of acenaphthenequinone (0.8 mmol), 222 mg of 1,3-bis(4-chlorophenyl)propan-2-one (0.8 mmol ) and 45 mg of KOH (0.8 mmol) in 4 mL of refluxing ethanol during 1h. After filtration, 6d was isolated as a dark blue solid (284 mg, 84%).
Spectroscopic data couldn’t be collected due to low solubility of the product in usual NMR solvents.\(^{19}\)

**m.p.** = 268.2-268.3°C.

\[
\begin{array}{c}
\text{Cl} \\
\text{Cl}
\end{array}
\]

7,9-Bis(4-bromophenyl)-8H-cyclopenta[a]acenaphthylen-8-one (6e) was synthesized using the general procedure starting from 222 mg of acenaphthenquinone (1.2 mmol), 298 mg of 1,3-bis(4-bromophenyl)propan-2-one (0.8 mmol) and 45 mg of KOH (0.8 mmol) in 5 mL of refluxing ethanol during 2h. After filtration, 6e was isolated as a dark blue solid (269 mg, 65%).

Spectroscopic data couldn’t be collected due to low solubility of the product in usual NMR solvents.\(^ {19}\)

**m.p.** = 264.2-265.3°C.

\[
\begin{array}{c}
\text{Br} \\
\text{Br}
\end{array}
\]

2-Bromomaleimide was synthesized using a procedure first described by Baker and coll.\(^{20}\) modified as follows:

To a stirring solution of 1.940 g of maleimide (20 mmol) in 16 mL of chloroform was added a solution of 1.02 mL of bromine (3.196 g, 20 mmol) in 16 mL of chloroform; the mixture was then refluxed for 1h. The obtained suspension was then cooled to room temperature and the white solid was filtered, washed with chloroform and dried to yield 2.532 g of crude 2,3-dibromosuccinimide (49%).

Crude 2,3-dibromosuccinimide (9.86 mmol) was dissolved in 16 mL of THF, 828 mg of NaHCO\(_3\) (9.86 mmol) and 1 mL of water were added. The mixture was then refluxed for 2h before being cooled to room temperature. The mixture was then extracted with AcOEt; the organic layer was washed with water (x3), brine and dried over anhydrous Na\(_2\)SO\(_4\). Evaporation under vacuum of the solvents yielded 1.584 g of 2-bromomaleimide (45%) as a pale yellow solid.
**General procedure for the [4+2] cycloaddition of tetracyclones and acecyclones with 2-bromomaleimide.**

A suspension of tetracyclone or acecyclone and 2-bromomaleimide was refluxed in bromobenzene for the indicated time. The reaction was followed by TLC. After complete conversion of the starting material the mixture was cooled to room temperature. If the product precipitates it was eventually filtered and washed with pentane before being dried under vacuum. Otherwise, the mixture was directly put on a flash chromatographic column and eluted first with pentane to remove bromobenzene and then with the indicated solvents.

5,6-Bis(4-methoxyphenyl)-4,7-diphenylsoindoline-1,3-dione (4b) was synthesized using the general procedure starting from 222 mg of tetracyclone 3b (0.50 mmol) and 92 mg of bromomaleimide (0.53 mmol) heated in 3 mL of bromobenzene for 24h. After filtration, 4b was isolated as a pale yellow solid (195 mg, 76%).

**1H NMR (CDCl₃, 300 MHz) δ (ppm) :** 7.36 (bs, 1H), 7.24-7.21 (m, 6H), 7.12-7.08 (m, 4H), 6.63 (d, J = 8.5 Hz, 4H), 6.44 (d, J = 8.6 Hz, 4H), 3.63 (s, 6H).

**13C NMR (CDCl₃, 100 MHz) δ (ppm) :** 167.05, 158.06, 148.49, 140.30, 136.07, 132.23, 130.78, 130.22, 128.79, 127.71, 127.65, 112.93, 55.28.

**MS (DCI, Methane) m/z (%) :** 552.36 (M+C₃H₅⁺, 3), 540.20 (M+C₂H₅⁺, 23), 512.19 (M+H⁺, 100).

**IR (ATR) ν (cm⁻¹) :** 3183, 3060, 1770, 1755, 1710, 1607, 1513, 1464, 1441, 1403, 1350, 1291, 1243, 1173, 1102, 1055, 1031, 839, 740, 699.

**m.p. = 347.1-347.2°C.**

**HRMS (M+Na⁺, ESI) m/z calcd for C₃₄H₂₅NO₄Na :** 534.1676; found : 534.1670.

4,7-Diphenyl-5,6-di-p-tolylsoindoline-1,3-dione (4c) was synthesized using the general procedure starting from 206 mg of tetracyclone 3c (0.50 mmol) and 92 mg of
bromomaleimide (0.53 mmol) heated in 5 mL of bromobenzene during 40h. After purification over silica gel using CH$_2$Cl$_2$/AcOEt, 4c was isolated as a pale yellow solid (239 mg, 99%).

$^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ (ppm): 7.37 (bs, 1H), 7.21-7.19 (m, 6H), 7.11-7.09 (m, 4H), 6.69 (d, $J = 7.9$ Hz, 4H), 6.60 (d, $J = 8.0$ Hz, 4H), 2.10 (s, 6H).

$^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ (ppm): 167.03, 148.70, 140.16, 136.05, 136.04, 135.33, 130.91, 130.22, 128.79, 128.04, 127.63, 127.60, 21.42.

MS (DCI, Methane) m/z (%): 520.01 (M+C$_3$H$_5^+$, 6), 508.04 (M+C$_2$H$_5^+$, 22), 480.15 (M+H$^+$, 100).

IR (ATR) $\nu$ (cm$^{-1}$): 3189, 3064, 1772, 1712, 1514, 1445, 1421, 1353, 1182, 1104, 1053, 1020, 832, 736, 697.

m.p. = 368.9-369.0 °C.

HRMS (M+Na$^+$, ESI) m/z calcld for C$_{34}$H$_{25}$NO$_2$Na: 502.1777 ; found: 502.1773.

5,6-Bis(4-fluorophenyl)-4,7-diphenylisoindoline-1,3-dione (4d) was synthesized using the general procedure starting from 168 mg of tetracyclone 3d (0.40 mmol) and 73 mg of bromomaleimide (0.42 mmol) heated in 3 mL of bromobenzene during 22h. After filtration, 4d was isolated as a grey solid (161 mg, 83%).

$^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ (ppm): 7.45 (ps, 1H), 7.24-7.23 (m, 6H), 7.10-7.08 (m, 4H), 6.71-6.67 (m, 4H), 6.65-6.60 (m, 4H).

$^{19}$F NMR (CDCl$_3$, 376 Mhz) $\delta$ (ppm): -115.35 (s, 2F).

$^{13}$C NMR (CDCl$_3$, 400 MHz) $\delta$ (ppm): 166.65, 162.79, 160.33 (d, $J = 247.2$ Hz), 147.59, 140.24, 135.52, 134.15, 132.65, 132.57 (d, $J = 8.1$ Hz), 130.12, 129.26, 127.99, 127.90, 114.83, 114.62 (d, 21.7 Hz).

MS (DCI, Methane) m/z (%): 527.86 (M+C$_3$H$_5^+$, 6), 515.93 (M+C$_2$H$_5^+$, 35), 487.94 (M+H$^+$, 100).

IR (ATR) $\nu$ (cm$^{-1}$): 3661, 3507, 3177, 3071, 1773, 1712, 1602, 1508, 1355, 1222, 1158, 1106, 1095, 1055, 1015, 846, 802, 768, 740, 697.

m.p. = 309.0-309.1°C.

HRMS (M+Na$^+$, ESI) m/z calcld for: C$_{32}$H$_{19}$F$_2$NO$_2$Na: 510.1276 ; found: 510.1270.
5,6-Bis(4-bromophenyl)-4,7-diphenylisoindoline-1,3-dione (4e) was synthesized using the general procedure starting from 271 mg of tetracyclone 3e (0.50 mmol) and 92 mg of bromomaleimide (0.53 mmol) heated in bromobenzene during 23h. After filtration, 4e was isolated as a white solid (265 mg, 87%).

\[ \text{H NMR (400 MHz, CDCl}_3 \text{) \( \delta \) (ppm): 7.43 (ps, 1H), 7.25-7.21 (m, 6H), 7.09-7.06 (m, 4H), 7.07 (d, \( J = 8.4 \text{ Hz} \), 4H), 6.60 (d, \( J = 8.4 \text{ Hz} \), 4H).} \]

\[ \text{\textsuperscript{13}C NMR (100 MHz, CDCl}_3 \text{) \( \delta \) (ppm): 166.17, 146.66, 139.74, 136.67, 134.90, 132.20, 130.52, 129.72, 129.05, 127.77, 127.60, 121.01.} \]

\[ \text{MS (DCI, Methane) m/z (%): 639.70 (M+C}_2\text{H}_5^+, [\text{81}Br, \text{79}Br], 12), 637.70 (M+C}_2\text{H}_5^+, [\text{79}Br, \text{81}Br], 30), 635.70 (M+C}_2\text{H}_5^+, [\text{79}Br, \text{79}Br], 18), 611.81 (M+H^+, [\text{81}Br, \text{81}Br], 29), 609.82 (M+H^+, [\text{79}Br, \text{81}Br], 100), 607.82 (M+H^+, [\text{79}Br, \text{79}Br], 41).} \]

\[ \text{IR (ATR) } \nu \text{ (cm}^{-1}) : 3160, 3106, 1769, 1711, 1488, 1446, 1417, 1355, 1100, 1057, 1072, 1008, 841, 764, 738, 699.} \]

\[ \text{m.p. > 380}^\circ\text{C.} \]

Anal. Calcd for \( \text{C}_{32}\text{H}_{19}\text{Br}_{2}\text{NO}_2 \text{ C, 63.08; H, 3.15; N, 2.30. Found : C, 62.84; H, 2.81; N, 2.37.} \]

4,7-Bis(4-methoxyphenyl)-5,6-diphenylisoindoline-1,3-dione (4f) was synthesized using a modified general procedure starting from 133 mg of tetracyclone 3f (0.30 mmol) and 54 mg of bromomaleimide (0.31 mmol) (0.5 eq. of bromomaleimide were added after 24h of reaction) heated in 3 mL of bromobenzene during 72h. After filtration, 4f was isolated as a yellow solid (133 mg, 87%).

\[ \text{H NMR (400 MHz, CDCl}_3 \text{) \( \delta \) (ppm): 7.52 (bs, 1H), 7.01 (d, \( J = 8.7 \text{ Hz} \), 4H), 6.91-6.89 (m, 6H), 6.74-6.71 (m, 8H), 3.75 (s, 6H).} \]

\[ \text{\textsuperscript{13}C NMR (100 MHz, CDCl}_3 \text{) \( \delta \) (ppm): 167.18, 159.06, 148.75, 139.69, 138.58, 131.53, 131.09, 129.08, 127.92, 127.38, 126.57, 113.17, 55.37.} \]

\[ \text{MS (ESI) m/z (%): 1045.2 (2M+Na}^+, 32), 534.1 (M+Na}^+, 100), 512.0 (M+H}^+, 18).} \]

\[ \text{IR (ATR) } \nu \text{ (cm}^{-1}) : 3468, 3415, 3185, 3055, 2838, 1770, 1716, 1610, 1515, 1406, 1350, 1290, 1250, 1175, 1104, 1025, 847, 825, 747, 697.} \]
m.p. = 277.2-281.9°C.

HRMS (M+Na⁺, ESI) m/z calcd for C₃₄H₂₅NO₄Na : 534.1676 ; found : 534.1669.

5,6-Diphenyl-4,7-di-p-tolylisoidoline-1,3-dione (4g) was synthesized using the general procedure starting from of 215 mg tetracyclone 3g (0.33 mmol, polluted by 0.37 mmol of residual benzyl) and 65 mg of bromomaleimide (0.36 mmol) heated in 2 mL of bromobenzene during 20h. After purification over silica gel using CH₂Cl₂/AcOEt, 4g was isolated as a beige solid (121 mg, 77%).

\[ \text{1H NMR (CDCl}_3, 400 MHz) \delta (ppm) : 7.37 (bs, 1H), 6.99 (bs, 8H), 6.90-6.88 (m, 6H), 6.74-6.73 (m, 4H), 2.28 (s, 6H). \]

\[ \text{13C NMR (CDCl}_3, 100 MHz) \delta (ppm) : 167.10, 148.57, 140.02, 138.54, 137.27, 132.75, 131.09, 130.12, 129.09, 128.42, 127.29, 126.53, 21.65. \]

IR (ATR) ν (cm⁻¹) : 3190, 3055, 1770, 1715, 1413, 1353, 1253, 1186, 1105, 1055, 818, 744, 699.

m.p. = 295.7-295.8°C.

HRMS (M+Na⁺, ESI) m/z calcd for C₃₄H₂₅NO₂Na : 502.1777 ; found : 502.1771.

4,7-Bis(4-chlorophenyl)-5,6-diphenylisoidoline-1,3-dione (4h) was synthesized using a modified general procedure starting from 181 mg of tetracyclone 3h (0.40 mmol ) and 73 mg of bromomaleimide (0.41 mmol) (0.2 eq of bromomaleimide were added after 30h of reaction) heated in 4 mL of bromobenzene. After filtration, 4h was isolated as a beige solid (161 mg, 77%).

\[ \text{1H NMR (CDCl}_3, 300.13 MHz) \delta (ppm) : 7.43 (bs, 1H), 7.18 (d, J = 8.5 Hz, 4H), 7.04 (d, J = 8.5 Hz, 4H), 6.95-6.90 (m, 6H), 6.74-6.69 (m, 4H). \]

\[ \text{13C NMR (CDCl}_3, 100 MHz) \delta (ppm) : 166.71 148.70, 139.02, 137.83, 134.05, 133.98, 131.59, 130.92, 129.08, 128.06, 127.62, 127.03. \]

MS (DCI, Methane) m/z (%) : 560.00 (M+C₃H₅⁺, [³⁵Cl, ³⁷Cl], 4), 559.94 (M+C₃H₅⁺, [³⁵Cl, ³⁷Cl], 7), 549.94 (M+C₃H₅⁺, [³⁵Cl, ³⁷Cl], 20), 547.90 (M+C₂H₅⁺, [³⁵Cl, ³⁷Cl], 24), 521.96 (M+H⁺, [³⁵Cl, ³⁷Cl], 78), 520.01 (M+H⁺, [³⁵Cl, ³⁷Cl], 100).
IR (ATR) ν (cm⁻¹) : 3177, 3054, 1770, 1714, 1495, 1397, 1350, 1189, 1088, 1052, 1014, 837, 741.

m. p. = 365.0-365.1°C.

HRMS (M+Na⁺, ESI) m/z calcd for C₃₂H₁₉Cl₂NO₂Na : 542.0685 ; found : 542.0680.

4,7-Bis(4-bromophenyl)-5,6-diphenylisoindoline-1,3-dione (4i) was synthesized using a modified general procedure starting from 163 mg of tetracyclone 3i (0.30 mmol) and 55 mg of bromomaleimide (0.32 mmol) (0.24 mmol of bromomaleimide were added after 16h of reaction) heated in 3 mL of bromobenzene. After purification over silica gel using AcOEt and CH₂Cl₂, 4i was isolated as a white solid (121 mg, 66%).

1H NMR (CDCl₃, 300.13 MHz) δ (ppm) : 7.42 (bs, 1H), 7.33 (d, J = 8.3 Hz, 4H), 6.98 (d, J = 8.3 Hz, 4H), 6.94-6.91 (m, 6H), 6.72-6.69 (m, 4H).

13C NMR (CDCl₃, 60 MHz) δ (ppm) : 166.37, 148.29, 138.65, 137.44, 134.18, 131.53, 130.63, 130.57, 128.68, 127.29, 126.71, 121.93.

MS (DCI, Methane) m/z (%) : 637.72 (M+C₂H₅⁺, [⁷⁹Br, ⁸¹Br], 20), 611.74 (M+H⁺, [⁸¹Br, ⁸¹Br], 54), 609.75 (M+H⁺, [⁷⁹Br, ⁸¹Br], 100), 607.79 (M+H⁺, [⁷⁹Br, ⁷⁹Br], 54).

IR (ATR) ν (cm⁻¹) : 3168, 3054, 2383, 2296, 1769, 1716, 1573, 1492, 1407, 1355, 1181, 1070, 1055, 1009, 817, 741, 700.

m.p. = 345.8-349.1°C.

HRMS (M+Na⁺, ESI) m/z calcd for C₃₂H₁₉Br₂NO₂Na : 631.9653 ; found : 631.9654.

4,5,6,7-Tetrakis(4-methoxyphenyl)isoindoline-1,3-dione (4j) was synthesized using a modified general procedure starting from 203 mg of of tetracyclone 3j (0.40 mmol, polluted by 0.08 mmol of residual 1,2-bis(4-methoxyphenyl)ethane-1,2-dione) and 78 mg of bromomaleimide (0.44 mmol) (0.5 eq. of bromomaleimide were added after 10h) heated in 4 mL of bromobenzene during 24h. After purification over silica gel using CH₂Cl₂/AcOEt, 4j was isolated as a yellow solid (207 mg, 91%).
\[ \delta (ppm) : 7.41 (bs, 1H), 7.00 (d, J = 8.8 \text{ Hz}, 4H), 6.74 (d, J = 8.8 \text{ Hz}, 4H), 6.61 (d, J = 8.9 \text{ Hz}, 4H), 7.46 (d, J = 8.8 \text{ Hz}, 4H), 3.77 (s, 6H), 3.64 (s, 6H). \]

\[ \delta (ppm) : 167.25, 158.97, 157.98, 148.73, 139.95, 132.25, 131.54, 131.06, 128.87, 128.23, 113.21, 112.98, 55.40, 55.30. \]

\[ m/z (\%) : 594.1 (M+Na^+, 30), 572.1 (M+H^+, 100). \]

\[ \nu (cm^{-1}) : 3176, 3069, 2833, 1771, 1715, 1609, 1350, 1288, 1245, 1174, 1102, 1058, 831, 804, 768. \]

\[ m.p. = 325.2-325.3^\circ C. \]

**Analysis**: Calculated for C\(_{36}\)H\(_{29}\)NO\(_6\) : C, 75.65; H, 5.12; N, 2.46. **Found**: C, 75.92, H, 5.12, N, 2.47.

**4,5,6,7-Tetra-p-tolylisoindoline-1,3-dione (4k)** was synthesized using the general procedure starting from 132 mg of tetracyclone 3k (0.30 mmol) and 53 mg of bromomaleimide (0.31 mmol) heated in 3 mL of bromobenzene. After purification over silica gel using CH\(_2\)Cl\(_2\)/AcOEt, 4k was isolated as a beige solid (123 mg, 81%).

\[ \delta (ppm) : 7.31 (bs, 1H), 7.00 (d, J = 8.2 \text{ Hz}, 4H), 6.97 (d, J = 8.3 \text{ Hz}, 4H), 6.69 (d, J = 7.9 \text{ Hz}, 4H), 2.29 (s, 6H), 2.12 (s, 6H). \]

\[ \delta (ppm) : 167.15, 148.77, 140.17, 137.11, 135.90, 135.58, 133.01, 130.94, 130.11, 128.88, 128.40, 128.01, 21.69, 21.43. \]

**IR (ATR) v (cm\(^{-1}\)) : 3198, 1771, 1714, 1416, 1350, 1253, 1181, 1101, 1052, 1018, 848, 828, 759.**

**m.p. = 346.0-346.1^\circ C.**

**HRMS (M+Na^+, ESI) m/z calculated for C\(_{36}\)H\(_{29}\)NO\(_2\)Na : 530.2090 ; found : 530.2086.**

**4,5,6,7-Tetrakis(4-bromophenyl)isoindoline-1,3-dione (4l)** was synthesized using a modified general procedure starting from 350 mg of tetracyclone 3l (0.50 mmol) and 89 mg of bromomaleimide (0.51 mmol) (0.5 eq. of bromomaleimide were added after 24h of
reaction) heated in 5 mL of bromobenzene during 38h. After filtration, 41 was isolated as a grey solid (361 mg, 94%).

\[ \text{\textsuperscript{1}H NMR (DMSO-}\text{d6, 400 MHz)} \delta (\text{ppm}): 11.33 \text{ (s, 1H), 7.41 (d, } J = 8.4 \text{ Hz, 4H), 7.20 (d, } J = 8.4 \text{ Hz, 4H), 7.08 (d, } J = 8.4 \text{ Hz, 4H).} \]

\[ \text{\textsuperscript{13}C NMR (DMSO-}\text{d6, 100 MHz)} \delta (\text{ppm}): 168.38, 146.51, 137.99, 137.68, 135.46, 133.41, 132.85, 131.07, 131.00, 129.98, 121.68, 120.96. \]

MS (ESI) m/z (%): 763.7 (M-H\textsuperscript{+}, \textsuperscript{79}Br, \textsuperscript{79}Br, \textsuperscript{79}Br, \textsuperscript{81}Br, 80), 765.7 (M-H\textsuperscript{+}, \textsuperscript{79}Br, \textsuperscript{79}Br, \textsuperscript{81}Br, \textsuperscript{81}Br, 100), 767.7 (M-H\textsuperscript{+}, \textsuperscript{79}Br, \textsuperscript{81}Br, \textsuperscript{81}Br, \textsuperscript{81}Br, 60).

IR (ATR) ν (\text{cm}^{-1}): 3166, 3064, 2757, 1772, 1714, 1491, 1422, 1350, 1071, 1050, 1009, 855, 826, 763.

\[ \text{m.p. > 380°C.} \]

HRMS (M+Na\textsuperscript{+}, ESI) m/z calcd for C\textsubscript{32}H\textsubscript{17}Br\textsubscript{4}NO\textsubscript{2}Na: 789.7848; found: 789.7840.

7,11-Diphenyl-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (7a) was synthesized using the general procedure starting from 178 mg of acecyclone 6a (0.50 mmol) and 91 mg of bromomaleimide (0.53 mmol) heated in 4 mL of bromobenzene during 20h. After filtration and purification over silica gel using AcOEt, 7a was isolated as a yellow solid (164 mg, 78%).

\[ \text{\textsuperscript{1}H NMR (DMSO, 400 MHz)} \delta (\text{ppm}): 11.14 \text{ (s, 1H), 8.03 (d, } J = 8.1 \text{ Hz, 2H), 7.64-7.62 (m, 6H), 7.57-7.55 (m, 4H), 7.48 (dd, } J = 7.8, 7.5 \text{ Hz, 2H), 6.72 (d, } J = 7.2 \text{ Hz, 2H).} \]

\[ \text{\textsuperscript{13}C NMR (DMSO, 100 MHz)} \delta (\text{ppm}): 173.86, 147.83, 141.36, 140.82, 139.71, 138.20, 135.24, 134.74, 134.67, 134.61, 134.53, 134.38, 134.11, 130.62. \]

MS (DCI, Methane) m/z (%): 500.44 (M+C\textsubscript{3}H\textsubscript{5}\textsuperscript{+}, 6), 452.04 (M+C\textsubscript{3}H\textsubscript{4}\textsuperscript{+}, 24), 424.09 (M+H\textsuperscript{+}, 100).

IR (ATR) ν (\text{cm}^{-1}): 3194, 3058, 1762, 1706, 1442, 1425, 1360, 1315, 1302, 1115, 1024, 826, 767, 759, 693.

\[ \text{m.p. = 343.3-343.4°C.} \]

HRMS (M+Na\textsuperscript{+}, ESI) m/z calcd for C\textsubscript{30}H\textsubscript{17}NO\textsubscript{2}Na: 446.1152; found: 446.1149.
7,11-Bis(4-methoxyphenyl)-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (7b) was synthesized using the general procedure starting from 166 mg of acecyclone 6b (0.40 mmol) and 72 mg of bromomaleimide (0.41 mmol) heated in 3 mL of bromobenzene during 23h. After purification over silica gel using CH₂Cl₂/AcOEt, 7b was isolated as a yellow solid (191 mg, 98%).

\[ \text{1H NMR (CDCl}_3, 400 \text{ MHz}) \delta (\text{ppm}): 7.88 (d, J = 8.2 \text{ Hz}, 2H), 7.50 (d, J = 8.7 \text{ Hz}, 4H), 7.46-7.42 (m, 2H), 7.17 (d, J = 8.7 \text{ Hz}, 4H), 7.03 (d, J = 7.2 \text{ Hz}, 2H), 4.00 (s, 6H). \]

\[ \text{13C NMR (CDCl}_3, 100 \text{ MHz}) \delta (\text{ppm}): 167.45, 160.21, 143.99, 136.03, 134.96, 133.60, 130.43, 129.95, 129.07, 128.82, 128.38, 127.82, 125.88, 114.57, 55.67. \]

\[ \text{MS (DCI, Methane) } m/z (\%) : 484.18 (M+H}^+, 100), 512.09 (M+C}_2H}_5^+, 14), 523.93 (M+C}_3H}_5^+, 2). \]

\[ \text{IR (ATR)} \nu (\text{cm}^{-1}) : 3637, 3190, 3062, 2835, 1763, 1708, 1609, 1524, 1437, 1426, 1358, 1318, 1287, 1171, 1115, 1026, 826, 763. \]

\[ \text{m.p. = 321.0-321.1°C.} \]

\[ \text{HRMS (M+Na}^+, \text{ ESI) } m/z \text{ calcd for C}_{32}H_{21}NO}_4Na : 506.1363 ; \text{ found : 506.1358.} \]

7,11-Di-p-tolyl-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (7c) was synthesized using the general procedure starting from 115 mg of acecyclone 6c (0.30 mmol) and 54 mg of bromomaleimide (0.31 mmol) heated in 3 mL of bromobenzene during 23h. After purification over silica gel using CH₂Cl₂/AcOEt, 7c was isolated as a yellow solid (119 mg, 85%).

\[ \text{1H NMR (CDCl}_3, 400 \text{ MHz}) \delta (\text{ppm}): 7.85 (d, J = 8.2 \text{ Hz}, 2H), 7.46-7.39 (m, 10H), 7.30 (bs, 1H), 6.96 (d, J = 7.2 \text{ Hz}, 2H), 2.56 (s, 6H). \]

\[ \text{13C NMR (CDCl}_3, 100 \text{ MHz}) \delta (\text{ppm}): 167.41, 143.74, 138.71, 136.32, 134.92, 134.91, 133.61, 132.73, 129.91, 129.87, 128.96, 128.79, 128.36, 125.89, 21.98. \]

\[ \text{MS (DCI, Methane) } m/z (\%) : 452.06 (M+H}^+, 100), 480.01 (M+C}_2H}_5^+, 16), 492.03 (M+C}_3H}_5^+, 4). \]
IR (ATR) ν (cm⁻¹) : 3194, 3057, 2980, 1768, 1708, 1425, 1358, 1317, 1113, 1044, 1021, 812, 762, 721.

m.p. = 315.0-315.1°C.

HRMS (M+Na⁺, ESI) m/z calcd for C₃₂H₂₁NO₂Na : 474.1465 ; found : 474.1461.

7,11-Bis(4-chlorophenyl)-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (7d) was synthesized using the general procedure starting from 213 mg of acecyclone 6d (0.50 mmol) and 91 mg of bromomaleimide (0.51 mmol) heated in 5 mL of bromobenzene during 21h. After filtration, 7d was isolated as a yellow solid (220 mg, 89%).

\[
\begin{align*}
1^H \text{NMR (DMSO-d}_6, \text{ 400 MHz) } & \delta \text{ (ppm)} : 11.23 \text{ (s, 1H), 8.09 (d, } J = 8.2 \text{ Hz, 2H), 7.73 (d, } J = 7.7 \text{ Hz, 4H), 7.64 (d, } J = 7.6 \text{ Hz, 4H), 7.63-7.60 \text{ (m, 2H), 6.91 (d, } J = 6.9 \text{ Hz, 2H).} \\
13^C \text{NMR (DMSO-d}_6, \text{ 100 MHz) } & \delta \text{ (ppm) : 168.84, 142.71, 135.07, 134.64, 134.47, 134.30, 133.22, 131.74, 130.37, 129.94, 129.92, 129.70, 129.37, 125.73.} \\
\text{MS (ESI) m/z} \text{ (%) : 491.9 (M-H⁺, [}^{35}\text{Cl, }^{37}\text{Cl], 83), 489.9 (M-H⁺, [}^{35}\text{Cl, }^{35}\text{Cl], 100).} \\
\text{IR (ATR) ν (cm⁻¹) : 3165, 3057, 1767, 1707, 1598, 1475, 1428, 1359, 1317, 1114, 1096, 1081, 1013, 829, 815, 760.} \\
m.p. = 365.0-365.1°C.
\end{align*}
\]

HRMS (M+Na⁺, ESI) m/z calcd for C₃₀H₁₅Cl₂NO₂ : 514.0372 ; found : 515.0370.

7,11-Bis(4-bromophenyl)-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (7e) was synthesized using the general procedure starting from 154 mg of acecyclone 6e (0.30 mmol) and of bromomaleimide (0.31 mmol) heated in 3 mL of bromobenzene during 24h. After purification over silica gel using CH₂Cl₂/AcOEt, 7e was isolated as a yellow solid (176 mg, quant.).
$^1$H NMR (DMSO-d$_6$, 400 MHz) $\delta$ (ppm) : 11.24 (s, 1H), 8.09 (d, $J = 8.2$ Hz, 2H), 7.87 (d, $J = 8.3$ Hz, 4H), 7.63 (dd, $J = 7.9$, 7.5 Hz, 2H), 7.58 (d, $J = 8.3$ Hz, 4H), 6.92 (d, $J = 7.2$ Hz, 2H).

$^{13}$C NMR (DMSO-d$_6$, 60 MHz) $\delta$ (ppm) : 168.81, 142.61, 135.44, 134.61, 134.44, 133.20, 132.59, 132.01, 130.35, 129.91, 129.88, 129.36, 125.71, 122.93.

MS (DCI, Methane) $m/z$ (%) : 580.34 (M+H$^+$, [79Br, 79Br], 53), 582.26 (M+H$^+$, [79Br, 81Br], 100), 584.21 (M+H$^+$, [81Br, 81Br], 49), 608.10 (M+C$_2$H$_5^+$, [79Br, 79Br], 18), 610.14 (M+C$_2$H$_5^+$, [79Br, 81Br], 28), 612.14 (M+C$_2$H$_5^+$, [81Br, 81Br], 13).

IR (ATR) $\nu$ (cm$^{-1}$) : 3167, 3054, 2922, 1764, 1707, 1591, 1426, 1357, 1317, 1113, 1068, 1039, 1010, 829, 814, 772, 760.

Decomposition occurred above 350°C before melting.

HRMS (M+Na$^+$, ESI) $m/z$ calcd for C$_{30}$H$_{15}$Br$_2$NO$_2$Na : 603.9344 ; found : 603.9342.
References