Supporting Information

Rhodium(III)-catalysed oxidative annulation through C-H activation: Expedient access to 8-aminoisocoumarin by weak coordination

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1. General remarks

All catalysts and starting materials were purchased from commercial sources and used as received. Unless otherwise noted, all commercially obtained reagents and solvents were used as received. Anhydrous solvents such as DMF, DMSO, and toluene were purchased from commercial sources in a sealed bottle and used as received. Thin layer chromatogram (TLC) was performed on precoated aluminium sheets of silica gel 60 F254 of 0.2 mm thickness and visualized via UV. Melting points were determined in capillary tubes and are uncorrected. Nuclear Magnetic Resonance spectra were recorded on 400 MHz (¹H NMR) and 100 MHz (¹³C NMR) instrument in CDCl₃ solutions referenced to TMS or solvent residual peak. UV-visible absorption spectra were measured using UV-visible spectrophotometer. The steady state fluorescence measurements were measured using fluorescence spectrophotometer.

2. General procedure for synthesis of alkynes (2)

Following the literature procedure, Pd(PPh₃)₂Cl₂ (105 mg, 0.15 mmol), 1,4-bis(diphenyl phosphino)butane (128 mg, 0.30 mmol), aryl halides (6.00 mmol), and propiolic acid (212 mg, 3.0 mmol) were combined with DBU (913 mg, 6.0 mmol) in a round bottom flask. DMSO (15 mL) was added and the reaction was maintained in the oxygen atmosphere. The resulting mixture was placed in an oil bath at 80 °C for 3 h. The reaction was poured in saturated ammonium chloride solution, and extracted with ethyl acetate 3× 25 mL. The combined ethyl acetate layer was washed with brine solution, dried over anhydrous sodium sulphate, filtered, and the solvent was removed under vacuum. The resulting crude product
was purified using flash column chromatography in silica gel 100-200 mesh using 5% EtOAc in pet ether.

3. General experimental procedure for synthesis 8-amino isocoumarins

\[
\begin{align*}
\text{R}_1 \quad \text{R}_2 & \quad + \quad \text{R}_3 \equiv \text{R}_4 \quad \xrightarrow{[\text{RhCp}^*\text{Cl}_2]_2, \text{Cu(}\text{OAc}_2, \text{DMF, } 110^\circ \text{C, } 24 \text{ h}} \\
\text{R}_1 \quad \text{R}_2 & \quad \text{NH}_2 \quad \text{R}_3 \quad \text{R}_4
\end{align*}
\]

A oven-dried screw cap reaction tube with a magnetic stir bar was charged with \([\text{RhCp}^*\text{Cl}_2]_2\) (5.4 mg, 2.5 mol%), isatoic anhydride (81.6 mg, 0.50 mmol), diphenylacetylene (44.6 mg, 0.25 mmol), Cu(OAc)_2 (weight, 1 equiv.), and DMF (3.0 mL). The tube was sealed with a Teflon-coated screw cap and the reaction solution was heated at 110 °C for 24 h. The mixture was then cooled to ambient temperature, diluted with 10 mL of ethyl acetate, filtered through a celite pad, and washed with 20-40 mL of ethyl acetate. The combined organic phases were concentrated under reduced pressure, and the residue was purified by column chromatography on silica gel to provide the desired product.

4. Competitive experiments

(a) Intermolecular competitive reaction between isatoic anhydride 1a with alkynes 2c and 2d
A oven-dried screw cape reaction tube with a magnetic stir bar was charged with a solution of isatoic anhydride 1a (50 mg, 0.30 mmol), 4-methoxy-diphenylacetylene 2c (73 mg, 0.30 mmol), 4-fluro-diphenylacetylene 2d (65 mg, 0.30 mmol), Cu(OAc)$_2$ (60 mg, 1.0 equiv.), and [RhCp*Cl$_2$]$_2$ (4.6 mg, 2.5 mol%). The tube was sealed with a Teflon-coated screw cap and the reaction solution was heated at 110 °C for 24 h. The mixture was then cooled to ambient temperature, diluted with 10 mL of ethyl acetate, filtered through a celite pad, and washed with 10-20 mL of ethyl acetate. Finally, it was dried over anhydrous Na$_2$SO$_4$. The combined organic phases were concentrated under reduced pressure and the residue was purified by column chromatography using EtOAc/Hexane as an eluent on silica gel (100-200 mesh) to provide the desired product 3m (63 mg, 55%) and 3n (34 mg, 31%) in the ratio of 1.8:1.0 (3m:3n).

(b) Intermolecular competitive reaction between 1a with alkynes 2a and 2p

A oven-dried screw cape reaction tube with a magnetic stir bar was charged with a solution of isatoic anhydride 1a (50 mg, 0.30 mmol), diphenylacetylene 2a (55 mg, 0.30 mmol), 4-octyne 2p (50 mg, 0.45 mmol), Cu(OAc)$_2$ (60 mg, 1.0 equiv.), and [RhCp*Cl$_2$]$_2$ (4.6 mg, 2.5 mol%). The tube was sealed with a Teflon-coated screw cap, and the reaction solution was heated at 110 °C for 24 h. The mixture was then cooled to ambient temperature, diluted with 10 mL of ethyl acetate, filtered through a celite pad, and washed with 10-20 mL of ethyl acetate. Finally, it was dried over anhydrous Na$_2$SO$_4$. The combined organic phases were concentrated under reduced pressure, and the residue was purified by column
chromatography using EtOAc/Hexane as the eluent on silica gel (100-200 mesh) to provide the desired product 3a (52 mg, 54%) and 3z (20 mg, 26%). 3a:3z (2.0:1).

(c) Intermolecular competitive reaction between 2a with Isatoic anhydrides 1g and 1i

A oven-dried screw cape reaction tube with a magnetic stir bar was charged with A solution of 2a diphenylacetylene (53 mg, 0.30 mmol), 7-methylsatoic anhydride 1g (53 mg, 0.30 mmol), 7-chlorosatoic anhydride 2a (59 mg, 0.30 mmol), Cu(OAc)₂ (60 mg, 1.0 equiv.) and [Cp*RhCl₂]₂ (4.6 mg, 2.5 mol%). The tube was sealed with a Teflon-coated screw cap and the reaction solution was heated at 120 °C for 24 h. The mixture was then cooled to ambient temperature, diluted with 10 mL of ethyl acetate, filtered through a celite pad, and washed with 10-20 mL of ethyl acetate. Finally, it was dried over anhydrous Na₂SO₄ the combined organic phases were concentrated under reduced pressure and the residue was purified by column chromatography EtOAc/Hexane as the eluant on silica gel (100-200 mesh) to provide the desired product 3g (29 mg, 30%) and 3i (62 mg, 59%). 3g:3i (0.5:1).

5. Regioselectivity of the reaction
A oven-dried screw cape reaction tube with a magnetic stir bar was charged with [RhCp*Cl₂]₂ (5.4 mg, 2.5 mol%), isatoic anhydride 81 mg (0.5 mmol), 1-Phenyl-1-propyne 88 mg (0.5 mmol), Cu(OAc)₂ and DMF (3.0 mL). The tube was sealed with a Teflon-coated screw cap and the reaction solution was heated at 110 °C for 24 h. The mixture was then cooled to ambient temperature, diluted with 10 mL of ethyl acetate, filtered through a celite pad, and washed with 20-40 mL of ethyl acetate. The combined organic phases were concentrated under reduced pressure and the residue was purified by column chromatography on silica gel to provide the desired product was isolated 84% of yield. The analysed the 1D NOE spectrum of compound 3v to further conform the regioselectivity.

1D NOE spectrum of compound 3v
Reaction with nonsymmetrical aromatic alkynes

(a) Isatoic anhydride 1a with 1-methoxy-4-(p-tolylethynyl)benzene

A oven-dried screw cap reaction tube with a magnetic stir bar was charged with [RhCp*Cl]$_2$ (5.4 mg, 2.5 mol%), isatoic anhydride 81 mg (0.5 mmol), 1-methoxy-4-(p-tolylethynyl)benzene 110 mg (0.5 mmol), Cu(OAc)$_2$ 90 mg (1.0 equiv) and DMF (3.0 mL). The tube was sealed with a Teflon-coated screw cap and the reaction solution was heated at 110 °C for 24 h. The mixture was then cooled to ambient temperature, diluted with 10 mL of ethyl acetate, filtered through a celite pad, and washed with 20-40 mL of ethyl acetate. The combined organic phases were concentrated under reduced pressure and the residue was purified by column chromatography on silica gel to provide the desired product was isolated pale yellow solid 78% of yield. Provided a mixture of isomers analysed by the $^1$H spectrum of compound (3ad+3ad'=1:1).

(a) Isatoic anhydride 1a with 1-fluoro-4-((4-methoxyphenyl)ethynyl)benzene
A oven-dried screw cape reaction tube with a magnetic stir bar was charged with [RhCp*Cl₂]₂ (5.4 mg, 2.5 mol%), isatoic anhydride 81 mg (0.5 mmol), 1-fluoro-4-((4-methoxyphenyl)ethynyl)benzene 111 mg (0.5 mmol), Cu(OAc) 90 mg (1.0 equiv) and DMF (3.0 mL). The tube was sealed with a Teflon-coated screw cap and the reaction solution was heated at 110 °C for 24 h. The mixture was then cooled to ambient temperature, diluted with 10 mL of ethyl acetate, filtered through a celite pad, and washed with 20-40 mL of ethyl acetate. The combined organic phases were concentrated under reduced pressure and the residue was purified by column chromatography on silica gel to provide the desired product was isolated pale yellow solid 72% of yield. Provided a mixture of isomers analysed by the 

6. Gram scale synthesize of 8-amino-3,4-diphenyl-1H-isocoumarin

An oven-dried 100 mL screw cape reaction tube with a magnetic stir bar was charged with isatoic anhydride 1.0 g (6.2 mmol), diphenylacetylene 1.09 g, [RhCp*Cl₂]₂ 0.113 g (3 mol %), Cu(OAc)₂ 1.11 g and DMF (20 mL). The tube was sealed with a Teflon-coated screw cap and the reaction solution was heated at 110 °C for 24 h. The mixture was then cooled to ambient temperature, diluted with 10 mL of ethyl acetate, filtered through a celite pad, and
washed with 20-40 mL of ethyl acetate, it was dried over anhydrous Na$_2$SO$_4$ the combined organic phases were concentrated under reduced pressure and the residue was purified by column chromatography EtOAc/Hexane as the eluent on silica gel (100-200 mesh) to provide the desired product colourless solid 3a 1.42 g was obtained 74% of yield.

7. Procedure for Sandmeyer reaction

![Chemical reaction diagram](attachment:image.png)

To the stirred solution of 8-amino isocoumarin in acetonitrile was added $p$-toluene sulphonic acid (2.5 equiv) and the resulting suspension was cooled to 0 °C. After a 10 min, the solution of sodium nitrite (2.0 equiv) and potassium iodide (2.5 equiv) in water were added dropwise to the reaction mixture and slowly bring the reaction mixture to the ambient temperature. The resulting combination allowed stirring for additional 3 h and the progress of reaction was monitored by TLC. After disappearance of amino isocoumarin, the reaction mass was quenched with saturated sodium thiosulphate. The aqueous layer was extracted with ethyl acetate (3 times) and combined organic layers were dried with anhydrous sodium sulphate, distilled under reduced pressure by rotavapor. The crude was purified by silica gel column chromatography (hexane/ethyl acetate) to obtained 8-iodo isocoumarin.
8. ORTEP diagram of compound 3m

![ORTEP diagram of compound 3m]

CCDC: 1815391

9. CO evoluation test:

The Palladium Chloride (PdCl$_2$) (200 mg) was dissolved in of conc. HCl and diluted with 10 mL of distilled water. Phosphomolybdic acid saturated solution in water was prepared separately. These two solutions were then mixed in a separate vial in 1:2 (PMA:PdCl$_2$) ratio. Some narrow pieces of filter papers were then dipped in this PMAPdCl$_2$ solution and then these were dried at rt. In 25 mL round bottom flask, isatoic anhydride 1a (1.0 mmol), alkyne 2a (1.0 mmol), [RhCp*Cl$_2$]$_2$ (2.5 mol %), and Cu(OAc)$_2$ (1.0 equiv) were taken and 5 mL DMF was added in to this reaction mixture. One strip was fitted inside the round bottom flask with the septum. The reaction mixture was then heated at 110 °C. After few hours of heating, it was observed that the yellow colour of the strip was changed to dark-blue colour, this is indicating the evolution of CO gas from the reaction mixture.

![Before reaction stripe inside](image1)

![After reaction stripe inside the round bottom flask](image2)

![Stripe removed](image3)
10. Spectral Data for synthesized compounds

8-amino-3,4-diphenyl-1H-isochromen-1-one (3a)

Yield: 85 %, White solid; Melting point: 134-136 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta 7.36 - 7.30 \text{ (m, 3H)}, 7.25 \text{ (dd, } J = 8.0, 1.9 \text{ Hz, 3H)}, 7.22 - 7.10 \text{ (m, 5H)}, 6.59 \text{ (dd, } J = 8.2, 1.0 \text{ Hz, 1H)}, 6.39 - 6.15 \text{ (m, 3H)} \text{ ppm}; \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta 163.94, 151.60, 150.18, 140.45, 135.57, 135.21, 133.18, 131.41, 129.23, 128.99, 128.84, 128.00, 127.90, 117.75, 114.17, 113.31, 103.61 \text{ ppm}; \text{ HRMS m/z (ESI): calculated for C}_{21}\text{H}_{16}\text{NO}_{2} [M + H]^+ 314.1181, found 314.1177.}

8-amino-5-methyl-3,4-diphenyl-1H-isochromen-1-one (3b)

Yield: 77 %, White solid; Melting point: 122-124 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta 7.25 - 7.21 \text{ (m, 3H)}, 7.17 - 7.08 \text{ (m, 8H)}, 6.60 \text{ (d, } J = 8.4 \text{ Hz, 1H)}, 6.34 - 6.20 \text{ (m, 2H)}, 1.51 \text{ (s, 3H)} \text{ ppm}; \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta 164.48, 150.95, 150.67, 140.89, 137.70, 136.38, 134.00, 132.01, 129.56, 128.40, 128.31, 127.70, 127.68, 122.11, 118.11, 114.94, 104.52, 22.74 \text{ ppm}; \text{ HRMS m/z (ESI): calculated for C}_{22}\text{H}_{18}\text{NO}_{2} [M + H]^+ 328.1338, found 328.1331.}

8-amino-5-fluoro-3,4-diphenyl-1H-isochromen-1-one (3c)

Yield: 68 %, White solid; Melting point: 180-182 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta 7.28 \text{ (ddd, } J = 4.1, 3.2, 1.7 \text{ Hz, 3H)}, 7.23 \text{ (dq, } J = 9.0, 2.5 \text{ Hz, 4H)}, 7.21 - 7.13 \text{ (m, 3H)}, 7.07 \text{ (dd, } J =
11.8, 9.0 Hz, 1H), 6.62 (dd, J = 9.0, 3.6 Hz, 1H), 6.23 (s, 2H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 163.20, 163.16, 151.07, 150.26, 148.47, 147.84, 136.25, 132.94, 130.68, 130.64, 129.34, 128.77, 128.00, 127.70, 127.47, 125.66, 124.76, 124.51, 114.99, 114.92, 113.92, 113.89, 102.93 ppm; HRMS m/z (ESI): calculated for C$_{21}$H$_{15}$FNO$_2$ [M + H]$^+$ 332.1087, found 332.1081.

8-amino-5-chloro-3,4-diphenyl-1H-isochromen-1-one (3d)

![3d](image)

Yield: 72 %, White solid; Melting point: 162-164 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.28 (d, J = 8.9 Hz, 1H), 7.21 (dd, J = 9.2, 6.9, 5.2 Hz, 3H), 7.13 (ddd, J = 6.9, 4.7, 1.7 Hz, 6H), 6.60 (d, J = 8.9 Hz, 1H), 6.46 (s, 2H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 163.50, 152.37, 151.22, 139.67, 135.95, 134.75, 133.53, 132.09, 129.59, 128.76, 128.05, 127.72, 127.69, 117.04, 116.44, 115.79, 104.78 ppm; HRMS m/z (ESI): calculated for C$_{21}$H$_{15}$ClNO$_2$ [M + H]$^+$ 348.0791, found 348.0788.

8-amino-5-bromo-3,4-diphenyl-1H-isochromen-1-one (3e)

![3e](image)

Yield: 66 %, White solid; Melting point: 138-140 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.32 – 7.28 (m, 3H), 7.25 – 7.18 (m, 3H), 7.18 – 7.06 (m, 6H), 6.57 (dd, J = 8.3, 1.0 Hz, 1H), 6.34 – 6.14 (m, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 163.94, 151.59, 150.16, 140.43, 135.57, 135.19, 133.17, 131.40, 129.21, 128.98, 128.83, 127.99, 127.89, 117.74, 114.17, 113.30, 103.58 ppm;
HRMS m/z (ESI): calculated for \( \text{C}_{21}\text{H}_{15}\text{BrNO}_2 \) \([\text{M + H}]^{+}\) 392.0286, found 392.0304.

8-amino-5-iodo-3,4-diphenyl-1H-isochromen-1-one (3f)

Yield: 63 %, White solid; Melting point: 148-150 °C; \(^1\text{H} \text{NMR} \) (400 MHz, CDCl\(_3\)) \( \delta \) 7.30 – 7.27 (m, 2H), 7.22 (ddd, \( J = 6.4, 3.7, 1.9 \) Hz, 3H), 7.17 – 7.13 (m, 2H), 7.12 – 7.05 (m, 3H), 6.56 (dd, \( J = 8.2, 0.9 \) Hz, 1H), 6.34 – 6.12 (m, 3H) ppm; \(^{13}\text{C} \text{NMR} \) (100 MHz, CDCl\(_3\)) \( \delta \) 163.92, 151.59, 150.15, 140.42, 135.55, 135.19, 133.17, 131.40, 129.21, 128.97, 128.82, 127.99, 127.88, 117.74, 114.17, 113.28, 103.58, 77.48, 77.16, 76.84 ppm; HRMS m/z (ESI): calculated for \( \text{C}_{21}\text{H}_{14}\text{INO}_2 \) 439.0069, found 439.0068.

8-amino-6-methyl-3,4-diphenyl-1H-isochromen-1-one (3g)

Yield: 85 %, Pale yellow solid; Melting point: 160-162 °C; \(^1\text{H} \text{NMR} \) (400 MHz, CDCl\(_3\)) \( \delta \) 7.34 – 7.26 (m, 3H), 7.23 – 7.19 (m, 2H), 7.17 – 7.06 (m, 5H), 6.40 (s, 1H), 6.17 (s, 2H), 6.03 (s, 1H), 2.11 (s, 3H) ppm; \(^{13}\text{C} \text{NMR} \) (100 MHz, CDCl\(_3\)) \( \delta \) 163.84, 151.60, 150.26, 146.69, 140.31, 135.29, 133.30, 131.44, 129.22, 128.95, 128.74, 127.93, 127.84, 117.62, 114.69, 114.36, 101.62, 22.31 ppm; HRMS m/z (ESI): calculated for \( \text{C}_{22}\text{H}_{15}\text{NO}_2 \) \([\text{M + H}]^{+}\) 328.1338, found 328.1341.

8-amino-6-methoxy-3,4-diphenyl-1H-isochromen-1-one (3h)
Yield: 89 %, Pale green solid; Melting point: 176-178 °C; \( ^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 7.69 – 7.63 (m, 3H), 7.61 – 7.57 (m, 2H), 7.54 – 7.43 (m, 5H), 6.68 (s, 2H), 6.44 (d, \( J = 2.3 \) Hz, 1H), 6.15 (d, \( J = 2.3 \) Hz, 1H), 3.98 (s, 3H) ppm; \( ^{13}\)C NMR (100 MHz, CDCl\(_3\)) \( \delta \) 165.29, 163.50, 153.78, 150.56, 142.37, 135.14, 133.23, 131.34, 129.23, 128.99, 128.81, 127.99, 127.84, 117.46, 100.76, 98.23, 97.94, 77.48, 77.16, 76.84, 55.27 ppm; HRMS \((\text{m/z (ESI)})\): calculated for C\(_{22}\)H\(_{18}\)NO\(_3\) [M + H]\(^+\) 344.1287, found 344.1291.

8-amino-6-chloro-3,4-diphenyl-1H-isochromen-1-one (3i)

\( ^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 7.42 – 7.37 (m, 3H), 7.30 – 7.26 (m, 2H), 7.24 – 7.14 (m, 5H), 6.64 (d, \( J = 1.9 \) Hz, 1H), 6.40 (s, 2H), 6.26 (d, \( J = 1.9 \) Hz, 1H) ppm; \( ^{13}\)C NMR (100 MHz, CDCl\(_3\)) \( \delta \) 163.34, 152.36, 151.34, 142.13, 141.95, 134.45, 132.81, 131.31, 129.25, 129.22, 129.16, 128.33, 127.96, 116.98, 113.44, 113.24, 102.14, 77.48, 77.16, 76.84 ppm; HRMS \((\text{m/z (ESI)})\): calculated for C\(_{21}\)H\(_{15}\)ClNO\(_2\) [M + H]\(^+\) 348.0791, found 348.0793.

8-amino-5,6-dimethoxy-3,4-diphenyl-1H-isochromen-1-one (3j)

Yield: 81 %, Pale yellow solid; Melting point: 192-194 °C; \( ^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 7.18 – 7.10 (m, 7H), 7.10 – 7.03 (m, 3H), 6.29 (s, 2H), 6.16 (s, 1H), 3.78 (s, 3H), 2.83 (s, 3H) ppm; \( ^{13}\)C NMR (100 MHz, CDCl\(_3\)) \( \delta \) 163.73, 160.15, 151.22, 150.41, 137.31, 135.83, 133.85, 131.49, 131.32, 129.69, 128.47, 127.62, 127.39, 126.77, 115.53, 97.90, 96.71, 60.97, 55.83
5-amino-1,2-diphenyl-4H-benzo[f]isochromen-4-one (3k)

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\text{1H NMR (400 MHz, CDCl}_3) \delta 7.49 (d, J = 8.2 \text{ Hz}, 1\text{H}), 7.36 – 7.15 (m, 11\text{H}), 7.10 – 7.01 (m, 2\text{H}), 6.65 (ddd, J = 9.0, 6.7, 1.5 \text{ Hz}, 1\text{H}), 6.15 (s, 2\text{H}) \text{ ppm; 13C NMR (100 MHz, CDCl}_3) \delta 163.75, 146.00, 139.48, 139.32, 138.20, 133.74, 131.60, 129.67, 129.19, 128.92, 128.76, 128.37, 128.01, 127.84, 126.32, 122.45, 121.20, 118.42, 113.62, 110.97, 108.03, 77.48, 77.16, 76.84 \text{ ppm; HRMS m/z (ESI)}: \text{calculated for C}_{23}\text{H}_{20}\text{NO}_4 [M+H]^+ 374.1392, \text{found 374.1387.}
\]

8-amino-3,4-di-p-tolyl-1H-isochromen-1-one (3l)

Yield: 88 %, White solid ; Melting point: 146-148 °C; \text{1H NMR (400 MHz, CDCl}_3) \delta 7.21 – 7.17 (m, 2\text{H}), 7.17 – 7.10 (m, 4\text{H}), 7.07 – 7.01 (m, 2\text{H}), 6.92 (d, J = 8.2 \text{ Hz}, 2\text{H}), 6.55 (dd, J = 8.2, 1.0 \text{ Hz}, 1\text{H}), 6.31 – 6.13 (m, 3\text{H}), 2.33 (s, 3\text{H}), 2.21 (s, 3\text{H}) \text{ ppm; 13C NMR (100 MHz, CDCl}_3) \delta 164.09, 151.53, 150.21, 140.87, 138.82, 137.64, 135.49, 132.30, 131.21, 130.42, 129.76, 129.08, 128.65, 117.14, 113.89, 113.29, 103.64, 77.48, 77.16, 76.84, 21.48, 21.41 \text{ ppm; HRMS m/z (ESI)}: \text{calculated for C}_{23}\text{H}_{18}\text{NO}_2 [M+H]^+ 364.1338, \text{found 364.1333.}

8-amino-3,4-bis(4-methoxyphenyl)-1H-isochromen-1-one (3m)

8-amino-3,4-bis(4-methoxyphenyl)-1H-isochromen-1-one (3m)
Yield: 84 %, White solid; Melting point: 120-122 °C; \( ^1H \) NMR (400 MHz, CDCl\(_3\)) \( \delta \) 7.25 – 7.20 (m, 3H), 7.14 – 7.07 (m, 2H), 6.93 – 6.87 (m, 2H), 6.71 – 6.64 (m, 2H), 6.57 (dd, \( J = 8.2, 1.0 \) Hz, 1H), 6.27 (dd, \( J = 7.8, 1.0 \) Hz, 3H), 3.82 (s, 3H), 3.73 (s, 3H) ppm; \( ^{13}C \) NMR (100 MHz, CDCl\(_3\)) \( \delta \) 164.08, 159.85, 159.29, 151.55, 150.15, 141.11, 135.50, 132.50, 130.63, 127.57, 125.76, 116.21, 114.57, 113.74, 113.39, 113.11, 103.55, 55.40, 55.33 ppm; HRMS m/z (ESI): calculated for C\(_{23}\)H\(_{20}\)NO\(_4\) [M+H]+ 374.1392, found 374.1404.

8-amino-3,4-bis(4-fluorophenyl)-1H-isochromen-1-one (3n)

\( ^1H \) NMR (400 MHz, CDCl\(_3\)) \( \delta \) 7.24 – 7.17 (m, 3H), 7.15 – 7.09 (m, 2H), 7.07 – 6.99 (m, 2H), 6.86 – 6.79 (m, 2H), 6.58 (dd, \( J = 8.3, 1.0 \) Hz, 1H), 6.24 (s, 2H), 6.18 (dd, \( J = 7.7, 0.9 \) Hz, 1H) ppm; \( ^{13}C \) NMR (100 MHz, CDCl\(_3\)) \( \delta \) 164.07, 163.82, 163.66, 161.58, 161.35, 151.70, 149.57, 140.18, 135.72, 133.12, 133.04, 131.27, 131.18, 130.94, 130.91, 129.19, 129.16, 116.69, 116.39, 116.17, 115.30, 115.08, 114.41, 113.04, 103.41, 77.48, 77.16, 76.84 ppm; HRMS m/z (ESI): calculated for C\(_{21}\)H\(_{14}\)F\(_2\)NO\(_2\) [M+H]+ 350.0993, found 350.0988.

8-amino-3,4-bis(4-chlorophenyl)-1H-isochromen-1-one (3o)

Yield: 71 %, White solid; Melting point: 164-166 °C; \( ^1H \) NMR (400 MHz, CDCl\(_3\)) \( \delta \) 7.34 – 7.27 (m, 2H), 7.22 (dd, \( J = 15.9, 7.9 \) Hz, 1H), 7.17 – 7.06 (m, 6H), 6.59 (dd, \( J = 8.3, 1.0 \) Hz, 1H), 6.41 – 6.18 (m, 2H), 6.16 (dd, \( J = 7.8, 1.0 \) Hz, 1H) ppm; \( ^{13}C \)
NMR (101 MHz, CDCl$_3$) δ 163.51, 151.73, 149.26, 139.80, 135.75, 135.15, 134.34, 133.42, 132.69, 131.36, 130.48, 129.51, 128.39, 116.97, 114.62, 113.06, 103.35, 77.48, 77.16, 76.84 ppm; HRMS m/z (ESI): Calculated for C$_{21}$H$_{13}$Cl$_2$NO$_2$ [M+ H]$^+$ 382.0402, found 382.0399.

8-amino-3,4-bis(4-bromophenyl)-1H-isochromen-1-one (3p)

Yield: 62 %, White solid; Melting point: 140-142 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.56 – 7.51 (m, 1H), 7.41 – 7.25 (m, 4H), 7.24 – 7.13 (m, 3H), 7.12 – 7.07 (m, 1H), 6.66 (d, $J$ = 8.2 Hz, 1H), 6.31 (s, 2H), 6.24 (d, $J$ = 7.8 Hz, 1H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) δ 151.73, 139.73, 135.77, 133.02, 133.00, 132.68, 132.49, 131.38, 131.36, 130.71, 130.49, 129.54, 128.42, 123.56, 122.52, 117.03, 114.66, 113.09, 103.36 ppm; HRMS m/z (ESI): Calculated for C$_{21}$H$_{14}$Br$_2$NO$_2$ [(M+2)+H]$^+$ 471.9371, found 471.9377.

8-amino-3,4-bis(4-nitrophenyl)-1H-isochromen-1-one (3q)

Yield: 33 %, Pale yellow solid; Melting point: 212-214 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ 8.36 – 8.24 (m, 2H), 8.24 – 8.14 (m, 1H), 8.09 – 8.04 (m, 1H), 7.83 (d, $J$ = 8.8 Hz, 1H), 7.47 – 7.38 (m, 3H), 7.08 – 6.95 (m, 1H), 6.73 (dd, $J$ = 8.3, 0.9 Hz, 1H), 6.37 (s, 2H), 6.22 – 6.04 (m, 1H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) δ 162.74, 152.02, 141.55, 138.57, 138.45,
8-amino-3,4-di(naphthalen-1-yl)-1H-isochromen-1-one (3r)

Yield: 80 %, White solid; Melting point: 180-182 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.08 (d, \(J = 8.4\) Hz, 1H), 7.86 – 7.73 (m, 3H), 7.70 – 7.61 (m, 2H), 7.52 – 7.38 (m, 4H), 7.24 – 7.11 (m, 4H), 7.01 (dd, \(J = 8.2, 7.1\) Hz, 1H), 6.68 (dd, \(J = 8.3, 1.0\) Hz, 1H), 6.38 (s, 2H), 5.97 (dd, \(J = 7.7, 1.0\) Hz, 1H) ppm; \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 164.13, 152.14, 151.73, 140.17, 135.86, 133.54, 133.44, 133.00, 132.28, 131.71, 130.70, 129.75, 128.77, 128.52, 128.35, 127.94, 126.79, 126.48, 126.09, 126.00, 125.73, 125.61, 124.65, 118.48, 114.42, 113.80, 103.68 ppm; HRMS m/z (ESI): calculated for C\(_{21}\)H\(_{14}\)N\(_3\)O\(_6\) [M + H]\(^+\) 404.0883, found 404.0889.

8-amino-3,4-bis(2-methoxyphenyl)-1H-isochromen-1-one (3s)

Yield: 81 %, White solid; Melting point: 194-196 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)+DMSO-d\(_6\)) \(\delta\) 7.13 – 7.02 (m, 3H), 6.99 (dd, \(J = 7.9, 1.8\) Hz, 1H), 6.86 (dd, \(J = 7.4, 1.8\) Hz, 1H), 6.71 – 6.64 (m, 2H), 6.64 – 6.57 (m, 2H), 6.51 (dd, \(J = 8.3, 1.1\) Hz, 1H), 6.36 (s, 2H), 6.01 (d, \(J = 7.8\) Hz, 1H), 3.52 (d, \(J = 8.5\) Hz, 6H) ppm; \(^{13}\)C NMR (100 MHz, CDCl\(_3\)+DMSO-d\(_6\)) \(\delta\) 164.11, 157.42, 157.02, 151.52, 149.24, 139.40, 135.05, 131.82, 130.59, 130.31, 129.06, 123.44, 122.63, 120.07, 119.51, 115.66, 113.55,
8-amino-3,4-di-m-tolyl-1H-isocoumarin-1-one (3t)

Yield: 85 %, White solid; Melting point: 128-130 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.26 – 7.18 (m, 3H), 7.14 (d, $J$ = 7.6 Hz, 1H), 7.06 – 6.93 (m, 5H), 6.60 (dd, $J$ = 8.2, 0.9 Hz, 1H), 6.42 – 6.12 (m, 3H), 2.30 (s, 3H), 2.19 (s, 3H). ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 164.05, 151.53, 150.09, 140.69, 138.60, 137.52, 135.53, 135.18, 133.05, 131.88, 129.74, 129.58, 128.48, 128.68, 128.43, 127.63, 126.40, 117.71, 114.01, 113.42, 103.63, 21.55, 21.47 ppm; HRMS m/z (ESI): Calculated for C$_{23}$H$_{20}$NO$_4$ [M+ H]$^+$ 374.1398, found 374.1398.

8-amino-6-chloro-3,4-di-p-tolyl-1H-isochromen-1-one (3u)

Yield: 76 %, yellow solid; Melting point: 168-170 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.22 – 7.17 (m, 4H), 7.10 – 7.06 (m, 2H), 6.99 (d, $J$ = 8.1 Hz, 2H), 6.60 (d, $J$ = 1.9 Hz, 1H), 6.38 (s, 2H), 6.26 (d, $J$ = 1.9 Hz, 1H), 2.40 (s, 3H), 2.27 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 163.47, 152.30, 151.33, 142.35, 142.01, 139.20, 137.97, 131.52, 131.08, 130.04, 129.96, 129.10, 128.69, 116.38, 113.16, 102.13, 21.49, 21.41 ppm; HRMS m/z
(ESI): calculated for C\textsubscript{23}H\textsubscript{19}ClNO\textsubscript{2} [M + H]\textsuperscript{+} 376.1104, found 376.1088.

8-amino-4-methyl-3-phenyl-1H-isochromen-1-one (3v)

Yield: 84 %, White solid; Melting point: 138-140 °C; \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta\) 7.61 – 7.54 (m, 2H), 7.50 – 7.40 (m, 4H), 6.77 (dd, \(J = 7.7, 1.0\) Hz, 1H), 6.67 (dd, \(J = 8.3, 1.0\) Hz, 1H), 6.30 (s, 2H), 2.21 (s, 3H) ppm; \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}) \(\delta\) 164.16, 151.73, 150.56, 140.25, 135.68, 133.59, 129.60, 129.26, 128.30, 114.13, 110.78, 109.87, 103.83, 14.11 ppm; HRMS m/z (ESI): calculated for C\textsubscript{16}H\textsubscript{14}NO\textsubscript{2} [M + H]\textsuperscript{+} 252.1025, found 252.1034.

8-amino-4-ethyl-3-phenyl-1H-isochromen-1-one (3w)

Yield: 74 %, White solid; Melting point: 160-162 °C; \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta\) 7.55 (dd, \(J = 7.7, 2.0\) Hz, 2H), 7.49 – 7.41 (m, 4H), 6.83 (dd, \(J = 7.8, 0.9\) Hz, 1H), 6.67 (dd, \(J = 8.2, 0.9\) Hz, 1H), 6.30 (s, 2H), 2.62 (q, \(J = 7.5\) Hz, 2H), 1.24 (t, \(J = 7.4\) Hz, 3H) ppm; \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}) \(\delta\) 164.10, 151.97, 150.73, 139.06, 135.65, 133.80, 129.33, 129.07, 128.42, 115.97, 113.98, 110.89, 104.34, 20.58, 14.70 ppm; HRMS m/z (ESI): calculated for C\textsubscript{17}H\textsubscript{16}NO\textsubscript{2} [M + H]\textsuperscript{+} 266.1181, found 266.1174.

8-amino-3,4-dimethyl-1H-isochromen-1-one (3x)

\textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta\) 7.42 (t, \(J = 8.0\) Hz, 1H), 6.64 (dd, \(J = 15.0, 8.0\) Hz, 2H), 6.26 (s, 2H), 2.29 (s, 3H), 2.09 (s,
3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 164.48, 151.71, 149.39, 140.18, 135.63, 113.31, 109.80, 108.37, 103.73, 17.30, 12.82 ppm; HRMS m/z (ESI): calculated for C$_{11}$H$_{12}$NO$_2$ [M + H]$^+$ 190.0868, found 190.0861.

8-amino-3,4-diethyl-1H-isochromen-1-one (3y)

Yield: 53 %, Colourless fluid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.39 (t, $J$ = 8.0 Hz, 1H), 6.70 (dd, $J$ = 7.8, 1.0 Hz, 1H), 6.58 (dd, $J$ = 8.2, 1.0 Hz, 1H), 6.24 (s, 2H), 2.56 (qd, $J$ = 7.5, 4.7 Hz, 4H), 1.26 (t, $J$ = 7.5 Hz, 3H), 1.16 (t, $J$ = 7.5 Hz, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 164.61, 154.33, 151.93, 139.25, 135.57, 113.72, 113.18, 109.87, 104.19, 24.09, 19.82, 14.28, 12.64 ppm; HRMS m/z (ESI): calculated for C$_{13}$H$_{16}$NO$_2$ [M+H]$^+$ 218.1181, found 218.1173.

8-amino-3,4-dipropyl-1H-isochromen-1-one (3z)

Yield: 85 %, White solid; Melting point: 166-170 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.38 (t, $J$ = 8.0 Hz, 1H), 6.67 (dd, $J$ = 7.9, 1.0 Hz, 1H), 6.58 (dd, $J$ = 8.2, 0.9 Hz, 1H), 6.25 (s, 2H), 2.55 – 2.45 (m, 4H), 1.77 – 1.67 (m, 2H), 1.56 (dq, $J$ = 15.0, 7.4 Hz, 2H), 0.99 (dt, $J$ = 9.0, 7.4 Hz, 6H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 164.56, 153.44, 151.85, 139.46, 135.50, 113.15, 112.89, 110.06, 104.12, 77.48, 77.16, 76.84, 32.68, 28.72,
22.80, 21.24, 14.34, 13.94 ppm; **HRMS m/z (ESI)**: calculated for C_{15}H_{20}NO_{2} [M+H]^+ 246.1494, found 246.1495.

8-amino-3,4-dibutyl-1H-isochromen-1-one (3aa)

![3aa](image)

Yield: 53 %, Pale yellow fluid; **{1H NMR (400 MHz, CDCl\textsubscript{3})} δ** 7.31 (t, J = 8.0 Hz, 1H), 6.60 (dd, J = 7.9, 0.9 Hz, 1H), 6.51 (dd, J = 8.2, 1.0 Hz, 1H), 6.18 (s, 2H), 2.50 – 2.39 (m, 4H), 1.65 – 1.55 (m, 2H), 1.49 – 1.27 (m, 6H), 0.88 (q, J = 7.2 Hz, 6H) ppm; **{13C NMR (100 MHz, CDCl\textsubscript{3})} δ** 164.58, 153.56, 151.85, 139.50, 135.51, 113.12, 112.87, 110.01, 104.14, 77.48, 77.16, 76.84, 31.78, 30.55, 30.07, 26.46, 23.05, 22.64, 14.08, 14.02 ppm; **HRMS m/z (ESI)**: calculated for C_{17}H_{24}NO_{2} [M+H]^+ 274.1807, found 274.1809.

8-amino-3,4-bis(methoxymethyl)-1H-isochromen-1-one (3ab)

![3ab](image)

Yield: 39 %, Pale yellow fluid; **{1H NMR (400 MHz, CDCl\textsubscript{3})} δ** 7.43 (t, J = 8.0 Hz, 1H), 6.90 (dd, J = 7.7, 1.0 Hz, 1H), 6.66 (dd, J = 8.3, 1.0 Hz, 1H), 6.23 (s, 2H), 4.50 (s, 2H), 4.38 (s, 2H), 3.44 (s, 3H), 3.41 (s, 3H) ppm; **{13C NMR (100 MHz, CDCl\textsubscript{3})} δ** 163.35, 151.71, 151.59, 137.84, 135.81, 114.60, 113.41, 111.21, 104.00, 68.84, 66.29, 58.67, 57.98 ppm; **HRMS m/z (ESI)**: calculated for C_{13}H_{16}NO_{4} [M+H]^+ 250.1079, found 250.1080.

8-amino-3,4-diethyl-6-methyl-1H-isochromen-1-one (3ac)

![3ac](image)
Yield: 68 %, Pale yellow solid; Melting point: 156-158 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 6.48 (s, 1H), 6.40 (s, 1H), 6.19 (s, 2H), 2.52 (p, $J$ = 7.4 Hz, 4H), 2.30 (s, 3H), 1.23 (t, $J$ = 7.5 Hz, 3H), 1.14 (t, $J$ = 7.5 Hz, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 164.45, 154.26, 151.86, 146.41, 139.00, 113.64, 113.45, 110.90, 102.01, 24.00, 22.39, 19.65, 14.24, 12.57 ppm; HRMS m/z (ESI): calculated for C$_{14}$H$_{18}$NO$_2$ [M+H]$^+$ 232.1338, found 232.1335.

8-amino-4-(4-methoxyphenyl)-3-(p-tolyl)-1H-isochromen-1-one & 8-amino-3-(4-methoxyphenyl)-4-(p-tolyl)-1H-isochromen-1-one (3ad)

Yield (1:1): 78 %, Pale yellow solid; Melting point: 154-156 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.34 – 7.26 (m, 5H), 7.23 (dd, $J$ = 8.2, 2.9 Hz, 4H), 7.19 – 7.10 (m, 4H), 7.02 (d, $J$ = 8.1 Hz, 2H), 6.98 – 6.92 (m, 2H), 6.76 – 6.69 (m, 2H), 6.63 (m, 2H), 6.48 – 6.23 (m, 6H), 3.87 (s, 3H), 3.78 (d, $J$ = 0.9 Hz, 3H), 2.42 (s, 3H), 2.30 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 163.96, 163.93, 159.69, 159.14, 151.42, 150.20, 149.83, 140.83, 140.79, 138.66, 137.47, 135.36, 132.33, 132.26, 131.10, 130.50, 130.31, 129.68, 128.94, 128.55, 127.29, 125.58, 116.68, 116.45, 116.42, 114.35, 113.79, 113.62, 113.20, 113.07, 112.98, 103.46, 103.36, 55.26, 55.19, 21.35, 21.28 ppm; HRMS m/z (ESI): calculated for C$_{23}$H$_{20}$NO$_3$ [M+H]$^+$ 358.1443, found 358.1445.
8-amino-4-(4-fluorophenyl)-3-(p-tolyl)-1H-isochromen-1-one & 8-amino-3-(4-fluorophenyl)-4-(p-tolyl)-1H-isochromen-1-one (3ae+3ae')

Yield (1:2): 72 %, Pale yellow solid: Melting point 86-88 °C; 

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.36 – 7.29 (m, 5H), 7.26 – 7.19 (m, 8H), 7.16 – 7.08 (m, 6H), 6.99 – 6.87 (m, 4H), 6.77 – 6.69 (m, 4H), 6.65 (m, \(J = 8.2, 6.2, 1.0\) Hz, 3H), 6.42 – 6.30 (m, 6H), 6.25 (dd, \(J = 7.8, 1.0\) Hz, 3H), 3.87 (s, 3H), 3.79 (s, 6H) ppm; 

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 163.77, 163.69, 163.57, 161.11, 159.86, 159.27, 151.50, 151.47, 150.29, 149.12, 140.53, 140.44, 135.47, 135.45, 133.06, 132.98, 132.27, 131.30, 131.26, 131.08, 131.00, 130.52, 129.34, 129.31, 126.86, 125.26, 117.11, 116.16, 115.94, 115.51, 115.01, 114.80, 114.45, 114.06, 113.82, 113.32, 113.17, 112.65, 103.39, 103.25, 55.27, 55.22 ppm; 

HRMS m/z (ESI): calculated for C\(_{22}\)H\(_{17}\)FNO\(_3\) \([\text{M+H}]^+\) 362.1192, found 362.1196.

8-iodo-3,4-diphenyl-1H-isochromen-1-one (4a)

Yield: 94 %, Colourless solid; 

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.10 (dd, \(J = 5.9, 2.9\) Hz, 1H), 7.35 (dd, \(J = 4.9, 1.9\) Hz, 3H), 7.25 – 7.22 (m, 2H), 7.17 – 7.12 (m, 5H), 7.10 – 7.08 (m, 2H) ppm; 

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 159.33, 151.28, 142.48, 141.36, 134.42, 132.60, 131.41, 131.17, 129.35, 129.29, 129.21, 128.44, 128.03, 126.15, 120.84, 116.79, 96.58, 77.48, 77.16, 76.84 ppm; 

HRMS m/z (ESI): calculated for C\(_{21}\)H\(_{13}\)IO\(_2\) 423.9960, found 423.9959.
### 11. Photophysical properties of compounds

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<th>$\lambda_{\text{max,abs.}}$ (nm) Solution $^a$</th>
<th>$\lambda_{\text{max,emi.}}$ (nm) Solution $^a,c$</th>
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$^a$ In dichloromethane solvent at 5 μM. $^b$ Thin film $^c$ Excitation wavelength: 350-400 nm. $^d$ Excitation wavelength: 350-440 nm.
12. Absorption and fluorescent spectra of synthesized compounds

Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3a.

Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3b.
Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3c.

Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3d.
Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3e.

Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3f.
Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3g.

Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3h.
Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3i.

Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3j.
Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3k.

Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3l.
Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3m.

Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3n.
Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3o.

Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3p.
Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3r.

Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3s.
Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3t.

Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3u.
Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3v.

Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3w.
Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3y.

Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3z.
Normalized absorption (black), Fluorescence in DCM (red) and Fluorescence in thin film (blue) spectra of compound 3ac.
13.AIE Characterization of compound 3a

Absorption spectra of 3a in CH$_3$CN/H$_2$O mixtures with different water fraction (fw), Concentration: 5.0 $\mu$M.

Emission spectra of 3a in CH$_3$CN/H$_2$O mixtures with different water fraction (fw), Concentration: 5.0 $\mu$M.
14. Copies of NMR and HRMS spectrum of synthesized compounds

**$^1$H NMR spectrum of compound 3a in CDCl$_3$**

**$^{13}$C NMR spectrum of compound 3a in CDCl$_3$**
DEPT-135 NMR spectrum of compound 3a in CDCl₃
HRMS spectrum of compound 3a
$^1$H-$^1$H COSY NMR spectrum of compound 3a

$^1$H-$^1$H COSY NMR spectrum of compound 3a Expansion
$^1\text{H}-^{13}\text{C} \text{ HSQC NMR spectrum of compound 3a}$

$^1\text{H}-^{13}\text{C} \text{ HSQC NMR spectrum of compound 3a Expansion}$
HMBC NMR spectrum of compound 3a

HMBC NMR spectrum of compound 3a Expansion
$^{13}$C NMR spectrum of compound 3b in CDCl$_3$

$^1$H NMR spectrum of compound 3b in CDCl$_3$
DEPT-135 NMR spectrum of compound 3b in CDCl₃
HRMS spectrum of compound 3b
$^1$H NMR spectrum of compound 3c in CDCl$_3$

$^{13}$C NMR spectrum of compound 3c in CDCl$_3$
DEPT-135 NMR spectrum of compound 3c in CDCl₃
HRMS spectrum of compound 3c
**1H NMR spectrum of compound 3d in CDCl₃**

**13C NMR spectrum of compound 3d in CDCl₃**
DEPT-135 NMR spectrum of compound 3d in CDCl$_3$
HRMS spectrum of compound 3d
H NMR spectrum of compound 3e in CDCl₃

1³C NMR spectrum of compound 3e in CDCl₃
DEPT-135 NMR spectrum of compound 3e in CDCl₃
**INdian institute of technology Hyderabad**
Dept Of Chemistry
HRMS Report

**Table**

<table>
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<th>Mass</th>
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<td>0.343</td>
<td>Found by formula</td>
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**MS Spectrum**

- **x10 3**
  - Cod 1: C21 H14 Br N O2: FBM Spectrum (0.289, 0.292, 0.279, 0.280, 0.282 ... min) TPK-29-01.1.d.
  - MS/MS Spectrum (C21H14BrN2O2)^+:
    - 392.0304
    - 302.2, 302.4, 302.6, 302.8, 303.0, 303.2, 303.4, 303.6
    - 392.1, 299.4, 394.4, 394.6, 394.8
    - Ratio vs. Mass-to-Charge (ppm)

**MS Spectrum Peak List**

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HRMS spectrum of compound 3e
$^1$H NMR spectrum of compound 3f in CDCl$_3$

$^{13}$C NMR spectrum of compound 3f in CDCl$_3$
DEPT-135 NMR spectrum of compound 3f in CDCl$_3$

HRMS spectrum of compound 3f
H NMR spectrum of compound 3g in CDCl\textsubscript{3}

\begin{align*}
\text{1H NMR spectrum of compound 3g in CDCl}_3
\end{align*}

\begin{align*}
\text{13C NMR spectrum of compound 3g in CDCl}_3
\end{align*}
DEPT-135 NMR spectrum of compound 3g in CDCl₃
INDIAN INSTITUTE OF TECHNOLOGY HYDERABAD
Dept Of Chemistry
HRMS Report

Data File: TFP-DK-31-00.d
Sample Name: TFP-DK-31
Position: F1-E2
Acq Method: HRMS-Dual_ESI_v46 SD NEW_Agilent.m
Acq Date: 09-07-18 06:02 PM
DA Method: SD_0T.m
Sample Group: Infs.
User Name: Acquisition SW
Version: Q-TOF 8.05 B1 (91125.3)

620D series TOF/TOF series Q-TOF 8.05 B1 (91125.3)

Compound Table

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MS Spectrum

Cpd 1: C22 H17 N O2 + ESI EIC[109.1148, 319.1226, 327.1294, 327.1492 ...] Beam Fog=100...

MS Zoomed Spectrum

Cpd 1: C22 H17 N O2 + ESI Spectrum [0.123, 0.157, 0.224, 0.258 ... mm] TFP-DK-31-00 d S...

MS Spectrum Peak List

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NH2

3g
HRMS spectrum of compound 3g
$^1$H NMR spectrum of compound 3h in CDCl$_3$

$^{13}$C NMR spectrum of compound 3h in CDCl$_3$
DEPT-135 NMR spectrum of compound 3h in CDCl₃
HRMS spectrum of compound 3h
1H NMR spectrum of compound 3i in CDCl$_3$

13C NMR spectrum of compound 3i in CDCl$_3$
DEPT-135 NMR spectrum of compound 3i in CDCl₃
HRMS Spectrum of compound 3i
$^{1}H$ NMR spectrum of compound 3j in CDCl$_3$

$^{13}$C NMR spectrum of compound 3j in CDCl$_3$
DEPT-135 NMR spectrum of compound 3j in CDCl$_3$
HRMS spectrum of compound 3j
H NMR spectrum of compound 3k in CDCl₃

13C NMR spectrum of compound 3k in CDCl₃
DEPT-135 NMR spectrum of compound 3k in CDCl₃
HRMS spectrum of compound 3k
H NMR spectrum of compound 3l in CDCl₃

13C NMR spectrum of compound 3l in CDCl₃
DEPT-135 NMR spectrum of compound 3l in CDCl₃
HRMS spectrum of compound 3l
$^1$H NMR spectrum of compound 3m in CDCl$_3$

$^{13}$C NMR spectrum of compound 3m in CDCl$_3$
DEPT-135 NMR spectrum of compound 3m in CDCl₃

$^1H-^1H$ COSY NMR spectrum of compound 3m
$^1$H-$^1$H COSY NMR spectrum of compound 3m Expansion

$^1$H-$^{13}$C HSQC NMR spectrum of compound 3m
$^1H-^{13}C$ HSQC NMR spectrum of compound 3m Expansion

HMBC-NMR spectrum of compound 3m
HMBC-NMR spectrum of compound 3m Expansion
Compound Table

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MS Spectrum

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3m
HRMS spectrum of compound 3m
$^1$H NMR spectrum of compound 3n in CDCl$_3$

$^{13}$C NMR spectrum of compound 3n in CDCl$_3$
DEPT-135 NMR spectrum of compound 3n in CDCl₃
HRMS spectrum of compound 3n
$\text{H NMR spectrum of compound 3o in CDCl}_3$  

$\text{C NMR spectrum of compound 3o in CDCl}_3$  

$\text{H NMR spectrum of compound 3o in CDCl}_3$  

$\text{C NMR spectrum of compound 3o in CDCl}_3$
DEPT-135 NMR spectrum of compound 3o in CDCl$_3$
HRMS spectrum of compound 3o
$^1$H NMR spectrum of compound 3p in CDCl$_3$

$^{13}$C NMR spectrum of compound 3p in CDCl$_3$
**INSTITUTE OF TECHNOLOGY HYDERABAD**  
Dept Of Chemistry  
HRMS Report

**Data File**  
TBP-07-36-08.sdf  
**Sample Name**  
Sample Brand  
**Method**  
TBP-DL-36  
**Sample Weight**  
6.25 g  
**Sample Method**  
HRMS_Sample  
**Sample Dilution**  
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**Sample Volume**  
1 ml  
**Sample Concentration**  
25 mg/ml  
**Sample Type**  
Liquid  
**Sample Description**  
None  
**Sample Notes**  
None  
**Sample Source**  
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**Sample Preparation**  
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**Sample Storage**  
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**Sample Stability**  
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**Compound Table**

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**MS Spectrum**

![MS Spectrum](image)

**MS Ion Spectrum**

![MS Ion Spectrum](image)

**MS Spectrum Peak List**

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<td>M+H+</td>
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**References**

- Agilent Technologies
HRMS spectrum of compound 3p
$^1$H NMR spectrum of compound 3q in CDCl$_3$

$^{13}$C NMR spectrum of compound 3q in CDCl$_3$
INSTITUTE OF TECHNOLOGY HYDERABAD
Dept Of Chemistry
HRMS Report

Data File: TNP-DK-37-01.d
Acquired Time: 2018-08-27 14:35 PM
Sample Name: TNP-DK-37
Acq Method: NMR

Sample Group: 620 series TNP/D330 series
Version: Q-TOP 0.01.1 (08/13/12)

Compound Table

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MS Spectrum

MS Spectrum Peak List

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3q
HRMS spectrum of compound 3q
$^1$H NMR spectrum of compound 3r in CDCl$_3$

$^{13}$C NMR spectrum of compound 3r in CDCl$_3$
DEPT-135 NMR spectrum of compound 3r in CDCl$_3$
HRMS spectrum of compound 3r
H NMR spectrum of compound 3s in CDCl$_3$

$^{13}$C NMR spectrum of compound 3s in CDCl$_3$
DEPT-135 NMR spectrum of compound 3s in CDCl$_3$
### Compound Table

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### Compound Label

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### MS Spectrum

**EIC (350, 1203, 316, 1281, 373, 1300, 373, 1547) Scan Peaks=103...**

### MS Dotted Spectrum

**EIC (350, 1203, 316, 1281, 373, 1300, 373, 1547) Scan Peaks=103...**

### MS Spectrum Peak List

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HRMS spectrum of compound 3s
$\text{H NMR spectrum of compound 3t in CDCl}_3$

$\text{13C NMR spectrum of compound 3t in CDCl}_3$
DEPT-135 NMR spectrum of compound 3t in CDCl₃
INDIAN INSTITUTE OF TECHNOLOGY HYDERABAD
Dept Of Chemistry
HRMS Report

Data File: TQK-DK-38 C:d
Sample Name: TQK-DK-38
Position: P1-C8
Acq Method: HRPS_Dual ESI_VHS_95 SD MDM_Agilent.m
Acq Time: 20-Mai-18 14:55 PM
DA Method: SD_DMix.m
Sample Group: Infos.
User Name: Acquisition SW
Version: Q-TOF B 8.05 G1 (G5125.3)

Compound Table

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<th>Formula</th>
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MS Spectrum

Counts vs. Mass-to-Charge (m/z)

MS Zoned Spectrum

Counts vs. Mass-to-Charge (m/z)

MS Spectrum Peak List

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Agilent Technologies
Page 1 of 2

Printed at: 4:18 PM on:08-08-18
HRMS spectrum of compound 3t
$^1$H NMR spectrum of compound 3u in CDCl$_3$

$^{13}$C NMR spectrum of compound 3u in CDCl$_3$
DEPT-135 NMR spectrum of compound 3u in CDCl$_3$
HRMS spectra of compound 3u
$^{1}$H NMR spectrum of compound 3v in CDCl$_3$

$^{13}$C NMR spectrum of compound 3v in CDCl$_3$
DEPT-135 NMR spectrum of compound 3v in CDCl$_3$

$^1$H-$^1$H COSY NMR spectrum of compound 3v
$^1$H-$^1$H COSY NMR spectrum of compound 3v Expansion

HMBC-NMR spectrum of compound 3v
HMBC-NMR spectrum of compound 3v Expansion

HMBC-NMR spectrum of compound 3v Expansion
$^1$H-$^{13}$C HSQC NMR spectrum of compound 3v

$^1$H-$^{13}$C HSQC NMR spectrum of compound 3v Expansion
$^1H-^{13}C$ HSQC NMR spectrum of compound 3ν Expansion
Qualitative Compound Identification Report

Sample Name: 1058.15 - 556.54 SP-16
Sample Type: Random
Instrument Name: 2551-0K2
Acq Method: Direct Analysis, HPLC
DA Method: Reflux

Compound Table

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Compound Chromatograms

MS Spectrum

Cpd 1: C16H13N2O2: 0.473 + FBF Spectrum (rt: 0.903-0.986 min) RDMKSF16.d

Agilent Technologies
HRMS spectrum of compound 3v
\[ \text{H NMR spectrum of compound 3w in CDCl}_3 \]

\[ \text{13C NMR spectrum of compound 3w in CDCl}_3 \]
DEPT-135 NMR spectrum of compound 3w in CDCl$_3$

$^1$H-$^1$H COSY NMR spectrum of compound 3w
$^1$H-$^1$H COSY NMR spectrum of compound 3w Expansion

$^1$H-$^1$H COSY NMR spectrum of compound 3w Expansion
HMBC-NMR spectrum of compound 3w

HMBC-NMR spectrum of compound 3w Expansion
$^1$H-$^{13}$C HSQC NMR spectrum of compound 3w

$^1$H-$^{13}$C HSQC NMR spectrum of compound 3w Expansion
\(^{1}H^{13}C\) HSQC NMR spectrum of compound 3w Expansion
HRMS spectrum of compound 3w
\textbf{\textsuperscript{1}H NMR spectrum of compound 3x in CDCl$_3$}

\textbf{\textsuperscript{13}C NMR spectrum of compound 3x in CDCl$_3$}
DEPT-135 NMR spectrum of compound 3x in CDCl$_3$
## INDIAN INSTITUTE OF TECHNOLOGY HYDERABAD
Dept Of Chemistry
HRMS Report

### Compound Table

<table>
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<th>Mass</th>
<th>Abund</th>
<th>Formula</th>
<th>TGI Mass</th>
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### MS Spectrum

![MS Spectrum](image)

3x

### MS Spectrum Peak List

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HRMS spectrum of compound 3x
$^1$H NMR spectrum of compound 3y in CDCl$_3$

$^{13}$C NMR spectrum of compound 3y in CDCl$_3$
DEPT-135 NMR spectrum of compound 3y in CDCl₃
HRMS spectrum of compound 3y
$^{1}H$ NMR spectrum of compound 3z in CDCl$_3$

$^{13}C$ NMR spectrum of compound 3z in CDCl$_3$
DEPT-135 NMR spectrum of compound 3z in CDCl₃
HRMS spectrum of compound 3z
$^1$H NMR spectrum of compound 3aa in CDCl$_3$

$^{13}$C NMR spectrum of compound 3aa in CDCl$_3$
DEPT-135 NMR spectrum of compound 3aa in CDCl$_3$
### Compound Table

<table>
<thead>
<tr>
<th>Compound Label</th>
<th>RT</th>
<th>Mass</th>
<th>Abundance</th>
<th>Formula</th>
<th>Type Mass</th>
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<th>D8 Formula</th>
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<tbody>
<tr>
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<td>273.1774</td>
<td>9.06%</td>
<td>C17 H23 N2 O2</td>
<td>273.1728</td>
<td>1.81%</td>
<td>C17 H23 N2 O2</td>
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### Compound Label

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<th>Mass</th>
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</thead>
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<td>0.276</td>
<td>Fixed by Formula</td>
<td>273.1774</td>
</tr>
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</table>

### HS Spectrum

![HS Spectrum](image)

### HS Zoomed Spectrum

![HS Zoomed Spectrum](image)

### MS Spectrum Peak List

<table>
<thead>
<tr>
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<td>C17H21N2O2-</td>
<td>NO2+</td>
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<tr>
<td>254.1376</td>
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<td>NO2+</td>
</tr>
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<td>274.1809</td>
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<td>C17H23N2O2</td>
<td>NO2+</td>
</tr>
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</table>

**3aa**
HRMS spectrum of compound 3aa
$^{1}H$ NMR spectrum of compound 3ab in CDCl$_3$

$^{13}C$ NMR spectrum of compound 3ab in CDCl$_3$
DEPT-135 NMR spectrum of compound 3ab in CDCl$_3$
Qualitative Compound Identification Report

Sample Name: 109B.4.4.D-MRDF-32

Acquisition Method: Direct Injection, HPLC

Sample Group: 1C.1

Retention Time: 14.46

Compound Table

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<th>RT (min)</th>
<th>Mass (Da)</th>
<th>M/z</th>
<th>M/z (Da)</th>
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<tbody>
<tr>
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Compound Chromatograms

MS Spectrum

Cpd 1: C13 H15 N O4, 0.397: +FAB Spectrum (rt: 0.231 min) RDMKS32.d

MS Zoomed Spectrum

Agilent Technologies
HRMS spectrum of compound 3ab
$^1$H NMR spectrum of compound 3ac in CDCl$_3$

$^{13}$C NMR spectrum of compound 3ac in CDCl$_3$
DEPT-135 NMR spectrum of compound 3ac in CDCl₃
INDIAN INSTITUTE OF TECHNOLOGY HYDERABAD
Dept Of Chemistry
HRMS Report

Data File: TFK DK-43 00.d 5NC
Sample Name: TFK DK-43
Acq Method: HRMS_Dual_EI_v1.0_5D_NEW_Agilent.m
DA Method: SD_5D.m

Sample Group: Info.
Acquisition SW: QTOF 8.05.51 (8125.3)
Version: 6250 series TOF/TOF 5.3

Compound Table:

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</thead>
<tbody>
<tr>
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MS Spectrum

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<th>m/z</th>
<th>Count vs. Mass-to-Charge (m/z)</th>
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<tr>
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<td>213</td>
<td>[14H17NO2]+</td>
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</table>

MS Spectrum Peak List

<table>
<thead>
<tr>
<th>m/z</th>
<th>x</th>
<th>Abund</th>
<th>Formula</th>
<th>Int</th>
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</thead>
<tbody>
<tr>
<td>213</td>
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<td>642</td>
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<td>M+H20</td>
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<td>231</td>
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<td>827</td>
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<tr>
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<td>828</td>
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<td>M+</td>
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3ac

Page 1 of 2
Printed at: 3:39 PM on: 11-08-18
### HRMS spectrum of compound 3ac

**HRMS Report**

**HRMS spectrum of compound 3ac**

### MS Spectrum

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<tr>
<th>Mass/Charge</th>
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<td>224.1415</td>
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<td>224.1415</td>
</tr>
<tr>
<td>294.1155</td>
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<td>294.1155</td>
</tr>
<tr>
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<td>1</td>
<td>254.1396</td>
</tr>
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<td>264.1542</td>
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<td>264.1542</td>
</tr>
<tr>
<td>275.0874</td>
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**MS Zoomed Spectrum**

<table>
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<th>Mass/Charge</th>
<th>Intensity</th>
<th>Formula</th>
</tr>
</thead>
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<tr>
<td>223.1336</td>
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<td>294.1155</td>
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<td>254.1396</td>
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<tr>
<td>264.1542</td>
<td>1</td>
<td>264.1542</td>
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<tr>
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**MS Spectrum Peak List**

<table>
<thead>
<tr>
<th>Peak</th>
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--- End Of Report ---
$\text{H NMR spectrum of compound 4a in CDCl}_3$

$\text{\textsuperscript{13}C NMR spectrum of compound 4a in CDCl}_3$
DEPT-135 NMR spectrum of compound 4a in CDCl₃

HRMS spectrum of compound 4a
$^1$H NMR spectrum of compound 3ad in CDCl$_3$

$^{13}$C NMR spectrum of compound 3ad in CDCl$_3$
DEPT-135 NMR spectrum of compound 3ad in CDCl$_3$
INDIAN INSTITUTE OF TECHNOLOGY HYDERABAD
Dept Of Chemistry
HRMS Report

Data File: TPR-DQ-44.002.mz
Sample Name: TPR-DQ-44
Acq Method: HRMS_dual_BSI+,VIE,SD;NMR_Agilent.m

Sample Group: Info.
Acquisition SW: 6200 series TQD/TOQ series
Version: Q/TOP 8.05 01 (88125.3)

Compound Table

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<th>m/z</th>
<th>RT</th>
<th>Abund</th>
<th>Formula</th>
<th>Tgl. Mass</th>
<th>Diff (ppm)</th>
<th>MF2 Formula</th>
<th>DB Formula</th>
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<tbody>
<tr>
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<td>398.1445</td>
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<td>357.1373</td>
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MS Spectrum

<table>
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<tr>
<th>MS Spectrum</th>
<th>m/z</th>
<th>RT</th>
<th>Abund</th>
<th>Formula</th>
<th>Tgl. Mass</th>
<th>Diff (ppm)</th>
<th>MF2 Formula</th>
<th>DB Formula</th>
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</thead>
<tbody>
<tr>
<td>Cod 1: C23 H19 N O3</td>
<td>398.1445</td>
<td>0.277</td>
<td>Find By Formula</td>
<td>357.1373</td>
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MS Spectrum Peak List

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<th>Int</th>
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<td>398.1445</td>
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<td>357.1373</td>
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3ad (1:1)
HRMS spectrum of compound 3ad
\textsuperscript{1}H NMR spectrum of compound 3ae in CDCl$_3$

\textsuperscript{13}C NMR spectrum of compound 3ae in CDCl$_3$
DEPT-135 NMR spectrum of compound 3ae in CDCl₃
IN indIAN INSTITUTE OF TECHNOLOGY HYDERABAD
Dept Of Chemistry
HRMS Report

Compound Table

<table>
<thead>
<tr>
<th>Compound Label</th>
<th>RT (min)</th>
<th>Mass</th>
<th>Abund</th>
<th>Formula</th>
<th>Tg. Mass</th>
<th>HR Mass (exact)</th>
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MS Spectrum

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3ae
(1:2)
HRMS spectrum of compound 3ae

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--- End Of Report ---