Supporting Information

Transition-Metal-Free N-Arylation of Pyrazoles with Diaryliodonium Salts

Zsombor Gonda and Zoltán Novák*[a]

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# Table of contents

General Informations........................................................................................................ 2

Experimental Section ........................................................................................................ 3

Optimization ....................................................................................................................... 3

Reactivity of diaryliodinium salts ....................................................................................... 6

Preparation and Analytical Data of N-H Pyrazoles......................................................... 8

Synthesis of Chalcones ....................................................................................................... 8

Synthesis of Pyrazoles Starting Materials....................................................................... 10

Synthesis of 4-iodopyrazoles ............................................................................................ 12

Preparation and Analytical Data of Diarylodonium Salts ......................................... 14

Synthesis of Iodonium Salts Starting Materials ............................................................... 14

Synthesis of Diarylodonium Salts ................................................................................... 16

Preparation and Analytical Data of N-arylpyrazoles ................................................. 46

References .......................................................................................................................... 62

NMR Spectras .................................................................................................................... 64
General Informations

Unless otherwise indicated, all starting materials were obtained from commercial suppliers, and were used without further purification. Analytical thin-layer chromatography (TLC) was performed on Merck DC pre coated TLC plates with 0.25 mm Kieselgel 60 F254. Visualization was performed with a 254 nm UV lamp. The $^1$H, $^{13}$C and $^{19}$F NMR spectra were recorded on a Bruker Avance-250 spectrometer and in CDCl$_3$, D$_2$COD, CD$_3$CN and DMSO-$d_6$. Chemical shifts are expressed in parts per million ($\delta$) using residual solvent protons as internal standards ($\delta$ 7.26 for $^1$H, $\delta$ 77.0 for $^{13}$C). Coupling constants ($J$) are reported in Hertz (Hz). Splitting patterns are designated as s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet). Combination gas chromatography and low resolution mass spectrometry was obtained on an Agilent 6890N Gas Chromatograph (30 m x 0.25 mm column with 0.25 $\mu$m HP-5MS coating, He carrier gas) and Agilent 5973 Mass Spectrometer (Ion source: EI+, 70eV, 230°C; interface: 300°C). IR spectra were obtained on a Bruker IFS55 spectrometer on a single-reflection diamond ATR unit. All melting points were measured on Büchi 501 apparatus and are uncorrected. High-resolution mass spectra were acquired on an Agilent 6230 time-of-flight mass spectrometer equipped with a Jet Stream electrospray ion source in positive ion mode. Injections of 0.1-0.3 µl were directed to the mass spectrometer at a flow rate 0.5 ml/min (70% acetonitrile-water mixture, 0.1 % formic acid), using an Agilent 1260 Infinity HPLC system. Jet Stream parameters: drying gas (N$_2$) flow and temperature: 10.0 l/min and 325 °C, respectively; nebulizer gas (N$_2$) pressure: 10 psi; capillary voltage: 4000V; sheath gas flow and temperature: 325 °C and 7.5 l/min; TOFMS parameters: fragmentor voltage: 120 V; skimmer potential: 120V; OCT 1 RF Vpp:750 V. Full-scan mass spectra were acquired over the m/z range 100-2500 at an acquisition rate of 250 ms/spectrum and processed by Agilent MassHunter B.03.01 software.
Experimental Section

For the optimization studies we monitored the reactions with GC-MS analysis of samples taken from the reaction mixture at the given reaction time. In the absence of any major side-products the conversions were determined on the basis of the are as of the starting aryl iodide and the product obtained in the GC chromatogram. The conversion was calculated with the following formula: conv % = [Area of product / (Area of product + Area of Aryl iodide)] * 100

Optimization

Base effects

$$\text{HN-}N$$

3,5-diphenyl-1H-pyrazole (1a) (0.025 mmol, 1 equiv), mesityl(phenyl)iodonium triflate (2a) (0.0275 mmol, 1.1 equiv), were placed in a 4 ml screw cap vial and dissolved in toluene or DCE (0.5 mL) and with the appropriate base stirred at RT for the indicated time and analyzed by GC-MS.

<table>
<thead>
<tr>
<th>Entry</th>
<th>Solvent</th>
<th>Base</th>
<th>Time</th>
<th>Yield[^b][%]</th>
</tr>
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<tr>
<td>1</td>
<td>-</td>
<td>n-butylamine</td>
<td>4 h</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>DCE</td>
<td>n-butylamine</td>
<td>4 h</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>DCE</td>
<td>ethanolamine</td>
<td>4 h</td>
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<td>4</td>
<td>DCE</td>
<td>25 w/w% NH\textsubscript{3} (aq) solution</td>
<td>20 min</td>
<td>&gt;99</td>
</tr>
<tr>
<td>5</td>
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<td>20 min</td>
<td>&gt;99</td>
</tr>
<tr>
<td>8</td>
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<td>8</td>
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<td>1 equiv NaOH</td>
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<tr>
<td>10</td>
<td>DCE</td>
<td>1 equiv NH\textsubscript{3}OH</td>
<td>4 h</td>
<td>43</td>
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<tr>
<td>11</td>
<td>DCE</td>
<td>1 equiv 'BuOK</td>
<td>7 h</td>
<td>10</td>
</tr>
<tr>
<td>12</td>
<td>toluene</td>
<td>1 equiv NaOH</td>
<td>20 min</td>
<td>&gt;99</td>
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<tr>
<td>13</td>
<td>toluene</td>
<td>1 equiv 'BuOK</td>
<td>20 min</td>
<td>81</td>
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<tr>
<td>14</td>
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<td>1 equiv K\textsubscript{2}CO\textsubscript{3}</td>
<td>7 h</td>
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1. Table[a] 1a (1 equiv) and 2a (1.1 equiv) were stirred at RT in solvent with base. [b] Determined by GC

Solvent effects

$$\text{HN-}N$$

3,5-diphenyl-1H-pyrazole (1a) (0.025 mmol, 1 equiv), mesityl(phenyl)iodonium triflate (2a) (0.0275 mmol, 1.1 equiv), were placed in a 4 ml screw cap vial and dissolved in 25 w/w%
NH₃ (aq) solution-appropriate solvent 1:1 (1 mL) and stirred at RT for the indicated time and analyzed by GC-MS.

<table>
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<td>84</td>
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<td>0</td>
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<td>acetone</td>
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<td>11</td>
<td>EtOH</td>
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<td>0</td>
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</tbody>
</table>

2. Table [a] 1a (1 equiv) and 2a (1.1 equiv) were stirred at RT in solvent with base. [b] Determined by GC

Anion effect

3,5-diphenyl-1H-pyrazole (0.025 mmol, 1 equiv), diphenyliodonium salt (2ba-d) (0.0275 mmol, 1.1 equiv), were placed in a 4 ml screw cap vial and dissolved in 25 w/w% NH₃ (aq) solution-DCE solvent 1:1 (1 mL) and stirred at RT for the indicated time and analyzed by GC-MS.

<table>
<thead>
<tr>
<th>Entry</th>
<th>Salt (X)</th>
<th>Time</th>
<th>Yield [b] [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2ba(OTf)</td>
<td>20 min</td>
<td>45</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4 h</td>
<td>89</td>
</tr>
</tbody>
</table>
3,5-diphenyl-1H-pyrazole (1a) (0.025 mmol, 1 equiv), mesityl(phenyl)iodonium triflate (2a) (0.0275 mmol, 1.1 equiv), TEMPO (0-2 equiv) were placed in a 4 ml screw cap vial and dissolved in 25 w/w% NH₃ (aq) solution-DCE 1:1 (1 mL) and stirred at RT for 20 min and analyzed by GC-MS.

3-phenyl-1H-pyrazole (1i) (0.025 mmol, 1 equiv), mesityl(phenyl)iodonium triflate (2a) (0.0275 mmol, 1.1 equiv), were placed in a 4 ml screw cap vial and dissolved in 25 w/w% NH₃ (aq) solution-DCE 1:1 (1 mL) and stirred at RT for 20 min and analyzed by GC-MS.
Reactivity of diaryliodinium salts

3,5-diphenyl-1H-pyrazole (0.025 mmol, 1 equiv) and the appropriate diaryliodonium salt (2a-2bn) (0.0275 mmol, 1.1 equiv), were placed in a 4 ml screw cap vial and dissolved in 25 w/w% NH₃ (aq) solution-DCE solvent 1:1 (1 mL) and stirred at RT for 20 min and analyzed by GC-MS.
Figure Conversion and ratio of N-arylated products (GC).
Preparation and Analytical Data of N-H Pyrazoles

Synthesis of Chalcones

1,3-Bis(4-chlorophenyl)prop-2-en-1-one (ii)\(^1,2\)

To a solution of 4-chlorobenzaldehyde (843 mg, 6 mmol) and 4-chloroacetophenone (927.6 mg, 6 mmol) in methanol (30 ml), 10\% (aq) NaOH (12 mmol) was added. The reaction mixture was stirred till completion of starting material. The obtained precipitates were washed with dilute HCl, excess of water, methanol, dried in air and finally recrystallized with methanol ii.

Yellowish-white solid (1.47 g, 5.30 mmol, yield: 88\% m.p. 154-155 °C, (Lit.: 156-157 °C). \(^1\)H NMR (250 MHz, CDCl\(_3\)) \(\delta\) 7.95 (d, \(J = 8.5\) Hz, 2H), 7.76 (d, \(J = 15.7\) Hz, 1H), 7.47 (ddd, \(J = 21.0, 17.2, 8.4\) Hz, 7H) ppm. \(^1^3\)C NMR (63 MHz, CDCl\(_3\)) \(\delta\) 188.95, 143.92, 139.51, 136.77, 136.42, 133.29, 130.02, 129.77, 129.42, 129.12, 121.95 ppm. MS (EI, 70 eV): m/z (\%) 278 (20), 277 (25, [M\(^+\)]), 276 (30), 275 (30), 243 (35), 241 (100), 178 (30), 165 (30), 139 (50), 111 (60), 102 (45), 101 (45), 75 (60).

IR (ATR), 1655, 1603, 1585, 1561, 1485, 1404, 1320, 1300, 1283, 1276, 1215, 1177, 1082, 1031, 1009, 983, 955, 876, 843, 834, 813, 799, 743, 727, 666, 632, 626, 545, 510, 492, 471, 433, 409 cm\(^{-1}\).

1,3-Bis(4-bromophenyl)prop-2-en-1-one (ii)\(^1,3\)

To a solution of 4-bromobenzaldehyde (1.110 g, 6 mmol) and 4-bromoacetophenone (1.194 g, 6 mmol) in methanol (30 ml), 10\% aqueous NaOH (6 mmol) was added. The reaction mixture was stirred till completion of starting material. The obtained precipitates were washed with dilute HCl, excess of water, methanol, dried in air and finally recrystallized with methanol ii.

Yellow solid (2.008 g, 5.49 mmol, yield: 91\%) m.p. 183-185 °C, (Lit.: 185-186 °C). \(^1\)H NMR (250 MHz, CDCl\(_3\)) \(\delta\) 7.95 – 7.84 (m, 2H), 7.74 (d, \(J = 15.7\) Hz, 1H), 7.68 – 7.62 (m, 2H), 7.60 – 7.38 (m, 5H) ppm. \(^1^3\)C NMR (63 MHz, CDCl\(_3\)) \(\delta\) 189.18, 144.07, 136.84, 133.72, 132.41, 132.13, 130.15, 129.98, 128.24, 125.21, 122.03 ppm. MS (EI, 70 eV): m/z (\%): 366 (20, [M\(^+\)]), 365 (20), 287 (70), 285 (70), 211 (20), 209 (20), 185 (30), 183 (35), 178 (55), 157 (40), 155 (40), 102 (100), 89 (30), 76 (70), 75 (60). IR (ATR), 1655, 1601, 1581, 1559, 1481, 1400, 1321, 1300, 1277, 1215, 1178, 1068, 1030, 1006, 983, 955, 894, 875, 846, 832, 810, 793, 738, 708, 663, 631, 620, 537, 492, 478, 452 cm\(^{-1}\).

1,3-Bis(4-tollyl)prop-2-en-1-one (iii)\(^1,4\)

\(^1\)H NMR (250 MHz, CDCl\(_3\)) \(\delta\) 7.95 – 7.84 (m, 2H), 7.74 (d, \(J = 15.7\) Hz, 1H), 7.68 – 7.62 (m, 2H), 7.60 – 7.38 (m, 5H) ppm. \(^1^3\)C NMR (63 MHz, CDCl\(_3\)) \(\delta\) 189.18, 144.07, 136.84, 133.72, 132.41, 132.13, 130.15, 129.98, 128.24, 125.21, 122.03 ppm. MS (EI, 70 eV): m/z (\%): 366 (20, [M\(^+\)]), 365 (20), 287 (70), 285 (70), 211 (20), 209 (20), 185 (30), 183 (35), 178 (55), 157 (40), 155 (40), 102 (100), 89 (30), 76 (70), 75 (60). IR (ATR), 1655, 1601, 1581, 1559, 1481, 1400, 1321, 1300, 1277, 1215, 1178, 1068, 1030, 1006, 983, 955, 894, 875, 846, 832, 810, 793, 738, 708, 663, 631, 620, 537, 492, 478, 452 cm\(^{-1}\).
To a solution of 4-tollylaldehyde (710.7 µL, 6.02 mmol) and 4-methylacetophenone (800 µL, 6.02 mmol) in methanol (30 ml), 10% aqueous NaOH (12 mmol) was added. The reaction mixture was stirred till completion of starting material. The obtained precipitates were washed with dilute HCl, excess of water, methanol, dried in air and finally recrystallized with methanol i3.

Yellowish-white solid (1.239 g, 4.24 mmol, yield: 70 %) m.p. 128-129 °C, (Lit.: 127.5-129 °C). 1H NMR (250 MHz, CDCl3) δ 7.94 (d, J = 8.1 Hz, 2H), 7.80 (d, J = 15.7 Hz, 1H), 7.55 (d, J = 7.5 Hz, 2H), 7.50 (d, J = 15.4 Hz, 1H), 7.30 (d, J = 8.0 Hz, 2H), 7.22 (d, J = 7.9 Hz, 2H), 2.44 (s, 3H), 2.39 (s, 3H) ppm.

13C NMR (63 MHz, CDCl3) δ 190.21, 144.60, 143.62, 141.05, 135.85, 132.34, 129.79, 129.40, 128.72, 128.55, 121.14, 21.79, 21.65 ppm. MS (EI, 70 eV): m/z (%): 236 (40, [M+]), 235 (45), 221 (100), 193 (15), 178 (15), 145 (20), 119 (20), 115 (35), 102 (10), 91 (45).

IR (ATR), 1653, 1606, 1590, 1562, 1511, 1411, 1328, 1307, 1286, 1223, 1201, 1180, 1114, 1027, 1014, 993, 955, 896, 851, 827, 810, 791, 732, 711, 678, 666, 641, 635, 601, 535, 508, 491 cm⁻¹.

1,3-Bis(4-methoxyphenyl)prop-2-en-1-one (i4)³,⁵

To a solution of 4-anisaldehyde (730 µL, 6 mmol) and 4-methoxyacetophenone (901 mg, 6 mmol) in methanol (30 ml), 10% aqueous NaOH (12 mmol) was added. The reaction mixture was stirred till completion of starting material. The obtained precipitates were washed with dilute HCl, excess of water, methanol, dried in air and finally recrystallized with methanol i4.

Yellowish-white solid (1.101 g, 4.10 mmol, yield: 68 %) m.p. 101-103 °C, (Lit.: 101-102 °C). 1H NMR (250 MHz, CDCl3) δ 8.03 (d, J = 8.9 Hz, 2H), 7.78 (d, J = 15.6 Hz, 1H), 7.59 (d, J = 8.7 Hz, 2H), 7.42 (d, J = 15.6 Hz, 1H), 6.94 (dd, J = 11.1, 8.8 Hz, 4H), 3.87 (s, 3H), 3.83 (s, 3H) ppm.

13C NMR (63 MHz, CDCl3) δ 188.81, 163.35, 161.59, 143.88, 131.42, 130.78, 130.19, 127.87, 119.59, 114.46, 113.86, 55.55, 55.48 ppm. MS (EI, 70 eV): m/z (%): 268 (100, [M+]), 253 (40), 237 (30), 225 (30), 161 (25), 153 (10), 135 (40), 118 (10), 108 (10), 92 (30), 77 (35). IR (ATR), 1654, 1591, 1570, 1508, 1454, 1439, 1418, 1333, 1314, 1300, 1250, 1212, 1185, 1177, 1166, 1113, 1108, 1033, 1013, 985, 937, 864, 846, 824, 810, 749, 715, 674, 635, 607, 534, 517 cm⁻¹.

1,3-Bis(4-nitrophenyl)prop-2-en-1-one (i5)⁶,⁷

To solution of 4-nitrobenzaldehyde (906.7 mg, 6 mmol) and 4-nitroacetophenone (990.9 mg, 6 mmol) in mixture of H2O/EtOH (3:1, 60 mL), Na3PO4·12H2O (22.8 mg, 0.06 mmol) was added. The mixture was stirred under reflux conditions for 2 h and then cooled to rt. The precipitate i5 was filtered off, washed thoroughly with water and dried to afford.

Yellow solid (1.686 g, 5.65 mmol, yield: 94 %) m.p. 195-200 °C, (Lit.: 215-216 °C, 178-193 °C). 1H NMR (250 MHz, [D₆]DMSO) δ 8.37 (s, 4H), 8.33 – 8.03 (m, 5H), 7.85 (d, J = 15.8
Hz, 1H) ppm. $^{13}$C NMR (63 MHz, [D$_6$]DMSO) $\delta$ 188.11, 149.98, 148.24, 142.46, 141.76, 140.79, 130.07, 125.58, 123.90, 123.86 ppm. LC-MS (EI, 70 eV): m/z (%): 297 (3, [M$^+$]), 264 (2), 239 (12), 182 (9), 155 (21), 146 (52), 141 (21), 105 (100). IR (ATR), 1697, 1673, 1599, 1516, 1404, 1345, 1318, 1288, 1261, 1239, 1064, 1028, 985, 957, 928, 880, 855, 836, 813, 788, 768, 751, 743, 734, 700, 682, 639, 535, 498 cm$^{-1}$.

**Synthesis of Pyrazoles Starting Materials**

3,5-Bis(4-chlorophenyl)-1H-pyrazole (1b)$^{8}$

A mixture of chalcones i$_1$ (290 mg, 1.046 mmol), sulfur (50 mg, 1.570 mmol) and hydrazine monohydrate (65 $\mu$L, 2.092 mmol) in ethanol (1 ml) was introduced into a glass cylindrical flask placed in an ANTON PAAR microwave reactor and irradiated (300 W) for 2 h at 150 °C under pressure. After cooling, the solvent was evaporated under reduced pressure. (The hydrogen sulfide was trapped using liquid nitrogen.) The residue was treated with ethyl acetate and filtered to remove the excess of sulfur. The solid compound is collected by filtration and evaporated under reduced pressure 1b.

Yellow solid (166.7 mg, 0.58 mmol, yield: 55%) m.p. 226-230 °C, (Lit. 241 °C; 237 °C). $^1$H NMR (250 MHz, [D$_6$]DMSO) $\delta$ 7.85 (d, $J$ = 8.5 Hz, 1H), 7.52 (d, $J$ = 8.5 Hz, 1H), 7.26 (s, 1H) ppm. $^{13}$C NMR (63 MHz, [D$_6$]DMSO) $\delta$ 132.45, 128.99, 126.91, 100.33 ppm. MS (EI, 70 eV): m/z (%): 288 (100, [M$^+$]), 259 (10), 225 (15), 189 (30), 138 (10), 111 (15), 75 (10). IR (ATR), 3137, 3099, 3065, 3018, 2994, 2904, 2859, 2795, 1491, 1472, 1447, 1383, 1304, 1173, 1058, 1013, 973, 961, 831, 818, 810, 774, 745, 556, 507, 491 cm$^{-1}$.

3,5-Bis(4-bromophenyl)-1H-pyrazole (1c)$^{8,9}$

A mixture of chalcones i$_2$ (1.83 g, 5 mmol), sulfur (240 mg, 7.5 mmol) and hydrazine monohydrate (485 $\mu$L, 10.0 mmol) in ethanol (6 ml) was introduced into a glass cylindrical flask placed in an ANTON PAAR microwave reactor and irradiated (300 W) for 2 h at 150 °C under pressure. After cooling, the solvent was evaporated under reduced pressure. (The hydrogen sulfide was trapped using liquid nitrogen.) The residue was treated with ethyl acetate and filtered to remove the excess of sulfur. The solid compound is collected by filtration and evaporated under reduced pressure 1c.

White solid (1.0826 g, 2.86 mmol, yield: 57%) m.p. 229-232 °C, (Lit.: 251 °C). $^1$H NMR (250 MHz, [D$_6$]DMSO) $\delta$ 13.46 (s, 1H), 7.78 (d, $J$ = 8.5 Hz, 4H), 7.35 (d, $J$ = 8.5 Hz, 4H), 7.26 (s, 1H) ppm. $^{13}$C NMR (63 MHz, [D$_6$]DMSO) $\delta$ 159.58, 131.87, 127.18, 121.01, 100.31 ppm. MS (EI, 70 eV): m/z (%): 378 (100, [M$^+$]), 349 (5), 269 (15), 218 (15) 189 (50), 163 (10), 116 (10), 89 (20). IR (ATR), 3137, 3096, 3058, 3011, 2989, 2923, 2900, 2856, 1607, 1597, 1487, 1471, 1444, 1420, 1381, 1303, 1284, 1267, 1225, 1174, 1109, 1073, 1057, 1009, 972, 960, 833, 825, 818, 809, 773, 729, 703, 681, 656, 628, 617, 530, 490, 481, 425 cm$^{-1}$.
3,5-Bis(4-methylphenyl)-1H-pyrazole (1d)

A mixture of chalcones (1.063 g, 4.5 mmol), sulfur (216 mg, 6.75 mmol) and hydrazine monohydrate (437 µL, 9.0 mmol) in ethanol (4.5 ml) was introduced into a glass cylindrical flask placed in an ANTON PAAR microwave reactor and irradiated (300 W) for 2 h at 150 °C under pressure. After cooling, the solvent was evaporated under reduced pressure. (The hydrogen sulfide was trapped using liquid nitrogen.) The residue was treated with ethyl acetate and filtered to remove the excess of sulfur. The solid compound is collected by filtration and evaporated under reduced pressure 1d.

White solid (867 mg, 3.49 mmol, yield: 78%) m.p. 229-232 °C, (Lit.: 234 °C). ¹H NMR (250 MHz, [D₆]DMSO) δ 13.11 (s, 1H), 7.59 (s, 5H), 7.13 (d, J = 5.6 Hz, 5H), 6.95 (s, 1H), 2.21 (s, 7H) ppm. ¹³C NMR (63 MHz, [D₆]DMSO) δ 129.45, 125.10, 99.03, 20.91 ppm. MS (EI, 70 eV): m/z (%): 248 (100, [M⁺]), 202 (10), 178 (5), 91 (5). IR (ATR), 3114, 3078, 3047, 3018, 3000, 2961, 2918, 2854, 1509, 1473, 1460, 1389, 1317, 1310, 1292, 1270, 1232, 1184, 1173, 1120, 1060, 1037, 1018, 974, 963, 935, 825, 816, 770, 715, 684, 658, 644, 513 cm⁻¹.

3,5-Bis(4-methoxiphenyl)-1H-pyrazole (1e)

A mixture of chalcones (1.805 g, 3.0 mmol), sulfur (144 mg, 4.5 mmol) and hydrazine monohydrate (291 µL, 6.0 mmol) in ethanol (3 ml) was introduced into a glass cylindrical flask placed in an ANTON PAAR microwave reactor and irradiated (300 W) for 2 h at 150 °C under pressure. After cooling, the solvent was evaporated under reduced pressure. (The hydrogen sulfide was trapped using liquid nitrogen.) The residue was treated with ethyl acetate and filtered to remove the excess of sulfur. The solid compound is collected by filtration and evaporated under reduced pressure 1e.

Yellowish white solid (835 mg, 2.97 mmol, yield: 99%) m.p. 160-164 °C, (Lit. 172 °C). ¹H NMR (250 MHz, [D₆]DMSO) δ 8.19 – 7.96 (m, 3H), 3.16 (s, 1H) ppm. ¹³C NMR (63 MHz, [D₆]DMSO) δ 159.01, 126.52, 114.27, 98.26, 55.23, 30.76 ppm. MS (EI, 70 eV): m/z (%): 280 (100, [M⁺]), 265 (60), 237 (25), 165 (40), 140 (10), 77 (5). IR (ATR), 3420, 3108, 3075, 3001, 2958, 2930, 2902, 2834, 1613, 1574, 1533, 1507, 1463, 1451, 1437, 1392, 1303, 1291, 1250, 1174, 1113, 1056, 1027, 973, 967, 829, 793, 774, 691, 642, 628, 595, 546, 520, 471, 457 cm⁻¹.

3,5-Bis(4-nitrophenyl)-1H-pyrazole (1f)

A mixture of chalcones (746 mg, 2.50 mmol), sulfur (120 mg, 3.75 mmol) and hydrazine monohydrate (243 µL, 5.0 mmol) in ethanol (5 ml) was introduced into a glass cylindrical flask placed in an ANTONPARR microwave reactor and irradiated (300 W) for 2 h at 150 °C.
under pressure. After cooling, the solvent was evaporated under reduced pressure. (The hydrogen sulfide was trapped using liquid nitrogen.) The residue was treated with ethyl acetate and filtered to remove the excess of sulfur. The solid compound is collected by filtration and evaporated under reduced pressure and purified by flash chromatography 1f. Yellow solid (201.5 mg, 0.65 mmol, yield: 26%) Rf.: 0.50 (in hexane:EtOAc 4:1) m.p. 266-268 °C, (Lit. 266-268 °C). 1H NMR (250 MHz, [D$_6$]DMSO) δ 14.03 (s, 1H), 8.33 (t, J = 9.6 Hz, 4H), 8.19 – 7.99 (m, 4H), 7.64 (s, 1H) ppm. 13C NMR (63 MHz, [D$_6$]DMSO) δ 146.59, 125.92, 124.43, 124.14, 103.16 ppm. MS (EI, 70 eV): m/z (%): 310 (100, [M$^+$]), 280 (30), 252 (20), 236 (10), 218 (25), 206 (20), 190 (25), 163 (15), 116 (15), 89 (30). IR (ATR), 3215, 3153, 3107, 3078, 1602, 1574, 1508, 1492, 1452, 1341, 1290, 1194, 1172, 1108, 1088, 1064, 971, 954, 854, 812, 750, 732, 703, 691, 666, 628, 554, 535, 490, 479 cm$^{-1}$.

**Synthesis of 4-iodopyrazoles**

4-Iodo-3,5-diphenyl-1H-pyrazole (1g)$^{10}$

In a 50 mL round-bottomed flask, iodine (507 mg, 2.0 mmol) was added to diacetoxyiodobenzene (644 mg, 2.0 mmol) in dichloromethane (40 mL). The mixture was stirred for half an hour, then the 3,5-diphenyl-1H-pyrazole (880 mg, 4.0 mmol) was added to the solution. After 15 min of stirring at rt, the solvent was distilled off under reduced pressure. The residue obtained was purified on a silica-gel plate. After removal of the solvent, the product 1g obtained was recrystallized with ethanol or water. White solid (1.120 g, 3.2 mmol, yield: 81%) Rf = 0.23 (hexane:EtOAc 4:1) m.p. 172-173 °C, (Lit.: 194-196 °C). 1H NMR (250 MHz, CDCl$_3$) δ 10.65 (s, 1H), 7.63 (s, 4H), 7.39 (s, 5H) ppm. 13C NMR (63 MHz, CDCl$_3$) δ 149.84, 130.90, 129.10, 128.67, 128.60 ppm. MS (EI, 70 eV): m/z (%): 346 (100, [M$^+$]), 218 (5), 189 (40), 165 (10), 116 (10), 77 (10). IR (ATR), 3195, 3092, 3057, 1486, 1459, 1448, 1435, 1383, 1252, 1143, 1113, 1073, 1025, 975, 958, 917, 763, 716, 692, 661, 617, 570, 512, 491 cm$^{-1}$.

4-Iodo-3,5-di-p-tolyl-1H-pyrazole (1i)

In a 50 mL round-bottomed flask, iodine (177.8 mg, 0.7 mmol) was added to diacetoxyiodobenzene (225.5 mg, 0.7 mmol) in dichloromethane (14 mL). The mixture was stirred for half an hour, then the 3,5-di-p-tolyl-1H-pyra-zole (347.2 mg, 1.4 mmol) was added to the solution. After 15 min of stirring at rt, the solvent was distilled off under reduced pressure. The residue obtained was purified on a silica-gel plate. After removal of the solvent, the product 1i obtained was recrystallized with ethanol. White solid (381 mg, 1.01 mmol, yield: 73%) m.p. 149-150 °C. 1H NMR (250 MHz, CDCl$_3$) δ 11.68 (s, 1H), 7.46 (d, J = 7.7 Hz, 4H), 7.13 (d, J = 7.6 Hz, 4H), 2.39 (s, 6H) ppm. 13C NMR (63 MHz, CDCl$_3$) δ 149.63, 138.51, 129.12, 128.43, 128.15, 58.46, 21.47 ppm. MS (EI, 70 eV): m/z (%): 344 (100, [M$^+$]), 218 (5), 189 (40), 165 (10), 116 (10), 77 (10). IR (ATR), 3195, 3092, 3057, 1486, 1459, 1448, 1435, 1383, 1252, 1143, 1113, 1073, 1025, 975, 958, 917, 763, 716, 692, 661, 617, 570, 512, 491 cm$^{-1}$.
A mixture of 1,3-dimethyl-pyrazole (961 mg, 10.0 mmol) and ICl (751 µL, 15.0 mmol), in ethyl acetate (50 mL), was irradiated in a water bath of the ultrasonic cleaner at 25-30 °C for 15 min. After the indicated time, the organic phase was washed with a solution of Na2S2O3 (3 × 50 mL). The organic extract was dried (MgSO4) and the solvent removed under reduced pressure. Finally, the 3,5-dimethyl-4-iodopyrazoles 1j were obtained in good purity. Yellowish-white solid (2.117 g, 9.53 mmol, yield: 95%) m.p. 130-133 °C, (Lit.: 134-136 °C). 1H NMR (250 MHz, CDCl3) δ 9.31 (s, 1H), 2.27 (s, 6H) ppm. 13C NMR (63 MHz, CDCl3) δ 146.17, 62.52, 12.76 ppm. MS (EI, 70 eV): m/z (%): 222 (100, [M+H]+), 127 (20), 95 (20), 65 (20). IR (ATR), 3163, 3067, 3027, 2958, 2918, 2828, 2724, 2603, 1576, 1463, 1411, 1305, 1164, 1077, 1033, 1003, 864, 764, 593, 454 cm⁻¹.

A mixture of pyrazole (340 mg, 5.0 mmol) and ICl (376 µL, 7.5 mmol), in ethyl acetate (5 mL), was irradiated in a water bath of the ultrasonic cleaner at 25-30 °C for one day. After the indicated time, the organic phase was washed with a solution of Na2S2O3 (3 × 5 mL). The organic extract was dried (MgSO4) and the solvent removed under reduced pressure. Finally, the 4-iodopyrazoles 1k were obtained in good purity. White solid (594 mg, 3.06 mmol, yield: 61%) m.p. 101-105 °C, (Lit.: 136-138 °C, 105-107 °C). 1H NMR (250 MHz, CDCl3) δ 11.39 (s, 1H), 7.64 (s, 2H) ppm. 13C NMR (63 MHz, CDCl3) δ 138.89, 56.70 ppm. MS (EI, 70 eV): m/z (%): 194 (100, [M+]), 165 (5), 139 (10), 127 (30), 67 (15). IR (ATR), 3113, 3034, 2897, 2840, 2780, 1622, 1525, 1473, 1364, 1321, 1260, 1176, 1141, 1030, 952, 934, 869, 803, 653, 648, 607 cm⁻¹.

A mixture of bis-(4-chlorophenyl)-pyrazole (186 mg, 0.64 mmol) and ICl (48.3 µL, 0.965 mmol), in ethyl acetate (7 mL), was irradiated in a water bath of the ultrasonic cleaner at 25-30 °C for 3 × 15 min. After the indicated time, the organic phase was washed with a solution of Na2S2O3 (3 × 20 mL). The organic extract was dried (MgSO4) and the solvent removed under reduced pressure. Finally, the bis-(4-chlorophenyl)-iodopyrazoles 1h were obtained in good purity.
White solid (190 mg, 0.46 mmol, yield: 72%) m.p. 189-192 °C. 1H NMR (250 MHz, [D6]DMSO) δ 13.83 (s, 1H), 7.82 (d, J = 8.3 Hz, 2H), 7.73 (d, J = 8.4 Hz, 2H), 7.63 (d, J = 8.4 Hz, 2H), 7.55 (d, J = 8.4 Hz, 2H) ppm. 13C NMR (63 MHz, [D6]DMSO) δ 130.29, 129.97, 129.03, 128.58, 127.01, 59.44 ppm. MS (EI, 70 eV): m/z (%): 415 (70), 413 (100, [M+]), 288 (5), 281 (20), 210 (25), 187 (30), 150 (15), 123 (10), 75 (10). IR (ATR), 3180, 2925, 1735, 1598, 1506, 1487, 1428, 1375, 1352, 1299, 1282, 1245, 1202, 1141, 1116, 1093, 1059, 1011, 976, 958, 831, 796, 772, 750, 727, 718, 693, 663, 629, 583, 552, 539 cm⁻¹. HRMS calcd for C15H10Cl2IN2 [M+H]+ 414.9266 found 414.9276

**Preparation and Analytical Data of Diaryiodonium Salts**

**Synthesis of Iodonium Salts Starting Materials**

![4-Diacetoxyiodoanisole (i6)](image)

A stirred suspension of NaIO4 (1.10 g, 5.15 mmol) and AcONa (0.902 g, 11.0 mmol) in glacial AcOH (7.5 mL) and Ac2O (0.75 mL) at rt was treated with the appropriate iodoarene (5.0 mmol). The reaction mixture was refluxed for 2 h. The reaction mixture was then poured into water (25 mL). The resulting mixture was filtered, and the residue was washed with 10% aq AcOH (2 × 5 mL) and dried under a stream of air. The remaining product was extracted from the filtrate with DCM and concentrated on rotary evaporator. Hexane was added to the obtained residue to collect the solid product. The product was filtered and washed with excess of hexane to provide corresponding iodoarene i6.

White solid (1.5407 g, 0.944 mmol, yield: 88%) m.p. 78-81 °C, (Lit.: 93-96 °C). 1H NMR (250 MHz, CDCl3) δ 8.01 (d, J = 9.0 Hz, 1H), 6.96 (d, J = 9.0 Hz, 1H), 3.86 (s, 2H), 1.99 (s, 3H) ppm. 13C NMR (63 MHz, CDCl3) δ 176.51, 162.30, 137.25, 116.78, 111.82, 111.82, 55.72, 20.50 ppm. IR (ATR), 1629, 1582, 1571, 1487, 1358, 1291, 1254, 1189, cm⁻¹.

![4-Diacetoxyiodotoluene (i7)](image)

A stirred suspension of NaIO4 (1.10 g, 5.15 mmol) and AcONa (0.902 g, 11.0 mmol) in glacial AcOH (7.5 mL) and Ac2O (0.75 mL) at rt was treated with the appropriate iodoarene (5.0 mmol). The reaction mixture was refluxed for 2 h. The reaction mixture was then poured into water (25 mL). The resulting mixture was filtered, and the residue was washed with 10% aq AcOH (2 × 5 mL) and dried under a stream of air. The remaining product was extracted from the filtrate with DCM and concentrated on rotary evaporator. Hexane was added to the obtained residue to collect the solid product. The product was filtered and washed with excess of hexane to provide corresponding iodoarene i7.
White solid (707.2 mg, 2.10 mmol, yield: 42%) m.p. 105-107 °C, (Lit.: 104-105 °C). $^1$H NMR (250 MHz, CDCl$_3$) $\delta$ 7.95 (d, $J = 8.4$ Hz, 2H), 7.27 (d, $J = 8.3$ Hz, 2H), 2.42 (s, 3H), 1.97 (s, 6H) ppm. $^{13}$C NMR (63 MHz, CDCl$_3$) $\delta$ 176.76, 143.06, 135.36, 132.13, 118.71, 21.92, 20.76 ppm. IR (ATR), 1639, 1582, 1482, 1363, 1288, 1256, 1190 cm$^{-1}$.

1-(Fluoro-[hydroxy(tosyloxy)iodo]benzene (i8)$^{14}$

$m$-Chloroperbenzoic acid ($m$-CPBA; 77% max.; 588 mg, 2.385 mmol) was added portion-wise to a solution of 4-fluoroiiodobenzene (250 µL, 2.168 mmol) in CHCl$_3$ (30 mL). p-TsOH·H$_2$O (453 mg, 2.385 mmol) was added to the pale yellow solution and the temperature was raised to 40 °C. The reaction mixture was stirred for 1 h and then cooled to rt. The resultant precipitate was filtered off, washed with Et$_2$O (3 × 50 mL), and dried in vacuo for 6 h to give 1-fluoro-[hydroxy(tosyloxy)iido]benzene i8 as a white solid.

White solid (425.3 mg, 1.04 mmol, 48%) m.p. 112-115 °C (Lit.: 145-146 °C). $^1$H NMR (250 MHz, [D$_6$]DMSO) $\delta$ 9.81 (s, 1H), 8.29 (dd, $J = 8.4$, 5.2 Hz, 1H), 7.46 (t, $J = 8.4$ Hz, 2H), 7.12 (d, $J = 7.8$ Hz, 1H), 2.29 (s, 2H) ppm. $^{13}$C NMR (63 MHz, [D$_6$]DMSO) $\delta$ 164.01 (d, $J = 251.7$ Hz), 145.33, 137.88, 137.76 (d, $J = 9.2$ Hz), 128.16, 125.53, 118.27 (d, $J = 3.3$ Hz) 20.82. IR (ATR), 3256, 3089, 1701, 1654, 1599, 1591, 1574, 1480, 1455, 1398, 1227, 1151, 1105, 1090, 1033, 1025, 1004, 991, 831, 825, 813, 708, 679, 636, 617, 590, 580, 566, 557, 508 cm$^{-1}$.

1,3,5-Trimethylphenyl-[hydroxy(tosyloxy)iido]benzene (i9)$^{15}$

Iodine (507.6 mg, 2.0 mmol) was dissolved in dichloromethane (20 mL) was added. To the resulting stirred solution was added mesitylene (556 µL, 4.0 mmol), followed by $m$-chloroperbenzoic acid ($m$-CPBA; 65% max.; 1.593 g, 6.0 mmol) and TsOH·H$_2$O (761 mg, 4.0 mmol). The mixture was stirred at room temperature for 60 min and concentrated under a stream of air, then diethyl ether (20 mL) was added to the remaining residue. The resulting precipitate was stirred for 30 min and cooled at –20 °C, over night to precipitate out an off-white solid filtered off and dried in vacuo to give compound as a solid i9.

White solid (1.173 g, 2.70 mmol, yield: 67.5%) m.p. 105-152 °C (Lit.: 104-105 °C). $^1$H NMR (250 MHz, D$_2$COD) $\delta$ 7.61, 7.58, 7.27, 7.21, 7.17, 2.71, 2.40, 2.36 ppm. $^{13}$C NMR (63 MHz, D$_2$COD) $\delta$ 147.38, 144.13, 142.91, 141.87, 130.56, 129.79, 126.87, 26.67, 21.32, 21.28 ppm. IR (ATR), 3142, 3021, 2981, 2919, 1453, 1379, 1230, 1140, 1103, 1028, 1005, 998, 950, 854, 820, 814, 682, 580, 562, 544 cm$^{-1}$.
The mixture of iodobenzene (0.8 mL, 7.149 mmol) and paratoluenesulfonic acid monohydrate (1.496 g, 7.684 mmol) in CHCl₃ (7.2 mL), was m-Chloroperbenzoic acid (70% active oxidant, 1.357 g, 7.684 mmol). The mixture obtained was stirred for 2 h at rt under an argon atmosphere. After the reaction, Et₂O (36 mL) was added to the reaction mixture, and the resulting mixture was filtered and the solids were washed with Et₂O to provide [hydroxy(tosyloxy)iodo]benzene i10. When precipitation does not occur smoothly, it is better to remove CHCl₃ before the addition of Et₂O.

White solid (2.6438 g, 6.74 mmol, yield: 94.3%) m.p. 134-136 °C (Lit. 135-138 °C). ¹H NMR (250 MHz, [D₆]DMSO) δ 8.21 (d, J = 7.4 Hz, 2H), 7.65 (dd, J = 17.4, 7.2 Hz, 3H), 7.48 (d, J = 7.8 Hz, 2H), 7.12 (d, J = 7.8 Hz, 2H), 2.29 (s, 3H) ppm. ¹³C NMR (63 MHz, [D₆]DMSO) δ 145.27, 137.93, 134.54, 132.42, 131.16, 128.18, 125.54, 123.59, 20.83 ppm. IR (ATR), 3088, 2448, 1597, 1439, 1226, 1143, 1102, 1063, 1022, 1008, 986, 851, 811, 742, 706, 679, 594, 555, 458 cm⁻¹.

[Hydroxy-(4-tosyloxy)iodo]-4-nitrobenzene (i11)¹⁷

Iodo-4-nitrobenzene (762 mg, 3.06 mmol) and paratoluenesulfonic acid monohydrate (628 mg, 3.30 mmol) were added sequentially at rt to a stirring solution of m-Chloroperbenzoic acid (70% active oxidant, 850 mg, 3.45 mmol) in CHCl₃ (9 mL). The flask was equipped with a water-jacketed condenser and the reaction mixture was heated in a 60 °C oil bath for 2 hours. After cooling to rt, the solvent was removed under a stream of cool air. The crude material was triturated with Et₂O and placed in a freezer at -20 °C overnight. Filtration and multiple washes with Et₂O yielded the product as a pale solid i11.

Pale-white solid (940 mg, 2.64 mmol, yield: 86%) m.p. 135-136 °C (Lit.: 157-160 °C, 149-152 °C). ¹H NMR (250 MHz, CD₃OD) δ 8.45 (d, J = 8.8 Hz, 1H), 8.34 (d, J = 8.9 Hz, 1H), 7.58 (d, J = 8.0 Hz, 1H), 7.17 (d, J = 7.9 Hz, 1H), 2.32 (s, 2H) ppm. ¹³C NMR (63 MHz, CD₃OD) δ 151.58, 142.45, 142.16, 137.46, 129.85, 127.45, 127.05, 126.87, 21.20 ppm. IR (ATR), 2980, 2476, 1601, 1530, 1492, 1466, 1350, 1311, 1203, 1182, 1142, 1113, 1100, 1050, 1034, 1024, 996, 848, 842, 827, 814, 734, 707, 677, 607, 562, 550, 493, 466, 453 cm⁻¹.

Synthesis of Diarylodonium Salts

Mesityl(phenyl)iodonium triflate (2a)¹⁸

m-Chloroperbenzoic acid (70% active oxidant, 2.465 g, 10 mmol), iodobenzene (1 mL, 9 mmol), and mesitylene (1.4 mL, 10 mmol) were dissolved in CH₂Cl₂ (35 mL) stirred at 0 °C.
Then, HOTf (0.95 mL, 11 mmol) was added dropwise to the solution and the mixture was stirred at rt for 2 h and the solution was concentrated in vacuo. Et₂O (35 mL) was added and the mixture was cooled at -20 °C, over night to precipitate out an off-white solid. The precipitate was filtered off, washed with cold Et₂O, and dried under vacuum at 100 °C to give salt. The mesityl(phenyl)iodonium triflate 2a was recrystallisation in EtOAc.

Off-white solid (3.028 g, 6.4 mmol, yield: 71%) m.p. 138-141 °C, (Lit.: 138-140 °C). ¹H NMR (250 MHz, [D₆]DMSO) δ 7.97 (d, J = 7.4 Hz, 2H), 7.64 (t, J = 7.4 Hz, 1H), 7.56 – 7.43 (m, 2H), 7.22 (s, 2H), 2.60 (s, 6H), 2.30 (s, 3H) ppm. ¹³C NMR (63 MHz, [D₆]DMSO) δ 143.13, 141.57, 134.45, 131.88, 131.79, 129.78, 122.53, 114.47, 26.28, 20.49 ppm. IR (ATR), 2917, 1581, 1474, 1381, 1246, 1157, 1025, 984, 741, 633 cm⁻¹.

Mesityl(2,4,6-trimethoxyphenyl)iodonium triflate (2f)

To a stirred solution of NaOTf (189.1 mg, 1.1 mmol) in water (10 mL), mesityl(2,4,6-trimethoxyphenyl)iodonium tosylate 2a (584.5 mg, 1.0 mmol) was added in one portion at room temperature under air, and it was stirred for 24 h. The precipitate was filtered and dried in vacuo to give pure as a powder 2f. Grey solid (496 mg, 0.88 mmol, yield: 88%) m.p. 146-150 °C. ¹H NMR (250 MHz, D₃COD) δ 7.12 (s, 2H), 6.40 (s, 2H), 3.92 (s, 6H), 3.88 (s, 3H), 2.63 (s, 6H), 2.31 (s, 3H) ppm. ¹³C NMR (63 MHz, D₃COD) δ 168.43, 161.57, 144.64, 143.61, 130.72, 121.79 (d, J = 318.4 Hz), 121.53, 93.07, 83.36, 57.46, 56.65, 26.52, 20.89 ppm. IR (ATR), 2944, 1581, 1473, 1454, 1434, 1417, 1380, 1351, 1301, 1276, 1244, 1231, 1212, 1191, 1165, 1155, 1128, 1070, 1028, 992, 951, 913, 848, 818, 756, 680, 666, 637, 572, 542, 514, 464 cm⁻¹. HRMS calcd for C₁₈H₂₂IO₃ [M-OTf]⁺ 413.0614 found 413.0608

4-Nitrophenyl(phenyl)iodonium triflate (2j)¹⁹

m-Chloroperbenzoic acid (65% active oxidant, 772 mg, 3.134 mmol) and 4-iodo-nitrobenzene (289 mg, 1.1615 mmol) were dissolved in CH₂Cl₂ (5 mL) in a bomb tube. The benzene (113 µL, 1.266 mmol) was added and the solution was cooled to 0 °C followed by dropwise addition of TfOH (200 µL, 2.277 mmol), resulting in a coloured solution. The reaction mixture was stirred at 80 °C and 15 h and subsequently concentrated under vacuum (while still cold for low-temperature reactions). Et₂O (10 mL) was added and the mixture was stirred at room temperature for 10 min to precipitate out an off-white solid. To ensure complete precipitation, the flask was stored in the freezer for at least 30 min before the solid was filtered off, washed with Et₂O and dried under vacuum to give diaryliodonium salt 2j. Off-white solid (464 mg, 0.98 mmol, yield: 84%) m.p. 180-185 °C, (Lit.189–190 °C) ¹H NMR (250 MHz, CDCl₃) δ 8.48 (d, J = 8.8 Hz, 1H), 8.37 – 8.23 (m, 2H), 7.69 (t, J = 7.3 Hz, 1H), 7.55 (t, J = 7.7 Hz, 1H) ppm. ¹³C NMR (63 MHz, CDCl₃) δ 149.54, 136.55, 135.59,
132.57, 132.11, 126.36, 122.71, 116.95 ppm. IR (ATR), 3069, 1601, 1572, 1521, 1473, 1445, 1355, 1339, 1315, 1291, 1219, 1190, 1165, 1138, 1109, 1021, 1003, 988, 928, 853, 847, 754, 734, 708, 679, 670, 656, 634, 575, 519, 513 cm\(^{-1}\).

**Phenyl(3-pyridinium)iodonium bistriflate (2k)**

To a solution of 3-iodopyridine (500 mg, 2.44 mmol) in CH\(_2\)Cl\(_2\) (10 mL) was added TfOH (850 μL, 9.7 mmol, 4 equiv) and the resulting mixture was stirred at rt for 5 min. m-chloroperbenzoic acid (70% active oxidant, 887.5 mg, 3.6 mmol, 1.5 equiv) followed by the benzene (236 μL, 2.64 mmol, 1.1 equiv) was then added. The reaction vessel was sealed and submitted to a 60 °C oilbath with stirring for 30 min. The reaction mixture was then allowed to reach rt after which it was concentrated in vacuo. Et\(_2\)O (10 mL) was added and the mixture was stirred at 0 °C for 30 min. The resulting precipitate was filtered through a glass-sintered funnel and washed with additional Et\(_2\)O (3 × 10 mL) to give the protonated N-heteroaryliodonium bistriflate 2k.

Off-white solid (1.0596 g, 1.82 mmol, yield: 75%) m.p. 135-140 °C, (Lit.: 127-130 °C). \(^1\)H NMR (250 MHz, D\(_3\)COD) δ 9.36 (d, \(J = 1.7\) Hz, 1H), 8.95 – 8.81 (m, 2H), 8.21 (d, \(J = 7.6\) Hz, 2H), 7.80 (dd, \(J = 8.2, 5.3\) Hz, 1H), 7.64 (d, \(J = 7.4\) Hz, 1H), 7.49 (t, \(J = 7.8\) Hz, 2H) ppm.

\(^13\)C NMR (63 MHz, D\(_3\)COD) δ 151.22, 149.89, 148.79, 136.94, 134.33, 133.54, 129.61, 121.75 (d, \(J = 318.3\) Hz), 116.41, 115.22 ppm. IR (ATR), 3088, 3055, 3004, 1588, 1520, 1471, 1458, 1445, 1314, 1264, 1210, 1194, 1184, 1161, 1107, 1082, 1019, 984, 913, 803, 763, 732, 681, 675, 669, 651, 634, 621, 574, 521, 514, 459 cm\(^{-1}\).

**4-Tolyl(3-pyridinium)iodonium bistriflate (2l)**

To a solution of 3-iodopyridine (500 mg, 2.44 mmol) in CH\(_2\)Cl\(_2\) (10 mL) was added TfOH (850 μL, 9.7 mmol, 4 equiv) and the resulting mixture was stirred at rt for 5 min. m-chloroperbenzoic acid (70% active oxidant, 887.5 mg, 3.6 mmol, 1.5 equiv) followed by the toluene (281 μL, 2.64 mmol, 1.1 equiv) was then added. The reaction vessel was sealed and submitted to a 60 °C oilbath with stirring for 30 min. The reaction mixture was then allowed to reach rt after which it was concentrated in vacuo. Et\(_2\)O (10 mL) was added and the mixture was stirred at 0 °C for 30 min. The resulting precipitate was filtered through a glass-sintered funnel and washed with additional Et\(_2\)O (3 × 10 mL) to give the protonated N-heteroaryliodonium bistriflate 2l.

Off-white solid (0.7094 g, 1.19 mmol, yield: 49%) m.p. 170-172 °C. \(^1\)H NMR (250 MHz, D\(_3\)COD) δ 9.35 (d, \(J = 1.8\) Hz, 1H), 8.99 – 8.80 (m, 2H), 8.06 (d, \(J = 8.4\) Hz, 2H), 7.84 (dd, \(J = 8.2, 5.5\) Hz, 1H), 7.28 (d, \(J = 8.3\) Hz, 2H), 2.30 (s, 3H) ppm. \(^13\)C NMR (63 MHz, D\(_3\)COD) δ
150.28, 149.73, 148.90, 146.03, 137.00, 134.28, 129.85, 121.73 (d, \( J = 318.5 \) Hz), 115.17, 112.68, 21.40 ppm. IR (ATR), 3546, 3468, 3102, 3065, 1635, 1617, 1581, 1513, 1486, 1447, 1283, 1241, 1179, 1161, 1109, 1082, 1033, 1020, 995, 921, 808, 802, 761, 685, 675, 648, 631, 575, 517, 482 cm\(^{-1}\). HRMS calcd for C\(_{12}\)H\(_{11}\)IN [M-HOTf-OTf]\(^+\) 295.9936 found 295.9947

4-Anisyl(3-pyridinium)iodonium bistriflate (2m)

To a solution of 3-iodopyridine (500 mg, 2.44 mmol) in CH\(_2\)Cl\(_2\) (10 mL) was added TfOH (850 \( \mu \)L, 9.7 mmol, 4 equiv) and the resulting mixture was stirred at rt for 5 min. \( m \)-chloroperbenzoic acid (70% active oxidant, 887.5 mg, 3.6 mmol, 1.5 equiv) followed by the anisole (287 \( \mu \)L, 2.64 mmol, 1.1 equiv) was then added. The reaction vessel was sealed and submitted to a 60 °C oilbath with stirring for 30 min. The reaction mixture was then allowed to reach rt after which it was concentrated in vacuo. Et\(_2\)O (10 mL) was added and the mixture was stirred at 0 °C for 30 min. The resulting precipitate was filtered through a glass-sintered funnel and washed with additional Et\(_2\)O (3×10 mL) to give the protonated \( N \)-heteroaryliodonium bistriflate 2m.

Biege solid (1.217 g, 1.99 mmol, yield: 82 %) m.p. 165-168 °C, (Lit.: 181-183 °C). \(^1\)H NMR (250 MHz, D\(_3\)COD) \( \delta \) 9.33 (s, 1H), 8.89 (dd, \( J = 10.2, 2.9 \) Hz, 2H), 8.10 (d, \( J = 9.1 \) Hz, 2H), 7.85 (dd, \( J = 8.1, 5.6 \) Hz, 1H), 6.99 (d, \( J = 9.1 \) Hz, 2H), 3.74 (s, 3H) ppm. \(^{13}\)C NMR (63 MHz, D\(_3\)COD) 165.03, 149.86, 149.77, 148.57, 139.22, 129.87, 121.74 (d, \( J = 316.1 \) Hz), 119.23, 115.44, 104.58, 56.45 ppm. IR (ATR), 3108, 3093, 2849, 1571, 1524, 1486, 1461, 1442, 1262, 1221, 1196, 1173, 1153, 1120, 1047, 1022, 892, 828, 788, 760, 669, 631, 589, 572, 513, 445 cm\(^{-1}\).

4-Anisyl(7-quinolinium)iodonium bistriflate (2n)

To a solution of 7-iodoquinoline (510 mg, 2.0 mmol) in CH\(_2\)Cl\(_2\) (13.3 mL) was added TfOH (702 \( \mu \)L, 8.0 mmol, 4 equiv) and the resulting mixture was stirred at rt for 5 min. \( m \)-chloroperbenzoic acid (70% active oxidant, 863 mg, 3.5 mmol, 1.75 equiv) was then added. The reaction vessel was sealed and submitted to a 60 °C oilbath with stirring for 30 min after which it was cooled down to 0 °C. H\(_2\)O (72 \( \mu \)L, 4.0 mmol, 2 equiv) was added, followed by the dropwise addition of the anisole (261 \( \mu \)L, 2.4 mmol, 1.2 equiv) dissolved in CH\(_2\)Cl\(_2\) (6.7 mL) via syringe. The reaction mixture was stirred for 15 min at 0 °C before it was concentrated in vacuo. Et\(_2\)O (1-3 mL) was added and the mixture was cooled to 0°C and stirred for 30 min. The resulting precipitate was filtered through a glass sintered funnel and
washed with additional Et₂O (3 × 3 mL) to give the protonated N-heteroaryliodonium bistriflate 2n.

Off-white solid (1.097 g, 1.66 mmol, yield: 83%) m.p. 179-184 °C. ¹H NMR (250 MHz, D₃COD) δ 9.34 (d, J = 5.3 Hz, 1H), 9.22 (dd, J = 9.0, 5.1 Hz, 2H), 8.71 (dd, J = 9.1, 1.8 Hz, 1H), 8.33 – 8.12 (m, 4H), 7.16 (s, 3H) ppm. ¹³C NMR (63 MHz, D₃COD) δ 164.88, 149.35, 148.81, 139.43, 139.10, 138.33, 131.46, 125.64, 124.96, 121.76 (d, J = 318.7 Hz), 119.15, 116.86, 104.74, 56.41 ppm. IR (ATR), 3080, 2817, 2762, 1646, 1590, 1577, 1566, 1487, 1462, 1442, 1307, 1275, 1257, 1231, 1174, 1139, 1020, 991, 939, 914, 898, 847, 835, 815, 769, 759, 748, 633, 618, 588, 575 cm⁻¹. HRMS calcd for C₁₆H₁₃INO [M-HOTf-OTf]⁺ 362.0042 found 362.0054

**4-Anisyl(4-nitrophenyl)iodonium tosylate (2o)**[^17]

[Hydroxy-(4-tosyloxy)iodo]-4-nitrobenzene i11 (440 mg, 1.005 mmol) was added at 0 °C to a stirring solution of anisole (109 µL, 1.000 mmol) in 1:1 CH₂Cl₂:CF₃CH₂OH (6.6 mL). The reaction mixture was allowed to reach rt and stirring was continued overnight before concentrating to dryness by rotary evaporation. This crude material was triturated with Et₂O. Isolation by filtration and multiple washes with Et₂O yielded the diaryliodonium salt 2o.

White solid (504.3 mg, 0.956 mmol, yield: 96%) m.p. 168–169 °C, (Lit. 196–198 °C (dec.)). ¹H NMR (250 MHz, D₃COD) δ 8.27 (q, J = 9.2 Hz, 1H), 8.12 (d, J = 9.1 Hz, 1H), 7.65 (d, J = 8.2 Hz, 1H), 7.18 (d, J = 7.9 Hz, 1H), 7.05 (d, J = 9.1 Hz, 1H), 3.82 (s, 1H), 2.33 (s, 1H) ppm. ¹³C NMR (63 MHz, D₃COD) δ 164.81, 143.54, 141.69, 139.03, 137.16, 129.82, 127.35, 126.95, 122.25, 119.08, 104.63, 56.41, 21.29 ppm. IR (ATR), 3093, 3067, 1583, 1571, 1521, 1489, 1462, 1442, 1408, 1351, 1342, 1303, 1265, 1211, 1179, 1166, 1118, 1107, 1057, 1029, 1019, 1006, 993, 847, 812, 735, 705, 681, 591, 569, 562, 512 cm⁻¹.

**4-Anisyl(6-chloro-3-pyridyl)iodonium triflate (2p)**[^21]

*m*-Chloroperbenzoic acid (*m*-CPBA 70% active oxidant, 536 mg, 2.17 mmol) and 2-chloro-5-iodopyridine (478.9 mg, 2.0 mmol) were dissolved in CH₂Cl₂ (8 mL) in a 25 mL round bottom flask. TfOH (527 µL, 6.0 mmol, 3 equiv) was added at rt and the reaction was stirred for 10 min at rt and change of color from a clear to a yellow transparent solution. Before cooling to -78 °C and slow addition of anisole (236 µL, 2.17 mmol) in CH₂Cl₂ (8 mL). The reaction was then stirred at -78 °C for 10 min and concentrated in vacuo while still cold. Et₂O (1–2 mL) was added and the mixture was stirred at rt for 10 min to precipitate out an off-white solid. The flask was stored in the freezer for 30 min, then the solid was filtered off, washed with cold Et₂O and dried under vacuum to give diaryliodonium salt 2p as a slightly grey solid. Grey solid (732.2 g, 1.48 mmol, yield: 74%) m.p. 92-98 °C, (Lit.: 101-103 °C). ¹H NMR (250 MHz, [D₆]DMSO) δ 9.12 (d, J = 2.2 Hz, 1H), 8.64 (dd, J = 8.5, 2.4 Hz, 1H), 8.28 – 8.10 (m,
2H), 7.72 (d, J = 8.5 Hz, 1H), 7.19 – 7.01 (m, 2H), 3.79 (s, 3H) ppm. \(^{13}\)C NMR (63 MHz, \([D_6]\)DMSO) \(\delta\) 162.20, 154.02, 153.19, 145.25, 137.33, 127.54, 120.68 (d, J = 322.5 Hz), 117.66, 114.57, 105.68, 55.76 ppm. IR (ATR), 3078, 3054, 1582, 1571, 1444, 1256, 1184, 1158, 1020 cm\(^{-1}\).

4-Anisyl(phenyl)iodonium tosylate (2q)\(^{22}\)

\(m\)-Chloroperbenzoic acid (\(m\)-CPBA 70% active oxidant, 616 mg, 2.5 mmol), iodobenzene (280 \(\mu\)L, 2.5 mmol), and anisole (272 \(\mu\)L, 2.5 mmol) were dissolved in CH\(_2\)Cl\(_2\) (5 mL) and 2,2,2-trifluoroethanol (5 mL). Then, TsOH·H\(_2\)O (475.5 mg, 2.5 mmol) was added to the solution and the mixture was stirred at rt for 6 h and the solution was concentrated in vacuo. Et\(_2\)O (10 mL) was added and the mixture was stirred at r.t. for 10 min to precipitate out an off-white solid. The precipitate was filtered off, washed with Et\(_2\)O, and dried under vacuum to give salt 2p. White solid (647.1 mg, 1.34 mmol, yield: 56\%) m.p. 146-149 \(^{\circ}\)C, (Lit.: 143-146 \(^{\circ}\)C). \(^1\)H NMR (250 MHz, CDCl\(_3\)) \(\delta\) 8.86 (s, 1H), 7.91 (t, J = 8.1 Hz, 5H), 7.60 – 7.21 (m, 4H), 7.03 (d, J = 7.9 Hz, 2H), 6.79 (d, J = 8.9 Hz, 2H), 3.74 (s, 3H), 2.29 (s, 3H) ppm. \(^{13}\)C NMR (63 MHz, CDCl\(_3\)) \(\delta\) 162.81, 140.48, 137.93, 135.05, 134.69, 133.37, 131.99, 130.40, 130.02, 129.08, 128.53, 126.42, 117.90, 115.63, 103.49, 66.27, 55.97, 30.09, 21.70, 15.63 ppm. IR (ATR), 3015, 1570, 1485, 1444, 1301, 1252, 1211, 1190, 1116, 1031, 840, 815, 788, 735, 675 cm\(^{-1}\).

4-Anisyl(4-tolyl)iodonium tosylate (2r)\(^{23}\)

\(p\)-TsOH·H\(_2\)O (190 mg, 1.0 mmol) was added to a suspension of 4-diacetoxyiodotoluene i7 (336 mg, 1.0 mmol) in MeCN (2.5 mL), giving an intense yellow solution, which was immediately diluted with chloroform (25 mL). Anisole (272 \(\mu\)L, 2.5 mmol) was added, and the resultant pale yellow solution was then refluxed for 4 h. The consumption of HTIA was monitored with KI – starch paper. Solvent was then removed under reduced pressure, and the yellow oily residue was triturated with Et\(_2\)O (35 mL). Precipitate was filtered off, washed with Et\(_2\)O, and dried under vacuum for 4 h, and recrystallized from MeOH/Et\(_2\)O to give 4-anisyl(4-tolyl)iodonium tosylate 2r.

White solid (231.1 mg, 0.47 mmol, 47\%) m.p. 147-157 \(^{\circ}\)C, (Lit.: 183-188 \(^{\circ}\)C). \(^1\)H NMR (250 MHz, CD\(_3\)CN) \(\delta\) 7.93 (d, J = 9.0 Hz, 1H), 7.87 (d, J = 8.3 Hz, 1H), 7.41 (d, J = 8.0 Hz, 1H), 7.22 (d, J = 8.2 Hz, 1H), 7.08 (d, J = 7.9 Hz, 1H), 6.93 (d, J = 8.9 Hz, 1H), 3.78 (s, 2H), 2.33 (s, 2H), 2.31 (s, 2H) ppm. \(^{13}\)C NMR (63 MHz, CD\(_3\)CN) \(\delta\) 163.65, 144.32, 139.96, 138.21, 135.80, 133.42, 129.30, 126.55, 118.46, 112.79, 104.51, 56.49, 21.29, 21.20 ppm. IR (ATR), 3088, 3059, 3017, 2981, 2944, 2917, 2839, 1581, 1571, 1486, 1457, 1436, 1408, 1303, 1251, 1212, 1193, 1168, 1116, 1055, 1030, 1022, 1007, 1002, 993, 972, 940, 839, 815, 800 cm\(^{-1}\).
4-Anisyl(4-fluorophenyl)iodonium tosylate (2s)

Anisole (272 μL, 2.5 mmol) was added to a suspension of 1-fluoro-4-[hydroxy(tosyloxy)-iodo]benzene i8 (205 mg, 0.5 mmol) in CHCl₃ (7 mL) and mixture was refluxed for 2 h. Solvent was removed under reduced pressure and the crude oil was triturated with Et₂O (7 mL). The resultant solid was filtered off, washed with Et₂O (7 mL × 2), and dried in vacuo for 4 h to give 4-anisyl(4-fluorophenyl)iodonium tosylate 2s.

White solid (160 mg, 0.32 mmol, yield: 64%) m.p. 133-137 °C, (Lit.: 138-140 °C). ¹H NMR (250 MHz, CDCl₃) δ 8.01 – 7.91 (m, 1H), 7.90 (d,  J = 8.9 Hz, 1H), 7.41 (d,  J = 7.9 Hz, 1H), 6.99 (d,  J = 8.2 Hz, 1H), 6.91 (d,  J = 8.5 Hz, 1H), 6.77 (d,  J = 8.7 Hz, 1H), 3.75 (s, 1H), 2.30 (s, 1H) ppm. ¹⁹F NMR (235 MHz, decoupled, CDCl₃) δ -106.92 ppm. ¹³C NMR (63 MHz, CDCl₃) δ 164.33 (d,  J = 253.5 Hz), 162.25, 142.41, 139.60, 137.64, 137.52, 128.57, 126.02, 118.81 (d,  J = 22.7 Hz), 117.35, 109.95, 105.00, 55.61, 21.32 ppm.

4-Anisyl(2-tolyl)iodonium triflate (2t)

m-Chloroperbenzoic acid (m-CPBA 65% active oxidant, 672 mg, 2.532 mmol) and iodobenzene (260 μL, 2.323 mmol) were dissolved in CH₂Cl₂ (10 mL) in a round bottom flask. The stirred solution was dropwise addition of TfOH (400 μL, 4.554 mmol), resulting in a coloured solution. The reaction mixture was stirred at room temperature and 10 min and cooled to -78 °C followed by dropwise addition of anizole (226 μL, 2.532 mmol) in CH₂Cl₂ (10 mL) was stirred at this temperature and 10 min subsequently concentrated under vacuum (while still cold for low-temperature reactions). Et₂O (10 mL) was added and the mixture was stirred at room temperature for 10 min to precipitate out an off-white solid. To ensure complete precipitation, the flask was stored in the freezer for at least 30 min before the solid was filtered off, washed with Et₂O and dried under vacuum. The crude product was applied on a silica plug (6.0 g) and eluted with CH₂Cl₂ (60 mL) followed by CH₂Cl₂/MeOH (120 mL, 20:1). The latter solution was concentrated, and diethyl ether (10 mL) was added to the residue to induce a precipitation. Precipitate was filtered off, washed with Et₂O, dried under vacuum for 4 h, to give 4-anisyl(2-tolyl)iodonium triflate 2t.

Darkgreen solid (61 mg, 0.13 mmol, yield: 5%). R₉ = 0.58 (DCM-MeOH, 10:1). ¹H NMR (250 MHz, [D₆]DMSO) δ 8.36 (d,  J = 7.9 Hz, 1H), 8.14 (d,  J = 9.0 Hz, 2H), 7.55 (d,  J = 6.1 Hz, 2H), 7.40 – 7.23 (m, 1H), 7.05 (d,  J = 9.0 Hz, 2H), 3.79 (s, 3H), 2.61 (s, 3H) ppm. ¹³C NMR (63 MHz, [D₆]DMSO) δ 161.90, 140.34, 137.08, 136.80, 132.68, 131.33, 129.20, 121.88, 120.68 (d,  J = 322.2 Hz), 117.49, 104.73, 55.68, 24.91 ppm.
Anisyl (2-thienyl)iodonium bromide (2u)⁴

The corresponding 4-diacetoxyiodoanisole (i) (704 mg, 2 mmol) and thiophene (480 µl, 6 mmol) were stirred in 10 ml acetic anhydride at -30 °C. Concentrated sulphuric acid (0.5 ml) was added dropwise over 1 h. The mixture was allowed to warm up to 5 °C and was further stirred at this temperature for 3 to 5 h. The dark solution was poured into 15 ml ice-water. The organic compounds were extracted with ether and discarded. The aqueous layers were treated with activated coal for 10 min at 40 °C to become a clear solution. To the filtered, clear solution 10 ml of a potassium bromide solution (25 %), respectively, was added. After 1h storage in the refrigerator, precipitates were collected, washed with a small portion of acetone and ether. The product 2u was dried and stored in an exsiccator. For eventual recrystallisation the compounds were dissolved in hot methanol and precipitated with ether. White solid (368 mg, 0.92 mmol, yield: 46%) m.p. 175 °C (dec.), (Lit.: 196 °C; 180-185 °C)

₁H NMR (250 MHz, [D₆]DMSO) δ 8.10 (d, J = 8.9 Hz, 2H), 7.87 (dd, J = 8.3, 4.4 Hz, 2H), 7.09 (dd, J = 5.1, 3.8 Hz, 1H), 7.01 (d, J = 9.0 Hz, 2H), 3.78 (s, 3H) ppm.

₁₃C NMR (63 MHz, [D₆]DMSO) δ 161.44, 138.55, 136.55, 135.70, 129.13, 117.02, 110.67, 105.96, 55.63 ppm.

IR (ATR), 1480, 1472, 1382, 1217, 1210, 1180, 1112, 1081, 1074, 1046, 999, 965, 938, 913, 840, 833, 803, 793, 717, 707, 688, 675, 661, 650, 642, 622, 611, 604, 596, 590, 574, 563 cm⁻¹.

4-Tolyl(phenyl)iodonium triflate (2v)²¹

m-Chloroperbenzoic acid (m-CPBA 70% active oxidant, 624 mg, 2.532 mmol) and iodobenzene (260 µL, 2.323 mmol) were dissolved in CH₂Cl₂ (10 mL) in a 25 mL round bottom flask. The toluene (270 µL, 2.532 mmol) was added and the solution was cooled to 0 °C followed by dropwise addition of TfOH (400 µL, 4.554 mmol), resulting in a slight heat increase and change of color from a clear to a yellow transparent solution. The solution was stirred at 0 °C and 10 min and concentrated in vacuo while still cold. Et₂O (1-2 mL) was added and the mixture was stirred at rt for 10 min to precipitate out an off-white solid. The flask was stored in the freezer for 30 min, then the solid was filtered off, washed with cold Et₂O and dried under vacuum to give diaryliodonium salt as a slightly off-white solid 2v. Offwhite solid (835 mg, 2.97 mmol, yield: 99%) m.p. 122-126 °C, (Lit.: 120-121°C, 122-125 °C). ₁H NMR (250 MHz, [D₆]DMSO) δ 8.21 (d, J = 7.4 Hz, 1H), 8.12 (d, J = 8.3 Hz, 1H), 7.65 (t, J = 7.4 Hz, 1H), 7.51 (t, J = 7.6 Hz, 1H), 7.33 (d, J = 8.2 Hz, 1H), 2.33 (s, 1H) ppm. ₁₃C NMR (63 MHz, [D₆]DMSO) δ 142.71, 135.26, 135.11, 132.48, 132.07, 131.81, 120.75 (d, J = 322.3 Hz), 116.71, 112.94, 20.93 ppm. IR (ATR), 3099, 3065, 2904, 1304, 1173, 1013 cm⁻¹.
3-Tolyl(phenyl)iodonium triflate (2w)

*m*-Chloroperbenzoic acid (*m*-CPBA 70% active oxidant, 640 mg, 3.0 mmol) was dissolved in CH₂Cl₂ (10 mL). To the solution was added iodobenzene (310 μL, 2.7 mmol) followed by slow addition of BF₃ · OEt₂ (850 μL, 6.8 mmol) at room temperature. The resulting yellow solution was stirred at rt for 30 min and then cooled to 0 °C, and 3-tolylboronic acid (370 mg, 3.0 mmol) was added. After 15 min of stirring at rt, triflic acid (240 μL, 2.75 mmol) was added at room temperature, and the mixture was stirred for an additional 15 min. The crude reaction mixture was applied on a silica plug (6.0 g) and eluted with CH₂Cl₂ (60 mL) followed by CH₂Cl₂-MeOH 20:1 (120 mL). The latter solution was concentrated, and diethyl ether (10 mL) was added to the residue to induce a precipitation. The solution was allowed to stir for 15 min, and then the ether phase was decanted. The solid was washed twice more with diethyl ether (2 × 10 mL) and then dried in vacuo to give salt 2w.

White solid (210 mg, 0.47 mmol, yield: 18%) m.p. 139-147 °C, (Lit. 151-153 °C).

IR (ATR), 1597, 1558, 1470, 1442, 1267, 1189, 1165, 1092, 1060, 1041, 1023, 990, 914, 816, 769, 758, 740, 679, 674, 650, 634, 606 cm⁻¹.

1H NMR (250 MHz, [D₆]DMSO) δ 8.23 (d, J = 7.9 Hz, 1H), 8.11 (s, 1H), 8.05 (d, J = 7.7 Hz, 1H), 7.67 (t, J = 7.1 Hz, 1H), 7.63 – 7.34 (m, 2H) ppm, 2.34 (s, 1H).

13C NMR (63 MHz, [D₆]DMSO) δ 141.79, 135.31, 135.11, 132.72, 132.23, 132.01, 131.74, 131.44, 121.99 (d, J = 316.6 Hz), 116.35, 116.24, 20.73 ppm.

2-Tolyl(phenyl)iodonium triflate (2x)

*m*-Chloroperbenzoic acid (*m*-CPBA 70% active oxidant, 616 mg, 2.5 mmol) and 2-iodotoluene (318 μL, 2.5 mmol) were dissolved in CH₂Cl₂ (10 mL) in a 25 mL round bottom flask. The benzene (233 μL, 2.5 mmol) was added and the solution was cooled to 0 °C followed by dropwise addition of TfOH (440 μL, 5.0 mmol), resulting in a slight heat increase and change of color from a clear to a yellow transparent solution. The solution was stirred at room temperature and 30 min and concentrated in vacuo while still cold. Et₂O (1-2 mL) was added and the mixture was stirred at rt for 10 min to precipitate out an off-white solid. The flask was stored in the freezer for 30 min, then the solid was filtered off, washed with cold Et₂O and dried under vacuum to give diaryliodonium salt as a slightly off-hite solid 2x.

Off-white solid (939 mg, 2.11 mmol, yield: 85%) m.p. 162-166 °C, (Lit.: 162-164 °C).

1H NMR (250 MHz, [D₆]DMSO) δ 8.40 (d, J = 7.9 Hz, 1H), 8.21 (d, J = 7.9 Hz, 2H), 7.65 (t, J = 7.3 Hz, 1H), 7.60 – 7.45 (m, 6H), 7.32 (dd, J = 8.1, 3.1 Hz, 1H), 2.62 (s, 3H) ppm.

13C NMR (63 MHz, [D₆]DMSO) δ 140.63, 137.12, 135.09, 132.90, 132.02, 131.85, 131.47, 129.33, 123.30, 121.42, 120.74 (q, J = 322.2 Hz), 118.18, 115.86, 25.03 ppm. IR (ATR), 3079, 3056, 1469, 1457, 1443, 1266, 1247, 1220, 1161, 1065, 1025, 1000, 989, 984, 948, 924, 757, 751, 745, 680, 652, 634, 571, 537, 514, 495, 452, 428 cm⁻¹.
3,5-Xylyl(phenyl)iodonium triflate (2y)

*m*-Chloroperbenzoic acid (*m*-CPBA 70% active oxidant, 616 mg, 2.5 mmol) was dissolved in CH$_2$Cl$_2$ (10 mL). To the solution was added iodobenzene 250 μL, 2.24 mmol) followed by slow addition of BF$_3$·OEt$_2$ (690 μL, 5.6 mmol) at room temperature. The resulting yellow solution was stirred at rt for 30 min and then cooled to 0 °C, and 3,5-dimethylphenylboronic acid (375 mg, 2.5 mmol) was added. After 15 min of stirring at rt, triflic acid (240 μL, 2.75 mmol) was added at room temperature, and the mixture was stirred for an additional 15 min. The crude reaction mixture was applied on a silica plug (6.0 g) and eluted with CH$_2$Cl$_2$ (60 mL) followed by CH$_2$Cl$_2$/MeOH (120 mL, 20:1). The latter solution was concentrated, and diethyl ether (10 mL) was added to the residue to induce a precipitation. The solution was allowed to stir for 15 min, and then the ether phase was decanted. The solid was washed twice more with diethyl ether (2 × 10 mL) and then dried in vacuo to give salt 2y.

White solid (305 mg, 0.66 mmol, yield: 27%) m.p. 147-151 °C, $R_f = 0.15$ (DCM-MeOH, 10:1). $^1$H NMR (250 MHz, [D$_6$]DMSO) $\delta$ 8.21 (d, $J = 7.5$ Hz, 2H), 7.90 (s, 2H), 7.66 (t, $J = 7.4$ Hz, 1H), 7.52 (t, $J = 7.6$ Hz, 2H), 7.29 (s, 1H), 2.29 (s, 6H) ppm. $^{13}$C NMR (63 MHz, [D$_6$]DMSO) $\delta$ 141.40, 135.05, 133.46, 132.42, 131.95, 131.70, 120.04 (d, $J = 299.5$ Hz), 116.18, 115.97, 20.60 ppm. IR (ATR), 3462, 1602, 1560, 1472, 1444, 1305, 1268, 1239, 1222, 1201, 1171, 1043, 1024, 990, 842, 778, 758, 741, 672, 647, 635, 607, 591, 573, 543, 514, 486, 468, 447, 436 cm$^{-1}$. HRMS calcd for C$_{14}$H$_{14}$I [M-OTf]$^+$ 309.0140 found 309.0149.

2,4-Xylyl(phenyl)iodonium triflate (2z)$^{21}$

*m*-Chloroperbenzoic acid (*m*-CPBA 70% active oxidant, 616 mg, 2.5 mmol) and iodobenzene (280 μL, 2.5 mmol) were dissolved in CH$_2$Cl$_2$ (10 mL) in a 25 mL round bottom flask. The *m*-xylene (307 μL, 2.5 mmol) was added and the solution was cooled to 0 °C followed by dropwise addition of TfOH (440 μL, 5.0 mmol), resulting in a slight heat increase and change of color from a clear to a yellow transparent solution. The solution was stirred at 0 °C and 2 h and concentrated in vacuo while still cold. Et$_2$O (1-2 mL) was added and the mixture was stirred at rt for 10 min to precipitate out an off-white solid. The flask was stored in the freezer for 30 min, then the solid was filtered off, washed with cold Et$_2$O and dried under vacuum to give diaryliodonium salt as a slightly off-white solid 2z.

Offwhite solid (790 mg, 1.72 mmol, yield: 69%) m.p. 127-128 °C, (Lit. 128-129 °C). $^1$H NMR (250 MHz, [D$_6$]DMSO) $\delta$ 8.25 (d, $J = 8.1$ Hz, 1H), 8.17 (d, $J = 7.4$ Hz, 2H), 7.65 (t, $J = 7.4$ Hz, 1H), 7.51 (t, $J = 7.6$ Hz, 2H), 7.37 (s, 1H), 7.13 (d, $J = 7.9$ Hz, 1H), 2.56 (s, 3H), 2.31 (s, 3H) ppm. $^{13}$C NMR (63 MHz, [D$_6$]DMSO) $\delta$ 143.30, 140.39, 137.00, 134.87, 132.05, 131.88, 131.76, 129.94, 117.71, 115.92, 24.79, 20.68 ppm. IR (ATR), 3083, 3057, 1595.
2,5-Xylyl(phenyl)iodonium triflate (2aa)

*m*-Chloroperbenzoic acid (*m*-CPBA 70% active oxidant, 616 mg, 2.5 mmol) and iodobenzene (280 µL, 2.5 mmol) were dissolved in CH₂Cl₂ (10 mL) in a 25 mL round bottom flask. The *p*-xylene (308 µL, 2.5 mmol) was added and the solution was cooled to 0 °C followed by dropwise addition of TfOH (440 µL, 5.0 mmol), resulting in a slight heat increase and change of color from a clear to a yellow transparent solution. The solution was stirred at room temperature and 5 h and concentrated in vacuo while still cold. Et₂O (1-2 mL) was added and the mixture was stirred at rt for 10 min to precipitate out an off-white solid. The flask was stored in the freezer for 30 min, then the solid was filtered off, washed with cold Et₂O and dried under vacuum to give diaryliodonium salt as a slightly off-white solid 2aa.

Off-white solid (640 mg, 1.40 mmol, yield: 56%) m.p. 127-128 °C. ¹H NMR (250 MHz, [D₆]DMSO) δ 8.25 (s, 1H), 8.19 (d, *J* = 7.4 Hz, 2H), 7.66 (t, *J* = 7.4 Hz, 1H), 7.52 (t, *J* = 7.6 Hz, 2H), 7.43 (t, *J* = 6.1 Hz, 2H), 2.56 (s, 3H), 2.31 (s, 3H) ppm. ¹³C NMR (63 MHz, [D₆]DMSO) δ 139.12, 137.37, 137.10, 135.00, 133.48, 131.95, 131.02, 121.03, 115.62, 24.42, 20.02 ppm. IR (ATR), 3055, 2990, 2931, 1580, 1560, 1492, 1472, 1458, 1444, 1384, 1268, 1238, 1222, 1162, 1067, 1023, 987, 928, 878, 842, 816, 759, 742, 680, 666, 650, 635, 572, 515, 455, 429, 415 cm⁻¹.

2-Fluorophenyl(phenyl)iodonium triflate (2ab)

*m*-Chloroperbenzoic acid (*m*-CPBA 70% active oxidant, 511 mg, 2.074 mmol), 2-iodofluorobenzene (220 µL, 1.886 mmol) and benzene (185 µL, 2.074 mmol) were dissolved in CH₂Cl₂ (10 mL) in a round bottom flask. The stirred solution was cooled to 0 °C followed by dropwise addition of TfOH (199 µL, 2.263 mmol), resulting in a coloured solution. The reaction mixture was stirred at room temperature for 2 h and concentrated under vacuum (while still cold for low-temperature reactions). Et₂O (10 mL) was added and the mixture was stirred at room temperature for 10 min to precipitate out an off-white solid. The solid 2ab was filtered off, washed with cold Et₂O and dried under vacuum.

Off-white solid (438.2 mg, 0.98 mmol, yield: 52%) m.p. 171-174 °C ¹H NMR (250 MHz, [D₆]DMSO) δ 8.50 – 8.34 (m, 1H), 8.23 (d, *J* = 7.6 Hz, 2H), 7.82 – 7.47 (m, 5H), 7.43 – 7.32 (m, 1H) ppm. ¹³C NMR (63 MHz, [D₆]DMSO) δ 159.20 (d, *J* = 249.3 Hz), 137.15, 135.64 (d, *J* = 8.0 Hz), 135.16, 132.28, 131.96, 127.67 (d, *J* = 3.2 Hz), 120.71 (q, *J* = 322.1 Hz), 117.10, 116.81, 116.74, 103.82 (d, *J* = 23.5 Hz) ppm. IR (ATR), 3088, 3065, 1590, 1579, 1475, 1443, 1313, 1268, 1243, 1234, 1219, 1169, 1161, 1091, 1064, 1023, 990, 949, 924, 821, 774, 754, 745, 735, 678, 653, 634, 607, 574, 533, 514, 461 cm⁻¹.
2-Chlorophenyl(phenyl)iodonium triflate (2ac)\textsuperscript{21}

\textit{m}-Chloroperbenzoic acid (\textit{m}-CPBA 70\% active oxidant, 678 mg, 2.75 mmol), 2-chloroiodobenzene (305 μL, 2.5 mmol) and benzene (246 μL, 2.75 mmol) were dissolved in CH\textsubscript{2}Cl\textsubscript{2} (10 mL) in a round bottom flask. The stirred solution was cooled to 0 °C followed by dropwise addition of TfOH (263 μL, 3.0 mmol), resulting in a coloured solution. The reaction mixture was stirred at room temperature for 2 h and concentrated under vacuum (while still cold). Et\textsubscript{2}O (10 mL) was added and the mixture was stirred at room temperature for 10 min to precipitate out an off-white solid. The solid 2ac was filtered off, washed with Et\textsubscript{2}O and dried under vacuum.

Off-white solid (672.9 mg, 1.45 mmol, yield: 58\%) m.p. 186-190 °C. \textsuperscript{1}H NMR (250 MHz, [D₆]DMSO) δ 8.60 – 8.53 (m, 1H), 8.22 (d, J = 7.5 Hz, 2H), 7.87 – 7.81 (m, 1H), 7.75 – 7.63 (m, 2H), 7.51 (ddd, J = 12.8, 9.5, 4.5 Hz, 3H) ppm. \textsuperscript{13}C NMR (63 MHz, [D₆]DMSO) δ 138.70, 135.89, 135.13, 134.67, 132.29, 131.94, 130.33, 130.27, 119.52, 116.84 ppm. IR (ATR), 3086, 3060, 1571, 1561, 1469, 1453, 1442, 1431, 1313, 1315, 1265, 1243, 1219, 1168, 1131, 1116, 1102, 1064, 1023, 1009, 1001, 988, 974, 952, 924, 865, 759, 750, 744, 720, 679, 652 cm\textsuperscript{-1}.

2-Bromophenyl(phenyl)iodonium triflate (2ad)\textsuperscript{19}

\textit{m}-Chloroperbenzoic acid (\textit{m}-CPBA 70\% active oxidant, 740 mg, 3.0 mmol) was dissolved in CH\textsubscript{2}Cl\textsubscript{2} (10 mL). To the solution was added iodobenzene (310 μL, 2.7 mmol) followed by slow addition of BF\textsubscript{3}-OEt\textsubscript{2} (850 μL, 6.8 mmol) at room temperature. The resulting yellow solution was stirred at rt for 30 min and then cooled to 0 °C, and 2-bromophenylboronic acid (602 mg, 3.0 mmol) was added. After 15 min of stirring at rt, triflic acid (24 μL, 0.27 mmol) was added at room temperature, and the mixture was stirred for an additional 15 min. The crude reaction mixture was applied on a silica plug (6.0 g) and eluted with CH\textsubscript{2}Cl\textsubscript{2} (60 mL) followed by CH\textsubscript{2}Cl\textsubscript{2}/MeOH (120 mL, 20:1). The latter solution was concentrated, and diethyl ether (10 mL) was added to the residue to induce a precipitation. The solution was allowed to stir for 15 min, and then the ether phase was decanted. The solid was washed twice more with diethyl ether (2 × 10 mL) and then dried in vacuo to give salt 2ad.

White solid (540 mg, 1.06 mmol, yield: 39\%) m.p. 188-191 °C. \textit{Rf} = 0.15 (DCM-MeOH, 10:1). \textsuperscript{1}H NMR (250 MHz, [D₆]DMSO) δ 8.55 (d, J = 6.9 Hz, 1H), 8.20 (d, J = 7.9 Hz, 2H), 7.96 (d, J = 7.7 Hz, 1H), 7.68 (t, J = 7.3 Hz, 1H), 7.63 – 7.47 (m, 4H) ppm. \textsuperscript{13}C NMR (63 MHz, [D₆]DMSO) δ 139.06, 135.08, 134.67, 133.72, 132.37, 132.01, 130.76, 127.01, 122.61, 120.73 (d, J = 322.4 Hz), 117.05 ppm. IR (ATR), 3085, 3058, 1567, 1469, 1455, 1442, 1428, 1265, 1244, 1220, 1165, 1125, 1106, 1094, 1065, 1023, 1008, 996, 987, 952, 924, 758, 750, 744, 692, 679, 652, 636, 606, 572, 515 cm\textsuperscript{-1}.
4-Fluorophenyl(phenyl)iodonium triflate (2ae)¹⁸

*m*-Chloroperbenzoic acid (*m*-CPBA 70% active oxidant, 794 mg, 3.22 mmol), iodobenzene (319 μL, 2.849 mmol) and fluorobenzene (302 μL, 3.22 mmol) were dissolved in CH₂Cl₂ (10 mL) in a round bottom flask. The stirred solution was cooled to 0°C followed by dropwise addition of TfOH (750 μL, 8.547 mmol), resulting in a coloured solution. The reaction mixture was stirred at room temperature for 1 h and concentrated under vacuum (while still cold). Et₂O (10 mL) was added and the mixture was stirred at room temperature for 10 min to precipitate out an off-white solid. The solid 2ae was filtered off, washed with Et₂O and dried under vacuum.

Off-white solid (1.16 g, 2.59 mmol, yield: 91%) m.p. 163-167 °C, (Lit.: 133-134 °C). ¹H NMR (250 MHz, [D₆]DMSO) δ 8.33 (dd, J = 8.7, 5.1 Hz, 1H), 8.25 (d, J = 7.6 Hz, 1H), 7.67 (t, J = 7.3 Hz, 1H), 7.53 (t, J = 7.6 Hz, 1H), 7.41 (t, J = 8.8 Hz, 1H) ppm. ¹³C NMR (63 MHz, [D₆]DMSO) δ 163.99 (d, J = 251.4 Hz), 138.09 (d, J = 9.1 Hz), 135.09, 132.12, 131.80, 120.70 (d, J = 322.3 Hz), 119.23 (d, J = 22.8 Hz), 116.93, 110.75 (d, J = 3.1 Hz) ppm. IR (ATR), 3092, 3058, 1574, 1478, 1473, 1445, 1400, 1298, 1267, 1248, 1167, 1162, 1108, 1094, 1023, 991, 982, 914, 845, 821, 807, 758, 745, 730, 676, 654, 634, 622, 576 cm⁻¹.

4-Chlorophenyl(phenyl)iodonium triflate (2af)¹⁸

*m*-Chloroperbenzoic acid (*m*-CPBA 65% active oxidant, 772 mg, 3.134 mmol) and 1-chloro-4-iodobenzene (679 mg, 2.849 mmol) were dissolved in CH₂Cl₂ (10 mL) in a round bottom flask. The benzene (277 μL, 3.100 mmol) was added and the solution was cooled to 0 °C followed by dropwise addition of TfOH (750 μL, 8.547 mmol), resulting in a coloured solution. The reaction mixture was stirred at room temperature and 17 h and subsequently concentrated under vacuum (while still cold for low-temperature reactions). Et₂O (10 mL) was added and the mixture was stirred at room temperature for 10 min to precipitate out an off-white solid. To ensure complete precipitation, the flask was stored in the freezer for at least 30 min before the solid was filtered off, washed with Et₂O and dried under vacuum to give diaryliodonium salt 2af.

Off-white solid (683.3 mg, 1.47 mmol, yield: 65%) m.p. 89-91 °C, (Lit.: 108-110 °C, 110-111 °C). ¹H NMR (250 MHz, CDCl₃) δ 7.99 (dd, J = 14.0, 8.3 Hz, 1H), 7.59 (t, J = 7.3 Hz, 1H), 7.40 (dd, J = 18.6, 8.3 Hz, 1H) ppm. ¹³C NMR (63 MHz, CDCl₃) δ 140.04, 137.32, 136.01, 133.18, 132.88, 132.81, 114.64, 111.36 ppm. IR (ATR), 3088, 3066, 2874, 2658, 2548, 1689, 1596, 1572, 1469, 1443, 1417, 1392, 1300, 1265, 1240, 1221, 1183, 1163, 1105, 1090, 1026, 998, 987, 916, 897, 850, 812, 804, 758, 740, 720, 677 cm⁻¹.
4-Bromophenyl(phenyl)iodonium triflate (2ag)\textsuperscript{18}

\textit{m}-Chloroperbenzoic acid (\textit{m}-CPBA 65% active oxidant, 772 mg, 3.134 mmol) and \textit{1}-bromo-4-iodobenzene (806 mg, 2.849 mmol) were dissolved in CH\textsubscript{2}Cl\textsubscript{2} (10 mL) in a round bottom flask. The benzene (277 \textmu L, 3.100 mmol) was added and the solution was cooled to 0 °C followed by dropwise addition of TfOH (750 \textmu L, 8.547 mmol), resulting in a coloured solution. The reaction mixture was stirred at room temperature and 22 h and subsequently concentrated under vacuum (while still cold for low-temperature reactions). Et\textsubscript{2}O (10 mL) was added and the mixture was stirred at room temperature for 10 min to precipitate out an off-white solid. To ensure complete precipitation, the flask was stored in the freezer for at least 30 min before the solid was filtered off, washed with Et\textsubscript{2}O and dried under vacuum to give diaryliodonium salt 2ag.

Off-white solid (877.5 g, 1.73 mmol, yield: 61%) m.p. 120-122 °C, (Lit.: 131-132 °C, 129-136 °C). \textsuperscript{1}H NMR (250 MHz, CDCl\textsubscript{3}) \textit{δ} 8.01 (d, \textit{J} = 7.5 Hz, 1H), 7.88 (d, \textit{J} = 8.7 Hz, 1H), 7.68 – 7.47 (m, 2H), 7.43 (t, \textit{J} = 7.7 Hz, 1H) ppm. \textsuperscript{13}C NMR (63 MHz, CDCl\textsubscript{3}) \textit{δ} 136.86, 135.53, 135.32, 132.68, 132.32, 127.88, 114.09, 111.77 ppm. IR (ATR), 3082, 3056, 1470, 1445, 1384, 1276, 1217, 1182, 1167, 1064, 1017, 994, 983, 917, 820, 813, 757, 731, 675, 651, 631, 573, 514, 482, 446 cm\textsuperscript{-1}.

4-Chlorophenyl(4-fluorophenyl)iodonium triflate (2ah)

\textit{m}-Chloroperbenzoic acid (\textit{m}-CPBA 70% active oxidant, 382 mg, 1.55 mmol), 4-iodochlorobenzene (339 mg, 1.424 mmol) and fluorobenzene (145 \textmu L, 1.55 mmol) were dissolved in CH\textsubscript{2}Cl\textsubscript{2} (10 mL) in a round bottom flask. The stirred solution was cooled to 0 °C followed by dropwise addition of TfOH (375 \textmu L, 4.272 mmol), resulting in a coloured solution. The reaction mixture was stirred at room temperature for 24 h and concentrated under vacuum (while still cold). Et\textsubscript{2}O (10 mL) was added and the mixture was stirred at room temperature for 10 min to precipitate out a solid, 2ah was filtered off, washed with Et\textsubscript{2}O and dried under vacuum.

White solid (547.4 mg, 1.13 mmol, yield: 80%) m.p. 116-121 °C. \textsuperscript{1}H NMR (250 MHz, [D\textsubscript{6}]DMSO) \textit{δ} 8.36 – 8.28 (m, 2H), 8.27 – 8.21 (m, 2H), 7.68 – 7.57 (m, 2H), 7.42 (t, \textit{J} = 8.9 Hz, 2H) ppm. \textsuperscript{13}C NMR (63 MHz, [D\textsubscript{6}]DMSO) \textit{δ} 164.02 (d, \textit{J} = 251.8 Hz), 138.10 (d, \textit{J} = 9.0 Hz), 137.43, 136.90, 131.78, 120.70 (d, \textit{J} = 322.5 Hz), 119.29 (d, \textit{J} = 22.8 Hz), 114.79, 111.08 (d, \textit{J} = 3.1 Hz) ppm. \textsuperscript{19}F NMR (235 MHz, DMSO-\textit{d}_6) \textit{δ} -77.77, -106.51 ppm. IR (ATR), 3092, 1577, 1560, 1484, 1472, 1402, 1392, 1290, 1257, 1229, 1219, 1182, 1162, 1154, 1111, 1087, 1052, 1021, 1003, 994, 830, 823, 806, 757, 724, 634, 574, 516, 505, 482, 414 cm\textsuperscript{-1}. HRMS calcd for C\textsubscript{12}H\textsubscript{8}ClFI [M-OTf]\textsuperscript{+} 332.9343 found 332.9347
**4-Bromophenyl(4-fluorophenyl)iodonium triflate (2ai)**

*m*-Chloroperbenzoic acid (*m*-CPBA 70% active oxidant, 382 mg, 1.55 mmol), 4-iodo-bromobenzene (403 mg, 1.424 mmol) and fluorobenzene (145 μL, 1.55 mmol) were dissolved in CH2Cl2 (10 mL) in a round bottom flask. The stirred solution was cooled to 0 °C followed by dropwise addition of TfOH (375 μL, 4.272 mmol), resulting in a coloured solution. The reaction mixture was stirred at room temperature for 24 h and concentrated under vacuum (while still cold). Et2O (10 mL) was added and the mixture was stirred at room temperature for 10 min to precipitate out as a solid. 2ai was filtered off, washed with Et2O and dried under vacuum.

White solid (612.7 mg, 1.16 mmol, yield: 82%) m.p. 145-146 °C. 1H NMR (250 MHz, [D6]DMSO) δ 8.36 – 8.27 (m, 2H), 8.21 – 8.11 (m, 2H), 7.83 – 7.70 (m, 2H), 7.42 (t, *J* = 8.9 Hz, 2H) ppm. 13C NMR (63 MHz, [D6]DMSO) δ 164.02 (d, *J* = 251.7 Hz), 138.12 (d, *J* = 9.0 Hz), 136.99, 134.67, 126.29, 120.69 (d, *J* = 322.2 Hz), 119.29 (d, *J* = 22.8 Hz), 115.49, 111.03 (d, *J* = 3.0 Hz) ppm. 19F NMR (235 MHz, [D6]DMSO) δ -77.77, -106.51 ppm. IR (ATR), 1574, 1559, 1481, 1473, 1401, 1385, 1299, 1266, 1247, 1229, 1184, 1168, 1162, 1107, 1093, 1066, 1027, 1002, 993, 958, 843, 816, 804, 758, 700, 632, 576, 537, 516, 505 cm⁻¹. HRMS calcd for C12H8BrFIO [M-OTf]⁺ 376.8838 found 376.8852

**2-Tolyl(4-fluorophenyl)iodonium triflate (2aj)**

*m*-Chloroperbenzoic acid (*m*-CPBA 70% active oxidant, 286 mg, 1.66 mmol), 2-iodotoluene (192 μL, 1.51 mmol) and fluorobenzene (156 μL, 1.66 mmol) were dissolved in CH2Cl2 (10 mL) in a round bottom flask. The stirred solution was cooled to 0 °C followed by dropwise addition of TfOH (26μL, 3.0 mmol), resulting in a coloured solution. The reaction mixture was stirred at room temperature for 24 h and concentrated under vacuum (while still cold for low-temperature reactions). Et2O (10 mL) was added and the mixture was stirred at room temperature for 10 min to precipitate out an off-white solid. The solid 2aj was filtered off, washed with Et2O and dried under vacuum.

Off-white solid (618.0 mg, 1.33 mmol, yield: 88.5%) m.p. 165-168 °C, (Lit.: 161-162 °C). 1H NMR (250 MHz, [D6]DMSO) δ 8.40 (d, *J* = 8.0 Hz, 1H), 8.29 (dd, *J* = 9.0, 5.1 Hz, 2H), 7.65 – 7.52 (m, 2H), 7.40 (t, *J* = 8.9 Hz, 2H), 7.35 – 7.24 (m, 1H) ppm. 13C NMR (63 MHz, [D6]DMSO) δ 163.93 (d, *J* = 251.5 Hz), 140.55, 137.95 (d, *J* = 9.1 Hz), 136.98, 132.93, 131.48, 129.33, 121.80, 120.70 (d, *J* = 322.5 Hz), 119.26 (d, *J* = 22.9 Hz), 110.07 (d, *J* = 3.0 Hz), 24.97 ppm. IR (ATR), 3095, 3055, 1574, 1481, 1457, 1399, 1383, 1268, 1251, 1234, 1220, 1161, 1093, 1025, 1003, 996, 948, 841, 823, 806, 751, 688, 634, 574, 536, 516, 508, 453, 433 cm⁻¹.
4-Tolyl(4-fluorophenyl)iodonium trflate (2ak)\textsuperscript{18}

\textit{m}-Chloroperbenzoic acid (\textit{m}-CPBA 70\% active oxidant, 794 mg, 3.22 mmol), 4-iodotoluene (621 mg, 2.849 mmol) and fluorobenzene (302 \(\mu\)L, 3.22 mmol) were dissolved in CH\(_2\)Cl\(_2\) (10 mL) in a round bottom flask. The stirred solution was cooled to 0 °C followed by dropwise addition of TfOH (500 \(\mu\)L, 5.698 mmol), resulting in a coloured solution. The reaction mixture was stirred at room temperature for 1 h and concentrated under vacuum (while still cold). Et\(_2\)O (10 mL) was added and the mixture was stirred at room temperature for 10 min to precipitate out an off-white solid. The solid \textbf{2ak} was filtered off, washed with Et\(_2\)O and dried under vacuum.

Off-white solid (785.8 mg, 1.70 mmol, yield: 60\%) m.p. 145-147 °C. \(^1\)H NMR (250 MHz, [D\(_6\)]DMSO) \(\delta\) 8.34 – 8.22 (m, 2H), 8.12 (d, \(J = 8.3\) Hz, 2H), 7.38 (dd, \(J = 18.2, 8.9\) Hz, 4H), 2.34 (s, 3H) ppm. \(^{13}\)C NMR (63 MHz, [D\(_6\)]DMSO) \(\delta\) 163.90 (d, \(J = 251.0\) Hz), 142.63, 137.87 (d, \(J = 8.9\) Hz), 135.04, 132.37, 130.36, 120.70 (d, \(J = 325.9\) Hz), 119.13 (d, \(J = 22.8\) Hz), 113.25, 110.83 (d, \(J = 3.0\) Hz), 20.83 ppm.

IR (ATR), 3093, 3058, 1575, 1482, 1477, 1401, 1300, 1268, 1249, 1230, 1221, 1187, 1162, 1093, 1026, 998, 844, 817, 807, 796, 785, 757, 635, 621, 575, 516, 506, 476, 437 cm\(^{-1}\).

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2-Azidomethylphenyl(4-anisyl)iodonium Tosylate (2al)\textsuperscript{25}

\textit{m}-Chloroperbenzoic acid (\textit{m}-CPBA 70\% active oxidant, 271 mg, 1.1 mmol) was added to a stirred solution of 1-azidomethyl-2-iodobenzene (259 mg, 1.0 mmol) in CHCl\(_3\) (10 mL) at room temp. The mixture was stirred at rt until it became a pale-yellow solution (ca. 15 min). \textit{p}-TsOH·H\(_2\)O (209 mg, 1.1 mmol) was added followed by anisole (1.087 mL, 10 mmol). The reaction mixture was gradually heated to 40 °C and held at this temperature for about 4 h as the mixture became a yellow solution. The consumption of 1-azidomethyl-2-iodobenzene was monitored by TLC (hexane) and formation of the intermediate [hydroxy(tosyl)iodo]arene was verified with KI starch paper. The solvent was then removed in vacuo. The residual yellow oil was triturated with Et\(_2\)O to give a solid that was washed with Et\(_2\)O and recrystallized from MeOH/Et\(_2\)O to give 2-azidomethylphenyl(4-anisyl)iodonium tosylate \textbf{2al} as a white solid.

White solid (298.2 mg, 0.55 mmol, yield: 55\%) m.p. 167-170 °C, (Lit.: 167-169 °C) \(^1\)H NMR (250 MHz, CDCl\(_3\)) \(\delta\) 8.34 (d, \(J = 8.0\) Hz, 1H), 8.13 (d, \(J = 8.7\) Hz, 2H), 7.79 – 7.64 (m, 4H), 7.51 (d, \(J = 8.1\) Hz, 1H), 7.24 (d, \(J = 7.7\) Hz, 2H), 7.08 (d, \(J = 8.7\) Hz, 2H), 4.78 (s, 2H), 3.87 (s, 3H), 2.39 (s, 3H) ppm. \(^{13}\)C NMR (63 MHz, CDCl\(_3\)) \(\delta\) 164.52, 143.60, 141.62, 139.10, 138.67, 138.50, 133.11, 135.07, 129.80, 126.94, 119.66, 118.85, 104.41, 57.26, 56.36, 21.29 ppm. IR (ATR), 3065, 3006, 2967, 2842, 2128, 2092, 2076, 1587, 1575, 1489, 1457, 1442, 1428, 1405, 1297, 1260, 1242, 1213, 1198, 1172, 1119, 1057, 1049, 1033, 1009, 993, 974, 948, 925, 898, 882, 846, 819, 802, 787, 765, 720, 710, 681, 646, 624, 617, 588, 577, 563, 550, 509, 491, 449, 407 cm\(^{-1}\).
2,6-Xylyl(phenyl)iodonium triflate (2am)

m-Chloroperbenzoic acid (m-CPBA 70% active oxidant, 616 mg, 2.5 mmol) was dissolved in CH₂Cl₂ (10 mL). To the solution was added iodobenzene 250 μL, 2.24 mmol) followed by slow addition of BF₃·OEt₂ (690 μL, 5.6 mmol) at room temperature. The resulting yellow solution was stirred at rt for 30 min and then cooled to 0 °C, and 2,6-dimethylphenylboronic acid (375 mg, 2.5 mmol) was added. After 15 min of stirring at rt, triflic acid (240 μL, 2.75 mmol) was added at room temperature, and the mixture was stirred for an additional 15 min. The crude reaction mixture was applied on a silica plug (6.0 g) and eluted with CH₂Cl₂ (60 mL) followed by CH₂Cl₂/MeOH (120 mL, 20:1). The latter solution was concentrated, and diethyl ether (10 mL) was added to the residue to induce a precipitation. The solution was allowed to stir for 15 min, and then the ether phase was decanted. The solid was washed twice more with diethyl ether (2 × 10 mL) and then dried in vacuo to give salt 2am.

White solid (358 mg, 0.78 mmol, yield: 31%) m.p. 152-158 °C, (Lit. 137-138 °C). Rᵢ = 0.15 (DCM-MeOH, 10:1). ¹H NMR (250 MHz, [D₆]DMSO) δ 8.01 (d, J = 7.7 Hz, 2H), 7.65 (t, J = 7.3 Hz, 1H), 7.58 – 7.46 (m, 3H), 7.40 (d, J = 7.1 Hz, 2H), 2.65 (s, 6H) ppm. ¹³C NMR (63 MHz, [D₆]DMSO) δ 141.76, 134.67, 132.84, 131.92, 129.13, 126.22, 123.25, 114.37, 26.52 ppm. IR (ATR), 3477, 1642, 1564, 1469, 1459, 1385, 1258, 1239, 1177, 1160, 1063, 1024, 975, 970, 796, 757, 735, 693, 674, 651, 634, 572, 550, 514, 490, 446 cm⁻¹.

2,4-Difluorophenyl(phenyl)iodonium triflate (2an)

m-Chloroperbenzoic acid (m-CPBA 70% active oxidant, 772 mg, 3.133 mmol), 2,4-difluoroiodobenzene (341 μL, 2.848 mmol) and benzene (280 μL, 3.133 mmol) were dissolved in CH₂Cl₂ (10 mL) in a round bottom flask. The stirred solution was cooled to 0 °C followed by dropwise addition of TfOH (300 μL, 3.418 mmol), resulting in a coloured solution. The reaction mixture was stirred at room temperature for 2 h and concentrated under vacuum (while still cold for low-temperature reactions). Et₂O (10 mL) was added and the mixture was stirred at room temperature for 10 min to precipitate out an off-white solid. The solid was filtered off, washed with Et₂O and dried under vacuum.

Off-white solid (532.9 mg, 1.14 mmol, yield: 40%) m.p. 150-152 °C. ¹H NMR (250 MHz, [D₆]DMSO) δ 8.50 (dt, J = 8.8, 6.8 Hz, 1H), 8.23 (d, J = 7.6 Hz, 2H), 7.58 – 7.62 (m, 2H), 7.35 (td, J = 8.3, 1.9 Hz, 1H) ppm. ¹³C NMR (63 MHz, [D₆]DMSO) δ 165.37 (d, J = 322.2 Hz), 117.13, 115.29 (dd, J = 22.7, 3.0 Hz), 105.96 (t, J = 27.4 Hz), 99.10 (dd, J = 24.3, 3.9 Hz) ppm. IR (ATR), 3084, 3061, 1599, 1589, 1566, 1485, 1471, 1445, 1425, 1317, 1302, 1272, 1240, 1219, 1165, 1150, 1110, 1092, 1065, 1023, 989, 965, 932, 915, 857, 849, 831, 815, 759, 748, 735, 678 cm⁻¹. HRMS calcd for C₁₂H₈F₂I [M-OTf]^+ 316.9639 found 316.9648
m-Chloroperbenzoic acid (m-CPBA 70% active oxidant, 770 mg, 3.133 mmol), 2-trifluoromethyl-iodobenzene (400 μL, 2.848 mmol) and benzene (280 μL, 3.133 mmol) were dissolved in CH₂Cl₂ (10 mL) in a round bottom flask. The stirred solution was cooled to 0 °C followed by dropwise addition of TfOH (300 μL, 3.418 mmol), resulting in a coloured solution. The reaction mixture was stirred at room temperature for 2 h and concentrated under vacuum (while still cold for low-temperature reactions). Et₂O (10 mL) was added and the mixture was stirred at room temperature for 10 min to precipitate out an off-white solid. The solid was filtered off, washed with Et₂O and dried under vacuum.

Off-white solid 547.5 mg (1.10 mmol, yield: 39 %) m.p. 168-172 °C. ¹H NMR (250 MHz, [D₆]DMSO) δ 8.93 (d, J = 7.3 Hz, 1H), 8.20 (d, J = 7.8 Hz, 2H), 8.03 (d, J = 7.2 Hz, 1H), 7.89 (p, J = 7.4 Hz, 2H), 7.67 (t, J = 7.3 Hz, 1H), 7.54 (t, J = 7.6 Hz, 2H) ppm. ¹³C NMR (63 MHz, [D₆]DMSO) δ 140.09, 136.08, 134.84, 133.31, 132.36, 131.93, 129.57 (d, J = 31.6 Hz), 128.68 (q, J = 5.7 Hz), 122.78 (d, J = 274.4 Hz), 120.73 (d, J = 322.4 Hz), 117.30, 112.84 (d, J = 1.7 Hz) ppm. ¹⁹F NMR (235 MHz, [D₆]DMSO) δ -58.04, -77.78 ppm. IR (ATR), 3094, 1588, 1472, 1444, 1435, 1313, 1295, 1267, 1237, 1223, 1193, 1181, 1174, 1159, 1129, 1109, 1090, 1065, 1027 999, 987, 917, 899, 787, 760, 737, 724, 677, 673, 653, 637, 631 cm⁻¹.

3-Chloro-2-tolyI(phenyl)iodonium triflate (2ap)

m-Chloroperbenzoic acid (m-CPBA 70% active oxidant, 678 mg, 2.75 mmol), 2-chlor-6-iodotoluene (349 μL, 2.5 mmol) and benzene (246 μL, 2.75 mmol) were dissolved in CH₂Cl₂ (10 mL) in a round bottom flask. The stirred solution was cooled to 0 °C followed by dropwise addition of TfOH (263 μL, 3.0 mmol), resulting in a coloured solution. The reaction mixture was stirred at room temperature for 2 h and concentrated under vacuum (while still cold for low-temperature reactions). Et₂O (10 mL) was added and the mixture was stirred at room temperature for 10 min to precipitate out an off-white solid. The solid was filtered off, washed with Et₂O and dried under vacuum.

Off-white solid 423.4 mg (0.88 mmol, yield: 35 %) m.p. 172-178 °C. ¹H NMR (250 MHz, DMSO-d₆) δ 8.40 (d, J = 8.0 Hz, 1H), 8.25 (d, J = 7.7 Hz, 2H), 7.74 (d, J = 8.0 Hz, 1H), 7.66 (d, J = 7.3 Hz, 1H), 7.53 (t, J = 7.6 Hz, 2H), 7.37 (t, J = 8.0 Hz, 1H), 2.73 (s, 3H) ppm. ¹³C NMR (63 MHz, DMSO-d₆) δ 137.97, 136.39, 135.16, 133.75, 133.60, 132.26, 131.98, 130.84, 122.57, 120.72 (d, J = 322.3 Hz), 116.62, 23.77 ppm. IR (ATR), 3079, 3054, 1579, 1551, 1470, 1443, 1267, 1246, 1221, 1167, 1076, 1054, 1025, 988, 912, 803, 772, 759, 739, 721, 685, 678, 651, 636, 606, 571, 513, 493, 445, 426 cm⁻¹. HRMS calcd for C₁₃H₁₁ClI [M-OTf]+ 328.9594 found 328.9606
4-Iodophenyl(phenyl)iodonium triflate (2aq)\textsuperscript{18}

\textit{m}-Chloroperbenzoic acid (\textit{m}-CPBA 70\% active oxidant, 1.63 g, 6.6 mmol) and iodobenzene (1.48 µL, 13.2 mmol) were dissolved in CH\textsubscript{2}Cl\textsubscript{2} (20 mL) in a round bottom flask. The stirred solution was cooled to 0 °C followed by dropwise addition of TfOH (1.16 mL, 13.2 mmol), resulting in a coloured solution. The reaction mixture was stirred at 80 °C for 2 h and concentrated under vacuum (while still cold). Et\textsubscript{2}O (10 mL) was added and the mixture was stirred at room temperature for 10 min to precipitate out an off-white solid. The solid was filtered off, washed with Et\textsubscript{2}O and dried under vacuum \textsuperscript{2aq}.

White solid (1.1 g, 1.98 mmol, yield: 30\%) m.p. 136-139 °C, (Lit.: 138-140 °C). \textit{\textsuperscript{1}H NMR (250 MHz, [D\textsubscript{6}])DMSO} δ 8.24 (d, J = 7.7 Hz, 1H), 8.00 (d, J = 8.5 Hz, 1H), 7.89 (d, J = 8.5 Hz, 1H), 7.67 (t, J = 7.3 Hz, 1H), 7.53 (t, J = 7.7 Hz, 1H) ppm. \textit{\textsuperscript{13}C NMR (63 MHz, [D\textsubscript{6}])DMSO} δ 140.42, 136.80, 135.20, 132.17, 131.83, 116.71, 115.88, 100.29 ppm.

IR (ATR), 2047, 1960, 1469, 1380, 1290, 1240, 1169, 1026, 985, 805, 735, 728, 702 cm\textsuperscript{-1}.

Phenyl(thiophen-2-yl)iodonium tosylate (2ar)\textsuperscript{22}

\textit{m}-Chloroperbenzoic acid (\textit{m}-CPBA 70\% active oxidant, 247 mg, 1.0 mmol), iodobenzene (112 µL, 1.0 mmol), and thiophene (80 µL, 1.0 mmol) were dissolved in CH\textsubscript{2}Cl\textsubscript{2} (2 mL) and 2,2,2-trifluoroethanol (2 mL). Then, TsOH\times H\textsubscript{2}O (190 mg, 1.0 mmol) was added to the solution and the mixture was stirred at r.t. for 6 h and the solution was concentrated in vacuo. Et\textsubscript{2}O (5 mL) was added and the mixture was stirred at r.t. for 10 min to precipitate out an white off-solid. The precipitate was filtered off, washed with Et\textsubscript{2}O and dried under vacuum to give salt 2ar.

White solid (399 mg, 0.87 mmol, yield: 87\%) m.p. 143-146 °C. \textit{\textsuperscript{1}H NMR (250 MHz, CDCl\textsubscript{3})} δ 7.95 (d, J = 7.5 Hz, 1H), 7.79 (dd, J = 3.7, 1.1 Hz, 1H), 7.55 (d, J = 1.1 Hz, 1H), 7.46 (t, J = 7.6 Hz, 2H), 7.31 (t, J = 7.7 Hz, 1H), 7.11 – 6.92 (m, 2H), 2.30 (s, 1H) ppm. \textit{\textsuperscript{13}C NMR (63 MHz, CDCl\textsubscript{3})} δ 140.61, 136.02, 134.35, 131.61, 129.64, 128.68, 126.10, 21.42 ppm. IR (ATR), 3066, 1495, 1472, 1464, 1440, 1384, 1208, 1179, 1157, 1117, 1079, 1064, 1030, 1006, 991, 949, 876, 849, 815, 801, 748, 740, 734, 718, 712, 678, 652, 625, 617, 564 cm\textsuperscript{-1}.

4-Tolyl(2-thienyl)iodonium bromide (2as)\textsuperscript{24,26}

The corresponding 4-(diacetoxyiodo)toluenel i7 (672 mg, 2 mmol) and thiophene (480 µL, 6 mmol) were stirred in 10 ml acetic anhydride at -30 °C. Concentrated sulphuric acid (0.5 ml) was added dropwise over 1 h. The mixture was allowed to warm up to 5 °C and was further stirred at this temperature for 3 to 5 h. The dark solution was poured into 15 ml ice-water. The organic compounds were extracted with ether and discarded. The aqueous layers were treated
with activated coal for 10 min at 40 °C to become a clear solution. To the filtered, clear solution 10 ml of a potassium bromide solution (25 w/w%), respectively, was added. After 1h storage in the refrigerator, precipitates were collected, washed with a small portion of acetone and ether. The product 2as was dried and stored in an exsiccator. For eventual re-crystallisation the compounds were dissolved in hot methanol and precipitated with ether.

White solid (215 mg, 0.56 mmol, yield: 28%) m.p. 160-162 °C, (Lit.: 195 °C, 180-185 °C). ^1^H NMR (250 MHz, [D_6]DMSO) δ 8.07 (d, J = 8.2 Hz, 2H), 7.92 (d, J = 3.5 Hz, 1H), 7.87 (d, J = 5.1 Hz, 1H), 7.28 (d, J = 8.1 Hz, 2H), 7.16 – 7.06 (m, 1H), 2.32 (s, 3H) ppm. ^13^C NMR (63 MHz, [D_6]DMSO) δ 141.99, 139.03, 136.07, 134.57, 132.07, 129.30, 117.80, 105.86, 20.86 ppm. IR (ATR), 3395, 3077, 3054, 1632, 1581, 1572, 1484, 1456, 1439, 1385, 1295, 1247, 1221, 1210, 1178, 1116, 1022, 991, 821, 732, 720, 586, 510, 460 cm^-1.

2,4,6-trimethoxiphenyl(phenyl)iodonium tosylate (2at)^27

To a stirred solution of 1,3,5-trimethoxibenzene (168.2 mg, 1.0 mmol) in 2,2,2-trifluoroethanol (5 mL), hydroxi(tosyloxi)iodobenzene i10 (392 mg, 1.0 mmol) was added in one portion at room temperature under air, and it was stirred for 24 h. MeOH was then added to the reaction mixture when the solvents were removed under vacuum. The resulting oily crude product was precipitated by addition of Et_2O with stirring. The precipitate was filtered and dried in vacuo to give pure 2at as a powder.

Khaki solid (419.2 mg, 0.77 mmol, yield: 77%) m.p. 163-166 °C, (Lit.: 178-180 °C). ^1^H NMR (250 MHz, [D_6]DMSO) δ 7.91 (d, J = 7.3 Hz, 1H), 7.61 (t, J = 7.4 Hz, 1H), 7.54 – 7.36 (m, 2H), 7.10 (d, J = 7.8 Hz, 1H), 6.46 (s, 1H), 3.94 (s, 3H), 3.86 (s, 2H), 2.28 (s, 2H) ppm. ^13^C NMR (63 MHz, [D_6]DMSO) δ 166.18, 159.38, 145.78, 137.54, 134.30, 131.55, 128.01, 125.48, 116.05, 92.07, 57.32, 56.15, 20.75 ppm. IR (ATR), 3090, 3058, 3017, 2997, 2845, 1583, 1495, 1468, 1455, 1445, 1432, 1413, 1381, 1344, 1237, 1222, 1202, 1185, 1175, 1161, 1119, 1063, 1033, 1023, 1009, 993, 943, 911, 842, 834, 811 cm^-1.

Bis-(4-fluorophenyl)iodonium triflate (2au)^18,28

Iodine (609 mg, 2.4 mmol) was dissolved in CH_2Cl_2 (20 mL). Then m-chloroperoxybenzoic acid (m-CPBA 70% active oxidant, 2.367 g, 9.6 mmol) and fluorobenzene (2.252 mL, 24.0 mmol) was added to the solution and stirred at 0 °C. Then, HOTf (0.807 mL, 9.2 mmol) was added dropwise to the solution and the mixture was stirred at RT. for 12 h and the solution was concentrated in vacuo. Et_2O (20 mL) was added and the mixture was stirred at 10 min, to precipitate out an off-white solid. The precipitate was filtered off, washed with cold Et_2O (3 × 5 mL), and dried under vacuum to give salt 2au.

Off-white solid (794.3 mg, 1.70 mmol, yield: 71 %) m.p. 165-171 °C, (Lit. 101 °C (dec.)). ^1^H NMR (250 MHz, D_3COD) δ 8.31 – 8.14 (m, 1H), 7.37 – 7.21 (m, 1H) ppm. ^13^C NMR (63
MHz, D₃COD) δ 166.32 (d, J = 254.2 Hz), 139.18 (d, J = 9.2 Hz), 121.66 (d, J = 318.6 Hz), 120.52 (d, J = 23.3 Hz), 110.07 ppm. ¹³F NMR (235 MHz, CDCl₃) δ 8.09 – 7.97 (m, 1H), 7.13 (ddd, J = 12.5, 5.1, 2.8 Hz, 1H) ppm. IR (ATR), 3096, 3059, 2762, 1727, 1594, 1268, 1162 cm⁻¹.

Bis(4-chlorophenyl)iodonium triflate (2av)¹⁸

m-Chloroperbenzoic acid (m-CPBA 70% active oxidant, 3.70 g, 15 mmol), 4-chloro-1-iodobenzene (3.578g, 15 mmol), and chlorobenzene (1.368 mL, 13.5 mmol) were dissolved in CH₂Cl₂ (80 mL) stirred at 0 °C. Then, HOTf (3.547 mL, 40.2 mmol) was added dropwise to the solution and the mixture was stirred at rt for 19 h and the solution was concentrated in vacuo. Et₂O (10 mL) was added and the mixture was cooled at -20 °C, over night to precipitate out a white solid. The solid was filtered off, washed with cold Et₂O, and dried under vacuum to give salt 2av.

White solid (3.96 g, 7.93 mmol, yield: 59%) m.p. 185-186 °C, (Lit.: 181-183 °C). ¹H NMR (250 MHz, [D₆]DMSO) δ 8.26 (d, J = 8.7 Hz, 1H), 7.62 (d, J = 8.7 Hz, 1H) ppm. ¹³C NMR (63 MHz, [D₆]DMSO) δ 137.51, 137.01, 131.82, 114.70 ppm. IR (ATR), 3089, 1265, 1184, 1163, 1049, 1019 cm⁻¹.

Bis(4-bromophenyl)iodonium triflate (2aw)¹⁸

m-Chloroperbenzoic acid (m-CPBA 70% active oxidant, 542 mg, 2.2 mmol), 4-iodo-bromobenzene (566 mg, 2.0 mmol) and bromobenzene (232 μL, 2.2 mmol) were dissolved in CH₂Cl₂ (17 mL) in a round bottom flask. The stirred solution was cooled to 0 °C followed by dropwise addition of TFOH (527 μL, 6.0 mmol), resulting in a coloured solution. The reaction mixture was stirred at room temperature for 24 h and concentrated under vacuum (while still cold). Et₂O (7 mL) was added and the mixture was stirred at room temperature for 10 min to precipitate out as a solid, 2aw was filtered off, washed with Et₂O and dried under vacuum.

White solid (996.5 mg, 1.70 mmol, yield: 85%) m.p. 185-190 °C, (Lit.: 203 °C (dec.)). ¹H NMR (250 MHz, [D₆]DMSO) δ 8.17 (d, J = 8.6 Hz, 1H), 7.76 (d, J = 8.6 Hz, 1H) ppm. ¹³C NMR (63 MHz, [D₆]DMSO) δ 137.05, 134.68, 126.34, 115.28 ppm. IR (ATR), 3079, 1472, 1271, 1184, 1165, 1021 cm⁻¹.

Mesityl(2,4,6-trimethoxyphenyl)iodonium tosilate (2ax)

To a stirred solution of 1,3,5-trimethoxibenzene (336.4 mg, 2.0 mmol) in 2,2,2-trifluoroethanol (10 mL), HTIM i9 (868.6 mg, 2.0 mmol) was added in one portion at room
temperature under air, and it was stirred for 24 h. MeOH was then added to the reaction mixture when the solvents were removed under vacuum. The resulting oily crude product was precipitated by addition of Et₂O with stirring. The precipitate was filtered and dried in vacuo to give pure 2a as a powder.

Grey solid (1.033 g, 1.77 mmol, yield: 88%) m.p. 160-162 °C. ¹H NMR (250 MHz, D₃COD) δ 7.67 (d, J = 8.1 Hz, 2H), 7.20 (d, J = 7.9 Hz, 2H), 7.10 (s, 2H), 6.39 (s, 2H), 3.91 (s, 6H), 3.87 (s, 3H), 2.62 (s, 6H), 2.35 (s, 3H), 2.30 (s, 3H) ppm. ¹³C NMR (63 MHz, D₃COD) δ 168.39, 161.57, 144.59, 143.62, 141.56, 130.70, 129.76, 126.94, 121.58, 93.05, 83.41, 57.46, 56.65, 26.54, 21.30, 20.90 ppm. IR (ATR), 2920, 2844, 1578, 1464, 1450, 1431, 1412, 1380, 1348, 1298, 1232, 1174, 1118, 1068, 1030, 1008, 991, 947, 911, 841, 808, 643. HRMS calcd for C₁₈H₂₂IO₃ [M + OTs]⁺ 413.0614 found 413.0624.

**Mesityl(3-pyridinium)iodonium bistriflate (2ay)**

To a solution of 3-iodopyridine (500 mg, 2.44 mmol) in CH₂Cl₂ (10 mL) was added TfOH (850 μL, 9.7 mmol, 4 equiv) and the resulting mixture was stirred at rt for 5 min. m-chloroperbenzoic acid (m-CPBA 70% active oxidant, 887.5 mg, 3.6 mmol, 1.5 equiv) followed by the mesitylene (370 μL, 2.64 mmol, 1.1 equiv) was then added. The reaction vessel was sealed and submitted to a 60 °C oilbath with stirring for 30 min. The reaction mixture was then allowed to reach rt after which it was concentrated in vacuo. Et₂O (10 mL) was added and the mixture was stirred at 0 °C for 30 min. The resulting precipitate was filtered through a glass-sintered funnel and washed with additional Et₂O (3 × 10 mL) to give the protonated N-heteroaryliodonium bistriflate 2ay.

Drabb solid (1.3281 g, 2.13 mmol, yield: 87%) m.p. 138-141 °C, (Lit.: 153-155 °C). ¹H NMR (250 MHz, D₃COD) δ 9.11 (s, 1H), 8.84 (d, J = 5.1 Hz, 1H), 8.56 (d, J = 8.4 Hz, 1H), 7.79 (dd, J = 8.3, 5.4 Hz, 1H), 7.19 (s, 2H), 2.59 (s, 6H), 2.28 (s, 3H) ppm. ¹³C NMR (63 MHz, D₃COD) δ 149.79, 149.12, 148.16, 146.71, 143.85, 131.69, 129.95, 124.26, 122.40, 121.72 (q, J = 318.6 Hz), 119.19, 113.25, 27.14, 21.06 ppm. IR (ATR), 3205, 3096, 3052, 3027, 2899, 2856, 1622, 1595, 1527, 1459, 1280, 1230, 1182, 1161, 1110, 1021, 987, 944, 892, 850, 799, 758, 671, 624, 582, 572, 542, 513, 452 cm⁻¹.

**Mesityl(2,5-dimethyl-3-pyrazolium)iodonium bistriflate (2az)**

To a solution of 4-iodo-3,5-dimethylpyrazole (444 mg, 2.0 mmol) in CH₂Cl₂ (20 mL) was added TfOH (702 μL, 8.0 mmol, 4 equiv) and the resulting mixture was stirred at rt for 5 min. m-chloroperbenzoic acid (70% active oxidant, 863 mg, 3.5 mmol, 1.75 equiv) and mesitylene (336 μL, 2.4 mmol, 1.2 equiv) was then added. The reaction vessel was sealed and submitted...
to a 60 °C oilbath with stirring for 30 min after which it was cooled down to RT after which it was concentrated in vacuo. Et₂O (1-3 mL) was added and the mixture was cooled to 0 °C and stirred for 30 min. The resulting precipitate was filtered through a glass sintered funnel and washed with additional Et₂O (3 × 3 mL) to give the protonated N-heteroaryliodonium bistriflate 2az.

Slightly brown solid (1.1057 g 1.56 mmol, yield: 86%) m.p. 142-145 °C, (Lit.: 148-152 °C, 169-171°C). ¹H NMR (250 MHz, D₂COD) δ 7.06 (s, 2H), 2.50 (s, 6H), 2.23 (s, 6H), 2.20 (s, 3H) ppm. ¹³C NMR (63 MHz, D₂COD) δ 149.99, 145.41, 142.94, 131.38, 121.76 (d, J = 318.5 Hz), 121.31, 114.18, 81.59, 66.86, 49.00, 26.68, 20.88, 15.41, 12.24 ppm. IR (ATR), 2926, 2700, 1589, 1452, 1291, 1267, 1216, 1158, 1020, 983, 942, 851, 811, 756, 749, 631, 574, 551 cm⁻¹.

Diphenyliodonium triflate (2ba)²¹

Iodine (1.218 g, 4.8 mmol) was dissolved in CH₂Cl₂ (40 mL). Then m-chloroperbenzoic acid (m-CPBA 65% active oxidant, 3.072 g, 17.8 mmol) and benzene (1.76 mL, 19.68 mmol) was added to the solution and stirred at 0 °C. Then, HOTf (1.64 mL, 26.7 mmol) was added dropwise to the solution and the mixture was stirred at 80 °C. for 10 min. and the solution was cooled to room temperature and concentrated in vacuo. Et₂O (40 mL) was added and the mixture was stirred at 10 min, to precipitate out an off-white solid. The precipitate was filtered off, washed with cold Et₂O (3×10 mL), and dried under vacuum to give salt 2ba.

Off-white solid (1.531 g, 3.58 mmol, yield: 75%) m.p. 170-176 °C, (Lit.: 172-174 °C, 176-177, 177-178, 177-180). ¹H NMR (250 MHz, [D₆]DMSO) δ 8.25 (d, J = 7.5 Hz, 1H), 7.67 (t, J = 7.4 Hz, 1H), 7.53 (t, J = 7.6 Hz, 1H) ppm. ¹³C NMR (63 MHz, [D₆]DMSO) δ 135.20, 132.09, 131.79, 116.53 ppm. IR (ATR), 3086, 3063, 1564, 1471, 1443, 1267, 1243, 1219, 1168, 1023, 987, 912, 758, 743, 728, 676, 649, 633, 573, 514, 462, 436 cm⁻¹.

Diphenyliodonium tetrafluoroborate (2bb)¹⁹

m-Chloroperbenzoic acid (m-CPBA 70% active oxidant, 640 mg, 3.0 mmol) was dissolved in CH₂Cl₂ (10 mL). To the solution was added iodobenzene (310 μL, 2.7 mmol) followed by slow addition of BF₃·OEt₂ (850 μL, 6.8 mmol) at room temperature. The resulting yellow solution was stirred at rt for 30 min and then cooled to 0 °C, and phenylboronic acid (366 mg, 3.0 mmol) was added. After 15 min of stirring at rt, triflic acid (240 μL, 2.75 mmol) was added at room temperature, and the mixture was stirred for an additional 15 min. The crude reaction mixture was applied on a silica plug (6.0 g) and eluted with CH₂Cl₂ (60 mL) followed by CH₂Cl₂/MeOH (120 mL, 20:1). The latter solution was concentrated, and diethyl ether (10 mL) was added to the residue to induce a precipitation. The solution was allowed to stir for 15 min, and then the ether phase was decanted. The solid 2bb was washed twice more with diethyl ether (2 × 10 mL) and then dried in vacuo.
Off-white solid (544 mg, 1.48 mmol, 55% yield) m.p. 134-145 °C, (Lit.: 132-134 °C, 136-137 °C). *R*<sub>g</sub> = 0.16 (DCM-MeOH, 10:1). <sup>1</sup>H NMR (250 MHz, <sup>[D]_6</sup>DMSO) δ 8.26 (d, *J* = 8.0 Hz, 1H), 7.67 (t, *J* = 7.0 Hz, 1H), 7.54 (t, *J* = 7.6 Hz, 1H) ppm. <sup>13</sup>C NMR (63 MHz, <sup>[D]_6</sup>DMSO) δ 135.17, 132.07, 131.78, 116.50 ppm.

IR (ATR), 1653, 1606, 1590, 1562, 1511, 1411, 1328, 1307, 1286, 1223, 1201, 1180, 1114, 1027, 1014, 993, 955, 896, 851, 827, 810, 791, 732, 711, 678, 666, 641, 635, 601, 535, 508, 491 cm<sup>-1</sup>.

**Diphenyliodonium bromide (2bc)<sup>29</sup>**

Powdered I<sub>2</sub> (1.442 g, 5.68 mmol) and NaIO<sub>4</sub> (1.839 g, 8.6 mmol) were suspended in stirred cc. H<sub>2</sub>SO<sub>4</sub> (20 mL), and the vigorous stirring was continued at 70-75 °C for 1 h. Next Ac<sub>2</sub>O (12 mL) was slowly added dropwise, with stirring, to the cooled suspension of yellow (IO)<sub>2</sub>SO<sub>4</sub> (ca. 10 mmol). Benzene (4.65 mL, 52 mmol) was added dropwise, with stirring and the stirring was continued firstly at rt for 2 h, and next at 50-55 °C for 1 h. The final mixtures were poured into stirred ice-water (600 g), and any precipitates were filtered off and rejected. The cold filtrates were extracted with Et<sub>2</sub>O (4 × 100 mL) and the ethereal extracts (containing the unreacted initial arenes) were discarded. A solution of KBr (4.0 g, 33.6 mmol) in H<sub>2</sub>O (20 mL) was added to the vigorously stirred remaining aquareous solution. After ca. 1 h, the precipitated diphenylonium bromide were collected by filtration and were washed well with cold H<sub>2</sub>O until the filtrates were neutral, dried preliminarily by the suction, and air-dried in the dark to give the crude product 2bc. (Small samples of the crude bromide were quickly recrystallized from EtOH, to give the purified analytical sample.)

Drabb solid (3.160 g, 8.75 mmol, yield: 77%) m.p. 160-164 °C, (Lit.: 228-229 (EtOH), 208-209, 210, 234-235, 246-247 °C). <sup>1</sup>H NMR (250 MHz, <sup>[D]_6</sup>DMSO) δ 8.21 (m, 4H), 7.50 (m, 6H) ppm. <sup>13</sup>C NMR (63 MHz, <sup>[D]_6</sup>DMSO) δ 134.92, 131.37, 119.78 ppm. IR (ATR), 3044, 1562, 1468, 1437, 1368, 1322, 1173, 1157, 1099, 1041, 1010, 985, 907, 853, 834, 807, 734, 677, 646, 608, 577, 475, 459, 442 cm<sup>-1</sup>.

**Diphenylidonium trifluoroacetate (2bd)<sup>30</sup>**

A solution of an iodobenzene (225 µL, 2.01 mmol) in a mixture of benzene (539 µL, 6.03 mmol), CF<sub>3</sub>COOH (18 mL) and CH<sub>2</sub>Cl<sub>2</sub> (4 mL) was heated with stirring to 36-38 °C. Next, potassium peroxodisulfate (2.173 g, 8.04 mmol) was added portcionwise during 10 min and the stirring was continued until TLC analysis indicated completion of reaction. Reaction time needed 28 h. After completion of the reaction, water (20 mL) was added. The precipitates were collected by filtration under reduced pressure, washed with CH<sub>2</sub>Cl<sub>2</sub> (20 mL), and discarded. The crude product 2bd was obtained by extraction of the filtrate with dichloromethane (3 × 20 mL) followed by drying (anhydrous Na<sub>2</sub>SO<sub>4</sub>), filtration, and removal of the solvent by evaporation under reduced pressure.
Off-white solid (124 mg, 0.03 mmol, yield: 1.6%) $^1$H NMR (250 MHz, CDCl$_3$) δ 7.94 (d, $J = 7.0$ Hz, 1H), 7.54 (d, $J = 6.6$ Hz, 1H), 7.42 (d, $J = 6.9$ Hz, 1H) ppm.

**General procedure for the one-pot synthesis of aryl-mesityliodonium triflates**

Aryl-mesityliodonium triflates were synthesised in a one-pot procedure from the appropriate iodoarene and mesitylene according to the general method of Olofsson.$^{18}$

$m$-Chloroperbenzoic acid (65% active oxidant, 1.320 g, 5.0 mmol, 1.1 equiv.) and the appropriate iodoarene (4.5 mmol, 1.0 equiv.) were dissolved in CH$_2$Cl$_2$ (20 ml). Mesitylene (696 μl, 5.0 mmol, 1.1 equiv.) was added and the solution was cooled to 0 °C. Trifluoromethanesulfonic acid (825 μl, 5.5 mmol, 1.2 equiv.) was added dropwise over 5 minutes and the reaction mixture was allowed to warm to room temperature over 2h. The volatile components were removed in vacuo/ evaporated under reduced pressure and the resulting material was suspended in Et$_2$O (20 ml). The suspension was stored at -20 °C for 2h. The obtained crystals were filtered under reduced pressure and were washed on the filter with Et$_2$O (3×10 ml) to give the appropriate aryl-mesityliodonium triflate as a solid, which was dried at 100 °C under vacuum.

2-**Tolyl**(mesityl)iodonium trifluoromethanesulfonate (2b)$^{31}$

Prepared according to the general procedure from 2-iodotoluene. The product 2b was obtained as a white solid.

White solid (2.023 g, 4.16 mmol, 93%). m.p. 166-167 °C; $^1$H NMR (250 MHz, [D$_6$]DMSO): δ 7.97 (d, 1H, $J = 8.0$ Hz), 7.55 (d, 2H, $J = 4.3$ Hz), 7.26 (dd, 1H, $J = 8.3$ Hz and 4.1 Hz), 7.21 (s, 2H), 2.57 (s, 9H), 2.29 (s, 3H); $^{13}$C NMR (62.5 MHz, [D$_6$]DMSO): δ 143.0, 141.7, 140.8, 136.8, 132.5, 131.9, 130.0, 129.4, 121.9, 118.6, 26.2, 24.4, 20.5; IR $\nu_{\text{max}}$/cm$^{-1}$ (solid): 1467, 1245, 1156, 1027, 759, 634, 516.

2-**Fluorophenyl**(mesityl)iodonium trifluoromethanesulfonate (2c)$^{31}$

Prepared according to the general procedure from 2-fluoroiodobenzene. The product 2c was obtained as an off-white solid.

White solid (0.948 g, 1.93 mmol, 43%). m.p. 161-162 °C; $^1$H NMR (250 MHz, [D$_6$]DMSO): δ 8.28 (ddd, 1H, $J = 7.7$ Hz and 6.1 Hz and 1.5 Hz), 7.72 (tdd, 1H, $J = 7.2$ Hz and 5.7 Hz and 1.5 Hz), 7.56 (td, 1H, $J = 8.7$ Hz and 1.3 Hz), 7.35 (td, 1H, $J = 8.0$ Hz and 1.3 Hz), 7.20 (s, 2H), 2.62 (s, 6H), 2.27 (s, 3H); $^{13}$C NMR (62.5 MHz, [D$_6$]DMSO): δ 159.57 (d, $J = 248.7$ Hz), 143.3, 141.6, 137.5, 135.43 (d, $J = 8.3$ Hz), 129.9, 127.6, 122.8, 117.18 (d, $J = 22.4$ Hz), 101.55 (d, $J = 23.6$ Hz), 26.1 (d, $J = 1.9$ Hz), 20.5; IR $\nu_{\text{max}}$/cm$^{-1}$ (solid): 1476, 1279, 1236, 1161, 1027, 770, 635, 515.
2-Chlorophenyl(mesityl)iodonium trifluoromethanesulfonate (2d)$^{31}$

Prepared according to the general procedure from 1-chloro-2-iodobenzene. The product 2d was obtained as an off-white solid.
White solid (1.411 g, 2.78 mmol, 62%). m.p. 167-168 °C; $^1$H NMR (250 MHz, [D$_6$]DMSO): $\delta$ 8.27 (dd, 1H, $J$ = 8.0 Hz and 1.2 Hz), 7.82 (dd, 1H, $J$ = 8.0 Hz and 1.4 Hz), 7.68 (td, 1H, $J$ = 7.9 Hz and 1.3 Hz), 7.45 (td, 1H, $J$ = 8.0 Hz and 1.4 Hz), 7.21 (s, 2H), 2.62 (s, 6H), 2.28 (s, 3H); $^{13}$C NMR (62.5 MHz, [D$_6$]DMSO): $\delta$ 143.3, 141.9, 139.0, 135.8, 134.4, 130.8, 130.2, 130.0, 122.7, 116.5, 26.2, 20.5; IR $\nu_{\max}$/cm$^{-1}$ (solid): 1449, 1276, 1239, 1160, 1024, 759, 631, 516, 432.

2-Bromophenyl(mesityl)iodonium trifluoromethanesulfonate (2e)$^{31}$

Prepared according to the general procedure from 2-bromiodobenzene. The product 2e was obtained as an off-white solid.
White solid (1.645 g, 2.98 mmol, 66%). m.p. 167-168 °C; $^1$H NMR (250 MHz, [D$_6$]DMSO): $\delta$ 8.18 (dd, 1H, $J$ = 7.9 Hz and 1.4 Hz), 7.95 (dd, 1H, $J$ = 7.9 Hz and 1.4 Hz), 7.59 (td, 1H, $J$ = 7.7 Hz and 1.5 Hz), 7.47 (td, 1H, $J$ = 7.7 Hz and 1.4 Hz), 7.22 (s, 2H), 2.62 (s, 6H), 2.29 (s, 3H); $^{13}$C NMR (62.5 MHz, [D$_6$]DMSO): $\delta$ 143.3, 141.9, 139.1, 134.3, 134.2, 130.5, 130.1, 126.6, 123.0, 119.5, 26.4, 20.5; IR $\nu_{\max}$/cm$^{-1}$ (solid): 1442, 1276, 1241, 1160, 1025, 757, 631, 516.

1-Naphthalenyl(mesityl)iodonium trifluoromethanesulfonate (2g)$^{32}$

Prepared according to the general procedure from 1-iodonaphthalene. The product 2g was obtained as an off-white solid.
Off-white solid (0.604 g, 1.16 mmol, 25.8%). m.p. 155-161 °C (dec.). $^1$H NMR (250 MHz, [D$_6$]DMSO): $\delta$ 8.43 – 8.29 (d, $J$ = 7.5 Hz, 1H), 8.25 (d, $J$ = 8.1 Hz, 1H), 8.20 (d, $J$ = 8.4 Hz, 1H), 8.06 (d, $J$ = 7.5 Hz, 1H), 7.84 (td, $J$ = 7.0, 1.4 Hz, 1H), 7.72 (t, $J$ = 7.2 Hz, 1H), 7.53 (d, $J$ = 15.6 Hz, 1H), 7.15 (s, 2H), 3.38 (s, 3H), 2.59 (s, 6H) ppm. $^{13}$C NMR (62.5 MHz, [D$_6$]DMSO): $\delta$ 142.8, 141.5, 136.9, 134.2, 133.1, 131.2, 129.9, 129.4, 128.8, 127.9, 127.8, 123.4, 117.4, 26.5, 20.4 ppm. IR $\nu_{\max}$/cm$^{-1}$ 1158, 1506, 1290, 1243, 1167, 1028, 951, 933, 854, 790.
4-Acetylphenyl(mesityl)iodonium trifluoromethanesulfonate (2h)\textsuperscript{31}

Prepared according to the general procedure from 1-acetyl-4-iodobenzene. The product 2h was obtained as an off-white solid.

White solid (0.689 g, 1.34 mmol, 30%). m.p. 183-185 °C; \( ^1\)H NMR (250 MHz, [D\textsubscript{6}]DMSO): 8.09 (d, 2H, \( J = 8.2 \) Hz), 7.98 (d, 2H, \( J = 8.3 \) Hz), 7.24 (s, 2H), 2.60 (s, 6H), 2.58 (s, 3H), 2.30 (s, 3H); \( ^{13}\)C NMR (62.5 MHz, [D\textsubscript{6}]DMSO): \( \delta \) 197.2, 143.4, 141.7, 138.9, 134.6, 131.0, 129.9, 122.7, 119.2, 26.8, 26.3, 20.5; IR \( \nu_{\text{max}}/\text{cm}^{-1} \) (solid): 1693, 1577, 1391, 1339, 1236, 1170, 1022, 825, 631, 516.

4-Nitrophenyl(mesityl)iodonium trifluoromethanesulfonate (2i)\textsuperscript{31}

Prepared according to the general procedure from 4-iodonitrobenzene with the modification that all the reagents except the trifluoromethanesulfonic acid were stirred together at rt for 24h before the addition of the acid. The product 2i was obtained as a white solid.

White solid (0.919 g, 1.78 mmol, 40%). m.p. 207-208 °C; \( ^1\)H NMR (250 MHz, [D\textsubscript{6}]DMSO): \( \delta \) 8.26 (d, 2H, \( J = 9.1 \) Hz), 8.18 (d, 2H, \( J = 9.1 \) Hz), 7.25 (s, 2H), 2.59 (s, 6H), 2.31 (s, 3H); \( ^{13}\)C NMR (62.5 MHz, [D\textsubscript{6}]DMSO): \( \delta \) 149.3, 143.6, 141.8, 135.6, 130.0, 126.3, 122.8, 120.8, 26.3, 20.6; IR \( \nu_{\text{max}}/\text{cm}^{-1} \) (solid): 1534, 1353, 1242, 1157, 1024, 846, 735, 632, 574, 519, 454.

4-Anisyl(mesityl)iodonium trifluoromethanesulfonate (2be)\textsuperscript{33}

Prepared according to the general procedure from 1-acetyl-4-iodobenzene. The product 2be was obtained as an off-white solid.

Off-white solid (0.513 g, 1.02 mmol, 22.7%). m.p. 148-151 °C, (Lit.: 160-162 °C). \( ^1\)H NMR (250 MHz, [D\textsubscript{6}]DMSO): \( \delta \) 7.69 (dt, \( J = 8.2, 1.4 \) Hz, 1H) 7.17 (s, 2H), 7.03 (d, \( J = 9.0 \) Hz, 2H), 3.78 (s, 3H), 2.60 (s, 6H), 2.27 (s, 3H) ppm. \( ^{13}\)C NMR (62.5 MHz, [D\textsubscript{6}]DMSO): \( \delta \) 166.1, 141.3, 133.3, 132.9, 132.7, 130.6, 128.8, 127.9, 117.5, 55.7, 26.2, 20.5 ppm. IR \( \nu_{\text{max}}/\text{cm}^{-1} \): 1735, 1702, 1576, 1485, 1435, 1413, 1294, 1244, 1172, 1074, 1031, 903, 850, 671.
Prepared according to the general procedure from ethyl-4-iodobenzoate with the exception that 3-chloroperoxybenzoic acid, mesitylene and the aryl iodide were stirred together at room temperature for 4 hours before the addition of the trifluoromethanesulfonic acid. The product 

2bf was obtained as a white solid.

White solid (1.159 g, 2.13 mmol, 47%). m.p. 174-175 °C; $^1$H NMR (250 MHz, [D$_6$]DMSO): δ 8.09 (d, 2H, $J = 8.6$ Hz), 7.99 (d, 2H, $J = 8.5$ Hz), 7.24 (s, 2H), 4.31 (q, 2H, $J = 7.1$ Hz), 2.59 (s, 6H), 2.30 (s, 3H), 1.30 (t, 3H, $J = 7.1$ Hz); $^{13}$C NMR (62.5 MHz, [D$_6$]DMSO): δ 164.5, 143.4, 141.7, 134.7, 132.7, 131.9, 129.9, 122.6, 119.3, 61.4, 26.3, 20.5, 14.0; IR $\nu_{max}$/cm$^{-1}$ (solid): 1723, 1584, 1458, 1395, 1272, 1238, 1161, 1103, 1025, 849, 753, 634, 516.

4-Tolyl(mesityl)iodonium trifluoromethanesulfonate (2bg)$^{31}$

Prepared according to the general procedure from 4-iodotoluene. The product 2bg was obtained as a white solid.

White solid (2.072 g, 4.26 mmol, 95%). m.p. 183-184 °C; $^1$H NMR (250 MHz, [D$_6$]DMSO): δ 7.87 (d, 2H, $J = 8.2$ Hz), 7.31 (d, 2H, $J = 8.1$ Hz), 7.20 (s, 2H), 2.60 (s, 6H), 2.32 (s, 3H), 2.28 (s, 3H); $^{13}$C NMR (62.5 MHz, [D$_6$]DMSO): δ 143.0, 142.3, 141.5, 134.5, 132.5, 129.7, 122.7, 110.9, 26.3, 20.8, 20.5; IR $\nu_{max}$/cm$^{-1}$ (solid): 1452, 1246, 1157, 1024, 804, 632, 481.

4-Fluorophenyl(mesityl)iodonium trifluoromethanesulfonate (2bh)$^{31}$

Prepared according to the general procedure from 4-fluoroiodobenzene. The product 2bh was obtained as an off-white solid.

White solid (1.133 g, 2.31 mmol, 51%). m.p. 178-179 °C; $^1$H NMR (250 MHz, [D$_6$]DMSO): δ 8.06 (dd, 2H, $J = 8.0$ Hz and 5.1 Hz), 7.37 (t, 2H, $J = 8.6$ Hz), 7.22 (s, 2H), 2.60 (s, 6H), 2.29 (s, 3H); $^{13}$C NMR (62.5 MHz, [D$_6$]DMSO): δ 161.8, 143.2, 141.6, 137.40 (d, $J = 8.8$ Hz), 129.9, 123.0, 119.30 (d, $J = 22.8$ Hz), 108.69 (d, $J = 3.0$ Hz), 26.3, 20.6; IR $\nu_{max}$/cm$^{-1}$ (solid): 1575, 1483, 1224, 1168, 1024, 849, 632, 508.
4-Chlorophenyl(mesityl)iodonium trifluoromethanesulfonate (2bi)
Prepared according to the general procedure from 1-chloro-4-iodobenzene. The product 2bi was obtained as a white solid.
White solid (1.367 g, 2.70 mmol, 60%). m.p. 177-178 °C; \(^1\)H NMR (250 MHz, [D\(_6\)]DMSO): \(\delta\) 7.98 (d, 2H, \(J = 8.7\) Hz), 7.57 (d, 2H, \(J = 8.7\) Hz), 7.23 (s, 2H), 2.59 (s, 6H), 2.29 (s, 3H); \(^{13}\)C NMR (62.5 MHz, [D\(_6\)]DMSO): \(\delta\) 143.3, 141.6, 137.0, 136.3, 131.8, 129.9, 122.8, 112.3, 26.3, 20.5; IR \(\nu_{\text{max}}/\text{cm}^{-1}\) (solid): 1473, 1245, 1163, 1024, 807, 631, 516.

4-Bromophenyl(mesityl)iodonium trifluoromethanesulfonate (2bj)
Prepared according to the general procedure from 4-bromiodobenzene. The product 2bj was obtained as a white solid.
White solid (1.690 g, 3.07 mmol, 68%). m.p. 179-180 °C; \(^1\)H NMR (250 MHz, [D\(_6\)]DMSO): \(\delta\) 7.90 (d, 2H, \(J = 8.5\) Hz), 7.70 (d, 2H, \(J = 8.5\) Hz), 7.22 (s, 2H), 2.59 (s, 6H), 2.29 (s, 3H); \(^{13}\)C NMR (62.5 MHz, [D\(_6\)]DMSO): \(\delta\) 143.3, 141.6, 136.4, 134.7, 129.9, 125.8, 122.8, 113.1, 26.3, 20.6; IR \(\nu_{\text{max}}/\text{cm}^{-1}\) (solid): 1473, 1245, 1232, 1024, 807, 631, 518, 475.

3-Tolyl(mesityl)iodonium trifluoromethanesulfonate (2bk)
Prepared according to the general procedure from 3-iodotoluene. The product 2bk was obtained as a white solid.
White solid (1.587 g, 3.26 mmol, 73%). m. p. 171-172 °C; \(^1\)H NMR (250 MHz, [D\(_6\)]DMSO): \(\delta\) 7.86 (s, 1H), 7.77 (d, 1H, \(J = 7.7\) Hz), 7.45 (d, 1H, \(J = 7.5\) Hz), 7.38 (t, 1H, \(J = 7.7\) Hz), 7.21 (s, 2H), 2.61 (s, 6H), 2.32 (s, 3H), 2.29 (s, 3H); \(^{13}\)C NMR (62.5 MHz, [D\(_6\)]DMSO): \(\delta\) 143.1, 142.0, 141.6, 134.6, 132.5, 131.7, 131.6, 129.8, 122.4, 114.3, 26.3, 20.7, 20.5; IR \(\nu_{\text{max}}/\text{cm}^{-1}\) (solid): 1455, 1275, 1245, 1156, 1025, 634, 516.
3-Bromophenyl(mesityl)iodonium trifluoromethanesulfonate (2bl)\textsuperscript{31}

Prepared according to the general procedure from 3-bromoiodobenzene. The product 2bl was obtained as an off-white solid.

White solid (1.353 g, 2.45 mmol, 55%). m.p. 173-174 °C; \textsuperscript{1}H NMR (250 MHz, [D\textsubscript{6}]DMSO): \(\delta 8.29\) (s, 1H), 7.90 (d, 1H, \textit{J} = 8.0 Hz), 7.84 (d, 1H, \textit{J} = 8.1 Hz), 7.44 (t, 1H, \textit{J} = 8.0 Hz), 7.23 (s, 2H), 2.60 (s, 6H), 2.30 (s, 3H); \textsuperscript{13}C NMR (62.5 MHz, [D\textsubscript{6}]DMSO): \(\delta 143.4, 141.8, 136.2, 134.8, 133.6, 133.2, 129.9, 123.5, 122.7, 115.0, 26.4, 20.6\); IR \(\nu_{\text{max}}/\text{cm}^{-1}\) (solid): 1454, 1222, 1024, 797, 634, 516.

2-Trifluoromethylphenyl(mesityl)iodonium trifluoromethanesulfonate (2bm)\textsuperscript{31}

Prepared according to the general procedure from 1-iodo-2-(trifluoromethyl)benzene. The product 2bm was obtained as an off-white solid.

White solid (1.113 g, 2.06 mmol, 46%). m.p. 180-181 °C; \textsuperscript{1}H NMR (250 MHz, [D\textsubscript{6}]DMSO): \(\delta 8.50\) (d, 1H, \textit{J} = 7.8 Hz), 8.05 (d, 1H, \textit{J} = 7.6 Hz), 7.91 (t, 1H, \textit{J} = 7.6 Hz), 7.80 (t, 1H, \textit{J} = 7.5 Hz), 7.23 (s, 2H), 2.57 (s, 6H), 2.30 (s, 3H); \textsuperscript{13}C NMR (62.5 MHz, [D\textsubscript{6}]DMSO): \(\delta 143.5, 142.1, 140.4, 135.8, 133.2, 130.2, 129.4\) (m), 124.8, 123.2, 109.7, 26.1, 20.5; IR \(\nu_{\text{max}}/\text{cm}^{-1}\) (solid): 1588, 1431, 1309, 1244, 1164, 1132, 1024, 773, 632, 516.

2-(Ethoxycarbonyl)phenyl(mesityl)iodonium trifluoromethanesulfonate (2bn)\textsuperscript{31}

Prepared according to the general procedure from ethyl-2-iodobenzoate with the exception that 3-chloroperoxybenzoic acid, mesitylene and the aryl iodide were stirred together at room temperature for 4 hours before the addition of the trifluoromethanesulfonic acid. The product 2bn was obtained as a white solid.

White solid (0.835 g, 1.53 mmol, 34%). m.p. 169-170 °C; \textsuperscript{1}H NMR (250 MHz, [D\textsubscript{6}]DMSO): \(\delta 8.34\) (dd, 1H, \textit{J} = 7.2 Hz and 1.9 Hz), 7.88 – 7.68 (m, 2H), 7.41 (s, 2H), 6.89 (d, 1H, \textit{J} = 7.5 Hz), 4.53 (q, 2H, \textit{J} = 7.1 HzH), 2.52 (s, 6H), 2.43 (s, 3H), 1.43 (t, 3H, \textit{J} = 7.1 Hz); \textsuperscript{13}C NMR (62.5 MHz, [D\textsubscript{6}]DMSO): \(\delta 167.4, 144.6, 143.3, 137.3, 132.9, 131.4, 130.1, 128.5, 127.6, 117.8, 113.5, 63.8, 26.1, 20.7, 13.9\); IR \(\nu_{\text{max}}/\text{cm}^{-1}\) (solid): 1673, 1310, 1272, 1246, 1222, 1154, 1027, 753, 637, 515.
Preparation and Analytical Data of N-arylpyrazoles

General procedure for the synthesis of N-arylpyrazoles

The appropriate 1H-pyrazole (0.5 mmol, 1.0 equiv), diaryliodonium salt (0.55 mmol, 1.1 equiv), were placed in a 30 ml vial and dissolved in 25w/w% NH₃ (aq) solution-toluene 1:1 (20 mL) stirred at RT for the indicated time. The reaction mixture was diluted with CH₂Cl₂ (10 mL). The aqueous layer was extracted with CH₂Cl₂ (2 × 10 mL) and all combined organic phase were dried over magnesium sulphate. The suspension was then filtered, concentrated in vacuo and purified by flash chromatography on silicagel, if not noted otherwise.

1-Mesityl-3,5-diphenyl-pirazole (3a)

The general procedure was followed. 3,5-diphenyl-1H-pyrazole 1a (220 mg, 1 mmol), mesityl(phenyl)iodonium triflate 2a (472 mg, 1 mmol), were dissolved in dichloroethane-25w/w% NH₃ (aq) solution-toluene 1:1 (40 mL) stirred at RT for 20 min. The reaction mixture was diluted with CH₂Cl₂ (20 mL). The aqueous layer was extracted with CH₂Cl₂ (2 × 20 mL) and all combined organic phase were dried over magnesium sulphate. The suspension was then filtered, concentrated in vacuo and purified by flash chromatography on silicagel. Colorless oil (320.6 mg, 0.948 mmol, yield: 95%). Rf.: 0.46 (in hexane:EtOAc 10:1). ¹H NMR (250 MHz, CDCl₃) δ 8.03 – 7.95 (m, 2H), 7.47 (t, J = 7.3 Hz, 2H), 7.38 (d, J = 7.2 Hz, 1H), 7.29 (s, 5H), 6.97 (s, 3H), 2.36 (s, 3H), 2.03 (s, 6H) ppm. ¹³C NMR (63 MHz, CDCl₃) δ 151.83, 145.24, 138.94, 136.39, 136.09, 133.49, 130.32, 130.28, 129.20, 128.69, 128.60, 128.17, 127.86, 127.20, 125.83, 102.59, 21.26, 17.77 ppm. MS (EI, 70 eV): m/z (%): 337 (95, [M+]), 323 (100), 261 (20), 234 (40), 189 (10), 104 (10), 77 (15). IR (ATR), 3040, 2952, 2919, 2856, 1605, 1546, 1484, 1460, 1407, 1377, 1357, 1300, 1283, 1213, 1183, 1156, 1104, 1074, 1027, 1000, 974, 956, 943, 912, 852, 803, 757, 747, 733, 714, 689, 667, 626, 618, 602, 576, 540, 517, 497, 483, 449 cm⁻¹. HRMS calcd for C₂₄H₂₃N₂ [M+H]+ 339.1861 found 339.1862

3,5-Bis(4-chlorophenyl)-1-mesityl-1H-pyrazole (3b)

The general procedure was followed. 3,5-bis(4-chlorophenyl)-1H-pyrazol 1b (115.6 mg, 0.4 mmol), mesityl(phenyl)iodonium triflate 2a (208 mg, 0.44 mmol), were dissolved in 25w/w% NH₃ (aq) solution-toluene 1:1 (20 mL) stirred at RT for 20 min.
Yellowish-white oil (106.6 mg, 0.26 mmol, yield: 65 %). Rₖ: 0.55 (in hexane:EtOAc 10:1) M.p. 112-113 °C. ¹H NMR (250 MHz, CDCl₃) δ 7.75 (d, J = 8.5 Hz, 2H), 7.28 (d, J = 8.5 Hz, 2H), 7.13 (d, J = 8.6 Hz, 2H), 7.02 (d, J = 8.6 Hz, 2H), 6.83 (s, 2H), 6.76 (s, 1H), 2.22 (s, 3H), 1.85 (s, 6H) ppm. ¹³C NMR (63 MHz, CDCl₃) δ 150.93, 144.32, 139.35, 135.97, 134.36, 133.72, 131.86, 129.37, 128.95, 128.56, 128.45, 127.74, 127.11, 102.66, 21.25, 17.71 ppm. MS (EI, 70 eV): m/z (%): 408 (70), 406 (95, [M⁺]), 393 (70), 391 (100), 295 (30), 268 (70), 178 (15), 138 (40), 115 (20), 103 (10), 91 (35), 77 (25). IR (ATR), 2958, 2920, 2853, 1601, 1478, 1439, 1406, 1378, 1352, 1341, 1290, 1274, 1241, 1209, 1186, 1174, 1106, 1091, 1065, 1035, 1012, 975, 955, 945, 848, 837, 828, 779, 754, 740, 728, 721, 715, 685, 632, 626, 607, 575, 546, 535, 518, 503, 486, 442, 416 cm⁻¹. HRMS calcd for C₂₄H₂₁Cl₂N₂ [M+H]⁺ 407.1082 found 407.1081

3,5-Bis(4-bromophenyl)-1-mesityl-1H-pyrazole (3c)

The general procedure was followed. 3,5-bis(4-bromophenyl)-1H-pyrazol 1c (110 mg, 0.5 mmol), mesityl(phenyl)iodonium triflate 2a (260 mg, 0.55 mmol), were dissolved in 25w/w% NH₃ (aq) solution-toluene 1:1 (20 mL) stirred at RT for 20 min. White crystals (188.9 mg, 0.38 mmol, yield: 76%). Rₖ: 0.53 (in hexane:EtOAc 10:1) m.p. 131-132 °C. ¹H NMR (250 MHz, CDCl₃) δ 7.79 (d, J = 8.5 Hz, 2H), 7.54 (d, J = 8.5 Hz, 2H), 7.39 (d, J = 8.5 Hz, 2H), 7.06 (d, J = 8.5 Hz, 2H), 6.93 (s, 2H), 6.87 (s, 1H), 2.33 (s, 3H), 1.95 (s, 6H) ppm. ¹³C NMR (63 MHz, CDCl₃) δ 150.97, 144.39, 139.43, 135.98, 132.26, 131.94, 131.87, 129.41, 128.99, 128.73, 127.46, 122.65, 121.96, 102.67, 21.29, 17.74 ppm. MS (EI, 70 eV): m/z (%): 498 (55), 496 (100, [M⁺]), 494 (55), 483 (50), 481 (100), 479 (50), 415 (10), 341 (30), 339 (30), 314 (70), 312(70), 232 (30), 218 (40), 200 (30), 184 (45), 182 (45), 160 (50), 115 (50), 103 (45), 91 (75), 77 (60). IR (ATR), 1475, 1438, 1403, 1378, 1349, 1289, 1268, 1211, 1186, 1105, 1070, 1033, 1011, 975, 963, 954, 946, 848, 833, 826, 818, 780, 733, 717, 685, 630, 605, 574, 539, 518, 503, 481, 467, 449, 424, 419, 414 cm⁻¹. HRMS calcd for C₂₄H₂₁Br₂N₂ [M+H]⁺ 495.0071 found 495.0085

3,5-Bis(4-tolyl)-1-mesityl-1H-pyrazole (3d)

The general procedure was followed. 3,5-bis(4-tolyl)-1H-pyrazol 1d (124 mg, 0.5 mmol), mesityl (phenyl)iodonium triflate 2a (260 mg, 0.55 mmol), were dissolved in 25w/w% NH₃ (aq) solution-toluene 1:1 (20 mL) stirred at RT for 20 min.
Yellowish oil (151.4 mg, 0.41 mmol, yield: 83%). R<sub>r</sub>: 0.50 (in hexane:EtOAc 10:1) m.p. 114-115 °C. <sup>1</sup>H NMR (250 MHz, CDCl<sub>3</sub>) δ 7.85 (d, J = 8.1 Hz, 2H), 7.25 (d, J = 8.0 Hz, 2H), 7.12 (t, J = 8.0 Hz, 4H), 6.93 (s, 2H), 6.88 (s, 1H), 2.41 (s, 3H), 2.34 (s, 3H), 2.32 (s, 3H), 2.00 (s, 6H) ppm.<sup>13</sup>C NMR (63 MHz, CDCl<sub>3</sub>) δ 151.84, 145.20, 138.83, 138.00, 137.54, 136.56, 136.19, 130.79, 129.37, 129.31, 129.16, 127.53, 127.06, 125.76, 102.10, 21.38, 21.29, 21.26, 17.77 ppm. MS (EI, 70 eV): m/z (%): 366 (90, [M+H]<sup>+</sup>), 351 (100), 275 (15), 248 (70), 232 (10), 281 (15), 175, (30), 118 (50), 103 (10), 91 (55), 77 (20). IR (ATR), 2914, 2857, 1495, 1438, 1377, 1349, 1280, 1264, 1218, 1205, 1181, 1113, 1069, 1035, 1022, 976, 965, 956, 943, 856, 828, 819, 784, 745, 722, 716, 692, 640, 614, 594, 573, 539, 521, 498, 485 cm<sup>-1</sup>. 

HRMS calcd for C<sub>26</sub>H<sub>27</sub>N<sub>2</sub>[M+H]<sup>+</sup> 367.2174 found 367.2176

3,5-bis(4-ansisyl)-1-mesityl-1H-pyrazole (3e)

The general procedure was followed. 3,5-bis(4-methoxiphenyl)-1H-pyrazol 1e (140 mg, 0.5 mmol), mesityl(phenyl)iodonium triflate 2a (260 mg, 0.55 mmol), were dissolved in 25w/w% NH<sub>3</sub> (aq) solution-toluene 1:1 (20 mL) stirred at RT for 20 min. White solid (157.6 mg, 0.40 mmol, yield: 79 %). R<sub>r</sub>: 0.21 (in hexane:EtOAc 10:1) m.p. 114-115 °C. <sup>1</sup>H NMR (250 MHz, CDCl<sub>3</sub>) δ 7.88 (d, J = 8.7 Hz, 2H), 7.15 (d, J = 8.8 Hz, 2H), 6.99 (s, 2H), 6.93 (s, 2H), 6.85 – 6.72 (m, 3H), 3.84 (s, 3H), 3.76 (s, 3H), 2.33 (s, 3H), 2.00 (s, 6H) ppm.<sup>13</sup>C NMR (63 MHz, CDCl<sub>3</sub>) δ 159.47, 159.41, 151.53, 144.95, 138.79, 136.48, 136.18, 129.14, 128.47, 127.04, 126.42, 122.92, 114.05, 113.98, 101.43, 55.36, 55.22, 21.22, 17.75 ppm. MS (EI, 70 eV): m/z (%): 398 (100, [M+H]<sup>+</sup>), 383 (90), 291 (10), 264 (50), 221 (10), 199 (15), 165, (10), 134 (20), 91 (15), 77 (15). IR (ATR), 2999, 2962, 2920, 2837, 1611, 1582, 1547, 1496, 1466, 1452, 1438, 1393, 1376, 1356, 1301, 1293, 1257, 1245, 1212, 1184, 1170, 1107, 1076, 1056, 1029, 972, 956, 941, 866, 831, 809, 789, 747, 731, 722, 693, 662, 637, 615, 594, 574, 536, 512, 497, 466, 447, 435, 425, 407 cm<sup>-1</sup>. HRMS calcd for C<sub>26</sub>H<sub>27</sub>N<sub>2</sub>O<sub>2</sub>[M+H]<sup>+</sup> 399.2073 found 399.2071

3,5-Bis(4-nitrophenyl)-1-mesityl-1H-pyrazole (3f)

The general procedure was followed. 3,5-bis(4-nitrophenyl)-1H-pyrazol 1f (155 mg, 0.5 mmol), mesityl(phenyl)iodonium triflate 2a (260 mg, 0.55 mmol), were dissolved in 25w/w% NH<sub>3</sub> (aq) solution-toluene 1:1 (20 mL) stirred at RT for 20 min. Yellow solid (157.5 mg, 0.50 mmol, yield: 74%). R<sub>r</sub>: 0.50 (in hexane:EtOAc 4:1) m.p. 210-214 °C. <sup>1</sup>H NMR (250 MHz, CDCl<sub>3</sub>) δ 8.30 (d, J = 8.8 Hz, 1H), 8.11 (dd, J = 16.5, 8.9 Hz,
2H), 7.37 (d, J = 8.9 Hz, 1H), 7.14 (s, 1H), 6.98 (s, 1H), 2.36 (s, 2H), 1.96 (s, 4H) ppm. $^{13}$C NMR (63 MHz, CDCl$_3$) δ 150.12, 147.52, 143.64, 140.16, 139.14, 135.68, 135.50, 129.72, 127.81, 126.35, 124.29, 124.18, 104.79, 21.34, 17.71 ppm. MS (EI, 70 eV): m/z (%): 398 (100, [M$^+$]), 383 (90), 291 (10), 264 (50), 221 (10), 199 (15), 165, (10), 134 (20), 91 (15), 77 (15). IR (ATR), 1596, 1541, 1518, 1508, 1491, 1478, 1445, 1376, 1338, 1286, 1214, 1194, 1107, 1075, 1036, 1010, 976, 956, 943, 858, 852, 831, 795, 751, 730, 710, 693, 678, 635, 623, 607 cm$^{-1}$. HRMS calcd for C$_{24}$H$_{21}$N$_4$O$_4$ [M+H]$^+$ 429.1563 found 429.1565

4-Iodo-1-mesityl-3,5-diphenyl-1H-pyrazole (3g)

The general procedure was followed. 4-iodo-3,5-diphenyl-1H-pyrazol 1g (173 mg, 0.5 mmol), mesityl(phenyl)iodonium triflate 2a (260 mg, 0.55 mmol), were dissolved in 25w/w% NH$_3$ (aq) solution-toluene 1:1 (20 mL) stirred at RT for 20 min. Yellowish-white solid (209.1 mg, 0.45 mmol, yield: 90%). R$_f$: 0.46 (in hexane:EtOAc 10:1) m.p. 163-166 °C. $^1$H NMR (250 MHz, CDCl$_3$) δ 7.99 (dd, $J = 8.2$, 1.6 Hz, 2H), 7.53 – 7.38 (m, 3H), 7.31 (s, 5H), 6.84 (s, 2H), 2.26 (s, 3H), 2.01 (s, 6H) ppm. $^{13}$C NMR (63 MHz, CDCl$_3$) δ 153.00, 146.68, 139.07, 135.99, 135.84, 133.23, 129.75, 129.01, 128.67, 128.37, 128.28, 60.49, 21.20, 17.89 ppm. MS (EI, 70 eV): m/z (%): 464 (100, [M$^+$]), 449 (25), 387 (25), 337 (60), 322 (25), 299 (10), 284 (10), 189 (30), 119, (10), 91 (20), 77 (20). IR (ATR), 3053, 3032, 2918, 2852, 1602, 1485, 1473, 1450, 1399, 1379, 1347, 1330, 1286, 1259, 1172, 1151, 1106, 1079, 1069, 1029, 1001, 967, 943, 916, 890, 860, 774, 767, 745, 729, 703, 693, 668, 640, 623, 614, 576, 555, 513, 440, 406 cm$^{-1}$. HRMS calcd for C$_{24}$H$_{21}$NI$_2$ [M+H]$^+$ 465.0828 found 465.0834

3,5-Bis(4-chlorophenyl)-4-iodo-1-mesityl-1H-pyrazole (3h)

The general procedure was followed. 4-jodo-2,5-bis(4-chlorophenyl)-1H-pyrazol 1h (83 mg, 0.2 mmol), mesityl(phenyl)iodonium triflate 2a (104 mg, 0.22 mmol), were dissolved in 25w/w% NH$_3$ (aq) solution-toluene 1:1 (20 mL) stirred at RT for 20 min. Yellowish-white solid (80 mg, 0.15 mmol, yield: 75%). R$_f$: 0.35 (in hexane:EtOAc 30:1) m.p. 154-160 °C. $^1$H NMR (250 MHz, CDCl$_3$) δ 7.82 (d, $J = 8.6$ Hz, 1H), 7.35 (d, $J = 8.5$ Hz, 1H), 7.18 (dd, 2H), 6.77 (s, 1H), 2.19 (s, 2H), 1.89 (s, 4H) ppm. $^{13}$C NMR (63 MHz, CDCl$_3$) δ 152.06, 145.72, 139.46, 135.65, 135.25, 134.46, 131.50, 130.98, 129.87, 129.19, 128.75, 128.56, 127.98, 127.14, 60.51, 21.23, 17.87 ppm. MS (EI, 70 eV): m/z (%): 533(0, [M$^+$]), 420 (40), 405 (100), 390 (35), 369 (15), 355 (15), 340 (10), 293 (15), 281 (15), 223 (70), 207 (60), 201 (40).
187 (70), 138 (80), 91 (95), 77 (50). IR (ATR), 2954, 2921, 2852, 1598, 1508, 1486, 1472, 1466, 1437, 1425, 1376, 1338, 1294, 1258, 1210, 1148, 1091, 1032, 1013, 968, 943, 884, 854, 839, 830, 825, 791, 786, 758, 750, 739, 726, 719, 701, 691, 649, 632, 619, 609, 576, 564, 539, 525, 500, 492, 483, 464, 426, 406 cm\(^{-1}\). HRMS calcd for C\(_{24}\)H\(_{19}\)Cl\(_2\)IN\(_2\) [M+H]\(^+\) 533.0048 found 533.0056

![Structure of 4-Iodo-1-mesityl-3,5-di(4-tolyl)-1H-pyrazole (3i)](image)

**4-Iodo-1-mesityl-3,5-di(4-tolyl)-1H-pyrazole (3i)**

The general procedure was followed. 4-iodo-3,5-di(4-tolyl)-1H-pyrazol 1i (187 mg, 0.5 mmol), mesityl(phenyl)iodonium triflate 2a (260 mg, 0.55 mmol), were dissolved in 25w/w% NH\(_3\) (aq) solution-toluene 1:1 (20 mL) stirred at RT for 20 min. Yellowish-white solid (212.3 mg, 0.43 mmol, yield: 86%). R\(_f\).: 0.57 (in hexane:EtOAc 10:1) m.p. 163-166 °C. \(^1\)H NMR (250 MHz, CDCl\(_3\)) \(\delta 7.91\) (d, \(J = 8.1\) Hz, 2H), 7.30 (d, \(J = 8.0\) Hz, 2H), 7.22 (d, \(J = 8.2\) Hz, 2H), 7.12 (d, \(J = 8.1\) Hz, 2H), 6.86 (s, 2H), 2.44 (s, 3H), 2.33 (s, 3H), 2.28 (s, 3H), 2.03 (s, 6H) ppm. \(^{13}\)C NMR (63 MHz, CDCl\(_3\)) \(\delta 152.90, 146.56, 138.86, 138.80, 138.01, 136.12, 135.83, 130.40, 129.50, 128.97, 128.93, 128.49, 126.82, 60.37, 21.44, 21.16, 17.86 ppm. MS (EI, 70 eV): \(m/z\) (%): 492 (100, [M]+), 477 (30), 401 (20), 365 (70), 350 (45), 273 (10), 218 (20), 202 (50), 175 (10), 119 (20), 91 (40), 77 (15). IR (ATR), 3018, 2954, 2919, 2854, 1479, 1426, 1385, 1374, 1342, 1310, 1300, 1282, 1257, 1186, 1144, 1113, 1074, 1031, 1020, 967, 942, 882, 852, 843, 837, 818, 801, 749, 724, 720, 706, 693, 677, 639, 626, 608, 586, 576, 552, 525, 488, 471, 427 cm\(^{-1}\). HRMS calcd for C\(_{26}\)H\(_{26}\)IN\(_2\) [M+H]\(^+\) 493.1141 found 493.1145

![Structure of 4-Iodo-3,5-dimethyl-1-mesityl-1H-pyrazole (3j)](image)

**4-Iodo-3,5-dimethyl-1-mesityl-1H-pyrazole (3j)**

The general procedure was followed. 3,5-dimethyl-4-jodo-1H-pyrazol 1j (111 mg, 0.5 mmol), mesityl(phenyl)iodonium triflate 2a (260 mg, 0.55 mmol), were dissolved in 25w/w% NH\(_3\) (aq) solution-toluene 1:1 (20 mL) stirred at RT for 20 min. Yellowish-white solid (151.1 mg, 0.44 mmol, yield: 89%). R\(_f\).: 0.37 (in hexane:EtOAc 10:1) m.p. 100-103 °C. \(^1\)H NMR (250 MHz, CDCl\(_3\)) \(\delta 6.91\) (s, 2H), 2.30 (s, 3H), 2.28 (s, 3H), 2.00 (s, 3H), 1.89 (s, 6H) ppm. \(^{13}\)C NMR (63 MHz, CDCl\(_3\)) \(\delta 150.33, 141.73, 139.03, 136.19, 135.48, 128.88, 62.25, 21.15, 17.21, 14.25 ppm. MS (EI, 70 eV): \(m/z\) (%): 340 (100, [M]+), 325 (100), 284 (5), 213 (20), 198 (50), 172 (20), 157 (20), 128, (10), 115 (20), 91 (30), 78 (20). IR (ATR), 2944, 2919, 2853, 1610, 1532, 1492, 1457, 1434, 1406, 1385, 1374, 1353,
4-Iodo-3,5-dimethyl-1-mesityl-1H-pyrazole (3j)

Mesityl-(2,5-dimethyl-3-pyrazolium)iodonium bistriflate 2az (111 mg, 0.5 mmol), were dissolved in 25w/w% NH₃ (aq) solution-toluene 1:1 (20 mL) stirred at RT for 2 days. The reaction was then diluted with CH₂Cl₂ (10 mL). The aqueous layer was then extracted with CH₂Cl₂ (2 × 10 mL) and all organics combined and dried (MgSO₄). The suspension was then filtered, concentrated and purified by flash chromatography.

Yellowish-white solid (130 mg, 0.38 mmol, yield: 75%). Rₜ.: 0.37 (in hexane:EtOAc 10:1) m.p. 99-102 °C, ¹H NMR (250 MHz, CDCl₃) δ 6.92 (s, 2H), 2.31 (s, 3H), 2.29 (s, 3H), 2.01 (s, 3H), 1.90 (s, 6H) ppm. ¹³C NMR (63 MHz, CDCl₃) δ 150.40, 141.87, 139.16, 136.26, 135.44, 128.95, 62.32, 21.23, 17.29, 14.34, 11.89 ppm. MS (EI, 70 eV): m/z (%): 340 (100, [M⁺]), 325 (100), 284 (5), 213 (20), 198 (50), 172 (20), 157 (20), 128, (10), 115 (20), 91 (30), 78 (20). IR (ATR), 2944, 2919, 2853, 1610, 1532, 1492, 1457, 1434, 1406, 1385, 1374, 1353, 1327, 1306, 1287, 1098, 1058, 1032, 1021, 946, 859, 787, 744, 657, 622, 596, 576, 510, 498 cm⁻¹. HRMS calcd for C₁₄H₁₈IN₂ [M+H]⁺ 341.0515 found 341.0513

4-Iodo-1-mesityl-1H-pyrazole (3k)

The general procedure was followed. 4-jodo-1H-pyrazol 1k (97 mg, 0.5 mmol), mesityl(phenyl)iodonium triflate 2a (260 mg, 0.55 mmol), were dissolved in 25w/w% NH₃ (aq) solution-toluene on 1:1 (20 mL) stirred at RT for 20 min.

Yellowish-white oil (113.7 mg, 0.36 mmol, yield: 72%) Rₜ.: 0.51 (in hexane:EtOAc 10:1). ¹H NMR (250 MHz, CDCl₃) δ 7.73 (s, 1H), 7.48 (s, 1H), 6.94 (s, 2H), 6.33 (s, 3H), 1.97 (s, 6H) ppm. ¹³C NMR (63 MHz, CDCl₃) δ 145.24, 139.36, 136.44, 135.77, 135.35, 129.02, 56.65, 21.20, 17.32 ppm. MS (EI, 70 eV): m/z (%): 312(85, [M⁺]), 186 (100), 170 (90), 158 (80), 143 (40), 130 (15), 115, (30), 103 (15), 91 (30), 77 (25). IR (ATR), 3121, 2954, 2920, 2857, 2857, 1608, 1508, 1485, 1439, 1395, 1377, 1365, 1342, 1315, 1302, 1251, 1179, 1167, 1129, 1030, 962, 940, 849, 801, 736, 618, 580, 554 cm⁻¹. HRMS calcd for C₁₂H₁₄IN₂ [M+H]⁺ 313.0202 found 313.0210

1,2,5-Tri(4-bromophenyl)-1H-pyrazole (3l)
The general procedure was followed. 3,5-p-bromophenyl-4-jodo-1H-pyrazol 1c (189 mg, 0.5 mmol), bis-(4-bromophenyl)iodonium triflate 2aw (323 mg, 0.55 mmol), were dissolved in 25w/w% NH₃ (aq) solution-toluene 1:1 (20 mL) stirred at rt for 4 h. Yellowish-white solid (195.7 mg, 0.37 mmol, yield: 73%). Rₜ: 0.42 (in hexane:EtOAc 10:1) m.p. 176-180 °C. ¹H NMR (250 MHz, CDCl₃) δ 7.77 (d, J = 8.5 Hz, 1H), 7.56 (d, J = 8.5 Hz, 1H), 7.50 (dd, J = 8.6, 2.0 Hz, 2H), 7.22 (d, J = 8.7 Hz, 1H), 7.13 (d, J = 8.5 Hz, 1H), 6.78 (s, 1H) ppm. ¹³C NMR (63 MHz, CDCl₃) δ 151.47, 143.62, 138.80, 132.39, 132.13, 132.01, 131.65, 130.34, 129.07, 127.50, 126.73, 123.25, 122.45, 121.64, 105.74 ppm. IR (ATR), 2923, 2851, 1893, 1649, 1588, 1489, 1480, 1431, 1408, 1398, 1357, 1342, 1300, 1294, 1260, 1232, 1213, 1177, 1098, 1068, 1057, 1007, 968, 953, 939, 845, 830, 825, 793, 750, 718, 709, 703, 681, 671, 632, 594, 547, 524, 488, 471 cm⁻¹. HRMS calcd for C₂₁H₁₄Br₃N₂ [M+H]^+ 530.8707 found 530.8712

1,3,5-Tri(4-chlorophenyl)-4-iodo-1H-pyrazole (3m)

The general procedure was followed. 4-jodo-2,5-bis(4-chlorophenyl)-1H-pyrazol 1h (83 mg, 0.2 mmol), bis(4-chlorophenyl)iodonium triflate 2av (110 mg, 0.22 mmol), were dissolved in 25w/w% NH₃ (aq) solution-toluene 1:1 (20 mL) stirred at RT for 4 h. Yellowish-white solid 72 mg, 0.14 mmol, yield: 69%). Rₜ: 0.47 (in hexane:EtOAc 30:1) m.p. 175-178 °C. ¹H NMR (250 MHz, CDCl₃) δ ¹H 7.88 (d, J = 8.6 Hz, 1H), 7.43 (dd, J = 11.7, 8.6 Hz, 2H), 7.32 – 7.26 (m, 3H), 7.20 (dd, J = 8.9, 6.4, 1.9 Hz, 3H) ppm. ¹³C NMR (63 MHz, CDCl₃) δ 152.48, 144.58, 138.16, 135.79, 134.86, 133.82, 131.86, 131.03, 129.90, 129.34, 129.27, 128.73, 128.30, 125.94 ppm. MS (EI, 70 eV): m/z (%): 524(0, [M⁺]), 488 (5), 398 (15), 362 (25), 326 (10), 292 (5), 258 (10), 223 (60), 187 (60), 111 (100), 75 (60). IR (ATR), 2959, 2923, 2852, 1598, 1492, 1473, 1437, 1417, 1399, 1382, 1357, 1341, 1301, 1259, 1150, 1090, 1076, 1025, 1014, 977, 963, 832, 827, 794, 769, 738, 729, 722, 700, 682, 641, 632, 619, 587, 519, 513, 508, 486, 464 cm⁻¹. HRMS calcd for C₂₁H₁₃Cl₃In₂ [M+H]^+ 530.8707 found 530.8712

1,3,5-Triphenyl-1H-pyrazole (3n)³⁴,³⁵

The general procedure was followed. 3,5-diphenyl-1H-pyrazol 1a (110 mg, 0.5 mmol), diphenyliodonium triflate 2ba (235 mg, 0.55 mmol), were dissolved in 25w/w% NH₃(aq) solution-toluene 1:1 (20 mL) stirred at RT for 2 h.
Yellowish-white solid (124.6 mg, 0.42 mmol, yield: 84 %). R\text{f}: 0.42 (in hexane:EtOAc 10:1) m.p. 135-137 °C, (Lit.: 136 °C). \textsuperscript{1}H NMR (250 MHz, CDCl\textsubscript{3}) \(\delta\) 7.87 – 7.79 (m, 1H), 7.38 – 7.13 (m, 7H), 6.72 (s, 1H) ppm. \textsuperscript{13}C NMR (63 MHz, CDCl\textsubscript{3}) \(\delta\) 152.05, 144.48, 140.23, 133.14, 130.67, 128.99, 128.84, 128.74, 128.57, 128.39, 128.10, 127.51, 125.92, 125.39, 105.31 ppm. MS (EI, 70 eV): \(m/z\) (%): 296 (100, [M\textsuperscript{+}]), 267 (5), 218 (5), 192 (15), 180 (10), 165 (30), 147 (10), 133 (10), 116 (10), 89, (15), 77 (65). IR (ATR), 1595, 1546, 1494, 1482, 1455, 1433, 1414, 1362, 1311, 1213, 1174, 1159, 1086, 1072, 1064, 1034, 1020, 1000, 972, 957, 921, 843, 814, 777, 770, 763, 700, 691, 680, 667, 598,524 cm\textsuperscript{-1}.

4-Iodo-1,3,5-triphenyl-1H-pyrazole (3o)

The general procedure was followed. 4-iodo-3,5-diphenyl-1H-pyrazol 1g (173 mg, 0.5 mmol), biphenyliodonium triflate 2ba (235 mg, 0.55 mmol), were dissolved in 25w/w% NH\textsubscript{3} (aq) solution-toluene 1:1 (20 mL) stirred at RT for 4 h.

White cristaline solid (147.8 mg, 0.35 mmol, yield: 70 %). R\text{f}: 0.42 (in hexane:EtOAc 10:1) m.p. 151-153 °C, (Lit.: 139-140 °C). \textsuperscript{1}H NMR (250 MHz, CDCl\textsubscript{3}) \(\delta\) 7.90 – 7.83 (m, 2H), 7.42 – 7.09 (m, 13H) ppm. \textsuperscript{13}C NMR (63 MHz, CDCl\textsubscript{3}) \(\delta\) 153.08, 145.49, 140.00, 132.91, 130.61, 130.33, 129.15, 128.89, 128.68, 128.58, 128.54, 128.35, 127.62, 124.84, 63.72 ppm. MS (EI, 70 eV): \(m/z\) (%): 498 (55), 422 (100, [M\textsuperscript{+}]), 294 (25), 189 (60), 180 (115), 165 (10), 147 (15), 115 (5), 89 (10), 77 (50). IR (ATR), 3065, 1591, 1495, 1477, 1458, 1450, 1444, 1431, 1397, 1357, 1350, 1328, 1318, 1256, 1202, 1181, 1169, 1159, 1147, 1087, 1072, 1028, 1001, 975, 961, 912, 834, 776, 767, 758, 738, 700, 691, 684, 676, 635, 615, 527, 516, 508, 463, 454 cm\textsuperscript{-1}.

1-(4-Fluorophenyl)-3,5-diphenyl-1H-pyrazole (3p)

The general procedure was followed. 3,5-diphenyl-1H-pyrazol 1a (110 mg, 0.5 mmol), bis-(4-fluorophenyl)iodonium triflate 2au (256 mg, 0.55 mmol), were dissolved in 25w/w% NH\textsubscript{3} (aq) solution-toluene 1:1 (20 mL) stirred at RT for 4 h.

Yellowish-white solid 131.9 mg, 0.42 mmol, yield: 84%). R\text{f}: 0.53 (in hexane:EtOAc 10:1) m.p. 106-108 °C, (Lit.: 106-107 °C). \textsuperscript{1}H NMR (250 MHz, CDCl\textsubscript{3}) \(\delta\) 7.86 – 7.76 (m, 2H), 7.32 (dd, \(J = 11.4, 4.4\) Hz, 2H), 7.18 (tdd, \(J = 7.2, 6.2, 4.2\) Hz, 8H), 6.96 – 6.84 (m, 2H), 6.70 (s, 1H) ppm. \textsuperscript{19}F NMR (235 MHz, CDCl\textsubscript{3}) \(\delta\) -113.94 (s) ppm. \textsuperscript{13}C NMR (63 MHz, CDCl\textsubscript{3}) \(\delta\) 163.68, 159.74, 152.11, 144.57, 136.40, 136.35, 132.97, 130.40, 128.82, 128.77, 128.66, 128.53, 128.19, 127.20, 127.06, 125.90, 116.08, 115.71, 105.26 ppm. MS (EI, 70 eV): \(m/z\) (%): 314 (100, [M\textsuperscript{+}]), 286 (5), 210 (15), 198 (15), 183 (15), 157 (5), 109, (10), 95 (20), 77...
The general procedure was followed. 3,5-diphenyl-1H-pyrazole 1a (220.2 mg, 1.0 mmol), bis-(4-chlorophenyl)iodonium triflate 2a (549 mg, 1.1 mmol), were dissolved in 25w/w% NH₃ (aq) solution-dichloroethane 1:1 (40 mL) stirred at RT for 20 min. The reaction mixture was diluted with CH₂Cl₂ (20 mL). The aqueous layer was extracted with CH₂Cl₂ (2 × 20 mL) and all combined organic phase were dried over magnesium sulphate. The suspension was then filtered, concentrated in vacuo and purified by flash chromatography on silicagel. White solid (288 mg, 0.87 mmol, yield: 87%). Rₛ: 0.53 (in hexane:EtOAc 10:1) m.p. 134-138 °C, (Lit.: 118-120 °C). ¹H NMR (250 MHz, CDCl₃) δ 7.83 (dd, J = 8.2, 1.3 Hz, 2H), 7.35 (dd, J = 8.0, 6.4 Hz, 2H), 7.29 – 7.11 (m, 10H), 6.73 (s, 1H) ppm. ¹³C NMR (63 MHz, CDCl₃) δ 152.37, 144.56, 138.74, 133.13, 132.91, 130.41, 129.17, 128.88, 128.82, 128.77, 128.68, 128.29, 126.42, 125.94, 105.70 ppm. MS (EI, 70 eV): m/z (%): 464 (100, [M⁺]), 450 (30), 387 (30), 337 (60), 321 (40), 259 (20), 218 (20), 189 (80), 119 (25), 91 (40). IR (ATR), 1546, 1494, 1458, 1449, 1434, 1412, 1362, 1300, 1209, 1101, 1091, 1082, 1065, 1026, 1013, 969, 956, 914, 834, 818, 807, 762, 751, 714, 702, 694, 676, 667, 631, 616, 596, 543, 524, 503, 473, 443, 425, 419 cm⁻¹.

The general procedure was followed. 3,5-diphenyl-1H-pyrazole 1a (110.1 mg, 0.55 mmol), bis-(4-bromophenyl)iodonium triflate 2a (323.4 mg, 0.55 mmol), were dissolved in 25% NH₃ (aq) solution- dichloroethane 1:1 (20 mL) stirred at RT for 20 min. White solid (150 mg, 0.20 mmol, yield: 80%). Rₛ: 0.53 (in hexane:EtOAc 10:1) m.p. 118-120 °C (Lit.: 133-135 °C). ¹H NMR (250 MHz, CDCl₃) δ 7.81 (d, J = 7.1 Hz, 2H), 7.50 – 7.01 (m, 12H), 6.71 (s, 1H) ppm. ¹³C NMR (63 MHz, CDCl₃) δ 152.38, 144.48, 139.21, 139.15, 133.53, 132.88, 132.09, 130.38, 128.86, 128.80, 128.66, 128.28, 126.65, 126.91, 121.03, 105.76 ppm. MS (EI, 70 eV): m/z (%): 464 (100, [M⁺]), 450 (30), 387 (30), 337 (60), 321 (40), 259 (20), 218 (20), 189 (80), 119 (25), 91 (40). IR (ATR), 1546, 1494, 1458, 1449, 1434, 1412, 1362, 1300, 1209, 1180, 1101, 1091, 1082, 1065, 1026, 1013, 969, 956, 914, 834, 818, 807, 762, 751, 714, 702, 694, 676, 667, 631, 616, 596, 543, 524, 503, 473, 443, 425, 419 cm⁻¹.
1073, 1062, 1026, 1010, 1002, 967, 955, 922, 915, 832, 809, 764, 741, 710, 694, 678, 667, 629, 616, 595, 531, 518, 484, 470, 446, 401 cm$^{-1}$.

1-(4-Nitrophenyl)-3,5-diphenyl-1H-pyrazole (3s)$^{40}$

The general procedure was followed. 3,5-diphenyl-1H-pyrazol 1a (110 mg, 0.5 mmol), 4-nitrophenyl(phenyl)iodonium triflate 2j (261 mg, 0.55 mmol), were dissolved in 25w/w% NH$_3$ (aq) solution-toluene 1:1 (20 mL) stirred at RT for 20 min. Yellowish-white solid (144.1 mg, 0.42 mmol, yield: 84%). R$_f$: 0.34 (in hexane:EtOAc 10:1) m.p. 104-106 °C, (Lit.: 137-138 °C, 177-177.5 °C). $^1$H NMR (250 MHz, CDCl$_3$) $\delta$ 8.22 – 8.14 (m, 2H), 7.93 (dd, $J = 8.2, 1.4$ Hz, 2H), 7.63 – 7.51 (m, 2H), 7.43 (ddd, $J = 10.0, 8.6, 5.3$ Hz, 6H), 7.35 – 7.27 (m, 2H), 6.86 (s, 1H) ppm. $^{13}$C NMR (63 MHz, CDCl$_3$) $\delta$ 153.44, 145.97, 145.10, 145.04, 132.43, 130.25, 129.25, 129.09, 128.99, 128.92, 128.75, 126.07, 124.55, 107.34 ppm. MS (EI, 70 eV): m/z (%): 341 (100, [M$^+$]), 294 (40), 190 (15), 179 (10), 165 (10), 146 (15), 102, (10), 89 (10), 77 (20). IR (ATR), 2922, 1613, 1594, 1552, 1506, 1499, 1488, 1459, 1449, 1435, 1406, 1368, 1327, 1308, 1262, 1219, 1171, 1110, 1084, 1076, 1066, 1025, 966, 952, 917, 854, 821, 764, 753, 700, 689, 671, 664, 631, 619, 615, 598, 542, 522, 493, 480, 424, 404 cm$^{-1}$.

1-(2-Tolyl)-3,5-diphenyl-1H-pyrazole (3t)$^{41}$

The general procedure was followed. 3,5-diphenyl-1H-pyrazol 1a (110 mg, 0.5 mmol), 2-tolyl(phenyl)iodonium triflate 2x (244 mg, 0.55 mmol), were dissolved in 25w/w% NH$_3$(aq) solution-toluene 1:1 (20 mL) stirred at RT for 4h. Yellowish-white solid (120 mg, 0.39 mmol, yield: 77% in dichloroethane), (128 mg, 0.41 mmol, yield: 83% in toluene). R$_f$: 0.38 (in hexane:EtOAc 10:1) m.p. 86-89 °C (Lit.: 95-96 °C). $^1$H NMR (250 MHz, CDCl$_3$) $\delta$ 8.04 – 7.91 (m, 1H), 7.46 (dd, $J = 8.0, 6.5$ Hz, 1H), 7.40 – 7.22 (m, 5H), 6.92 (s, 1H), 2.07 (s, 2H) ppm. $^{13}$C NMR (63 MHz, CDCl$_3$) $\delta$ 151.80, 145.56, 139.58, 135.76, 133.26, 131.18, 130.29, 129.10, 128.73, 128.54, 128.30, 128.23, 128.01, 127.95, 126.76, 125.87, 103.27, 17.79 ppm. MS (EI, 70 eV): m/z (%): 310 (100, [M$^+$]), 295 (50), 387 (30), 233 (20), 206 (40), 191 (10), 178 (10), 165 (15), 154 (15), 104, (30), 91 (15), 77 (15). IR (ATR), 3040, 2924, 2851, 1604, 1584, 1545, 1493, 1484, 1461, 1411, 1381, 1360, 1281, 1212, 1203, 1186, 1173, 1155, 1123, 1082, 1074, 1065, 1028, 1000, 973, 955, 946, 912, 864, 795, 767, 751, 726, 716, 691, 671, 602, 561, 531, 512, 503, 460, 453, 432, 420, 402 cm$^{-1}$. HRMS calcd for C$_{22}$H$_{19}$N$_2$ [M+H]$^+$ 311.1548
The general procedure was followed. 3,5-diphenyl-1H-pyrazol 1a (110 mg, 0.5 mmol), 2,6-dimethylphenyl(phenyl)iodonium triflate 2am (252 mg, 0.55 mmol), were dissolved in 25w/w% NH₃ (aq) solution-toluene 1:1 (20 mL) stirred at RT for 20 min.

Colorless solid (153.8 mg, 0.47 mmol, yield: 95%). Rₜ: 0.39 (in hexane:EtOAc 10:1) m.p. 124-126 °C. ¹H NMR (250 MHz, CDCl₃) δ 8.07 – 8.00 (m, 1H), 7.49 (dd, J = 8.0, 6.6 Hz, 1H), 7.34 – 7.24 (m, 1H), 7.16 (d, J = 7.6 Hz, 1H), 7.00 (s, 1H), 2.10 (s, 1H) ppm.

13C NMR (63 MHz, CDCl₃) δ 151.98, 145.21, 138.88, 136.50, 133.41, 130.13, 129.16, 128.67, 128.57, 128.47, 128.21, 127.89, 127.15, 125.82, 102.66, 17.79 ppm. MS (EI, 70 eV): m/z (%): 324 (100, [M⁺]), 309 (95), 247 (30), 220 (40), 204 (15), 189 (10), 178 (10), 165 (10), 154 (20), 104, 91 (10), 77 (35). IR (ATR), 3039, 2922, 2853, 1603, 1547, 1484, 1474, 1460, 1406, 1379, 1356, 1326, 1282, 1254, 1208, 1184, 1175, 1162, 1153, 1104, 1079, 1072, 1028, 999, 971, 955, 909, 837, 793, 777, 769, 754, 745, 709, 690, 606, 590, 556, 524, 496, 445 cm⁻¹. HRMS calcd for C₂₃H₂₁N₂ [M+H]⁺ 325.1705 found 325.1709

1-(2-Trifluoromethylphenyl)-3,5-diphenyl-1H-pyrazole (3v)

The general procedure was followed. 3,5-diphenyl-1H-pyrazol 1a (110 mg, 0.5 mmol), 2-trifluoromethylphenyl(phenyl)iodonium triflate 2ao (274 mg, 0.55 mmol), were dissolved in 25w/w% NH₃ (aq) solution-toluene 1:1 (20 mL) stirred at RT for 2 h.

Yellowish-white solid (155.5 mg, 0.43 mmol, yield: 85%). Rₜ: 0.33 (in hexane:EtOAc 10:1) m.p. 104-106 °C. ¹H NMR (250 MHz, CDCl₃) δ 7.90 – 7.78 (m, 1H), 7.73 (dd, J = 5.6, 3.8 Hz, 1H), 7.46 (dd, J = 5.5, 3.8 Hz, 1H), 7.36 (dd, J = 11.5, 4.5 Hz, 1H), 7.30 – 7.22 (m, 1H), 7.22 – 7.10 (m, 3H), 6.79 (s, 1H) ppm. ¹³C NMR (63 MHz, CDCl₃) δ 152.26, 146.33, 133.00, 130.77, 128.97, 128.38, 128.62, 128.37 (t, J = 6.6 Hz), 127.91 (d, J = 4.9 Hz), 126.10, 104.28, 29.83 (q, J = 3.5 Hz) ppm. ¹⁹F NMR (235 MHz, CDCl₃) δ -59.92 (s) ppm. MS (EI, 70 eV): m/z (%): 310 (100, [M⁺]), 295 (50), 387 (30), 233 (20), 206 (40), 191 (10), 178 (10), 165 (15), 154 (15), 104, 91 (15), 77 (15). IR (ATR), 3063, 2922, 1605, 1585, 1543, 1500, 1485, 1476, 1464, 1456, 1411, 1361, 1311, 1268, 1211, 1187, 1170, 1154, 1133, 1111, 1079, 1074, 1063, 1047, 1034, 1029, 1001, 992, 971, 962, 954, 916, 883, 853, 802, 783, 776, 769, 757, 725, 694, 675, 666, 641, 620, 606, 593, 530, 514, 504, 484, 461, 437, 419, 412, 402 cm⁻¹. HRMS calcd for C₂₂H₁₆F₃N₂ [M+H]⁺ 365.1266 found 365.1267
1-(3-Chloro-2-tolyl)-3,5-diphenyl-1H-pyrazole (3w)

The general procedure was followed. 3,5-diphenyl-1H-pyrazol 1a (110 mg, 0.5 mmol), 3-chloro-2-tolyl(phenyl)iodonium triflate 2ap (263 mg, 0.55 mmol), were dissolved in 25w/w % NH₃ (aq) solution-toluene 1:1 (20 mL) stirred at RT for 2 h.

Yellowish-white solid (150.8 mg, 0.44 mmol, yield: 87%). Rₖ: 0.57 (in hexane:EtOAc 10:1) m.p. 107-111 °C. ¹H NMR (250 MHz, CDCl₃) δ 7.87 – 7.79 (m, 1H), 7.38 – 7.29 (m, 1H), 7.28 – 7.23 (m, 1H), 7.22 – 7.05 (m, 4H), 6.78 (s, 1H), 1.98 (s, 2H) ppm. ¹³C NMR (63 MHz, CDCl₃) δ 152.20, 145.95, 140.83, 135.74, 134.81, 133.05, 130.00, 128.79, 128.68, 128.50, 128.21, 128.04, 127.07, 127.03, 125.93, 103.59, 15.42 ppm. MS (EI, 70 eV): m/z (%): 344 (100, [M+H]+), 343 (95), 329 (50), 309 (10), 267 (25), 240 (20), 204 (20), 154 (25), 104 (30), 89 (25), 77 (35). IR (ATR), 3038, 2927, 2853, 1598, 1572, 1543, 1484, 1473, 1459, 1445, 1408, 1380, 1359, 1306, 1269, 1213, 1201, 1182, 1150, 1079, 1028, 1011, 998, 970, 955, 914, 794, 770, 756, 720, 689, 625, 616, 595, 541, 524, 504, 480, 439 cm⁻¹. HRMS calcd for C₂₂H₁₈ClN₂ [M+H]+ 345.1159 found 345.1153

1,2,5-Tri-(4-nitrophenyl)-1H-pyrazole (3x)

The general procedure was followed. 3,5-(4-nitrophenyl)-1H-pyrazol 1f (155 mg, 0.5 mmol), 4-nitrophenyl(4-anisyl)iodonium tosylate 2o (290 mg, 0.55 mmol), were dissolved in 25w/w % NH₃ (aq) solution-toluene 1:1 (20 mL) stirred at RT for 20 min.

Yellow solid (135.7 mg, 0.31 mmol, yield: 63%). Rₖ: 0.35 (in hexane:EtOAc 4:1) m.p. 255-257 °C, (Lit.: 218-222 °C). ¹H NMR (250 MHz, [D₆]DMSO) δ 8.30 (ddd, J = 31.0, 15.9, 8.7 Hz, 8H), 7.66 (t, J = 8.3 Hz, 5H) ppm. ¹³C NMR (63 MHz, [D₆]DMSO) δ 150.50, 147.54, 147.34, 146.47, 143.80, 143.33, 138.11, 135.40, 130.10, 126.64, 125.92, 124.99, 124.41, 124.14, 109.23 ppm. MS (EI, 70 eV): m/z (%): 423 (4, [M+H]+), 372 (5), 347 (9), 345 (22), 310 (16), 309 (100). IR (ATR), 3119, 2925, 2851, 1594, 1546, 1508, 1420, 1338, 1293, 1218, 1188, 1107, 1075, 1065, 1013, 974, 954, 911, 860, 852, 831, 812, 752, 744, 711, 698, 633, 595, 569, 553, 531, 514 cm⁻¹. HRMS calcd for C₂₁H₁₄N₅O₆ [M+H]+ 432.0944 found 432.0950
The general procedure was followed. 4-iodo-3,5-di(4-tolyl)-1H-pyrazol 1i (187 mg, 0.5 mmol), 2-azidomethylphenyl(4-anisyl)iodonium tosylate 2al (260 mg, 0.55 mmol), were dissolved in 25w/w% NH₃ (aq) solution-toluene 1:1 (20 mL) stirred at RT for 20 min. Yellowish-white oil (208.3 mg, 0.41 mmol, yield: 82%). Rᶠ: 0.46 (in hexane:EtOAc 10:1) m.p. 108-110 °C. ¹H NMR (250 MHz, CDCl₃) δ 7.87 (d, J = 8.1 Hz, 2H), 7.47 (dd, J = 7.7, 1.1 Hz, 1H), 7.41 – 7.19 (m, 6H), 7.19 – 7.07 (m, 3H), 4.42 (s, 2H), 2.44 (s, 3H), 2.35 (s, 3H) ppm. ¹³C NMR (63 MHz, CDCl₃) δ 153.46, 146.92, 139.18, 138.55, 138.44, 132.92, 130.18, 129.91, 129.61, 129.24, 129.20, 129.07, 128.66, 128.54, 128.46, 126.50, 62.32, 50.91, 21.48 ppm. MS (EI, 70 eV): m/z (%): 477 (70, [M⁺]), 449 (20), 386 (25), 350 (40), 308 (100), 281(10), 258, (15), 233 (50), 202 (45), 175 (10), 91 (35), 77 (25) 65 (20). IR (ATR), 2919, 2853, 2114, 2095, 2071, 2020, 1991, 1979, 1613, 1603, 1495, 1477, 1456, 1419, 1391, 1378, 1343, 1307, 1280, 1240, 1151, 1125, 1103, 1069, 1037, 1018, 964, 890, 871, 839, 823, 799, 775, 753, 729, 723, 712, 700, 682, 668, 641, 623, 580, 558, 538, 527, 510, 487, 469, 463, 447 cm⁻¹. HRMS calcd for C₂₄H₂₁N₅ [M+H]⁺ 506.0842 found 506.0846

1-(2-(Azidomethyl)phenyl)-3,5-diphenyl-1H-pyrazole (3z)

The general procedure was followed. 3,5-diphenyl-1H-pyrazol 1a (110 mg, 0.5 mmol), 2-azidomethylphenyl(4-anisyl)iodonium tosylate 2al (296 mg, 0.55 mmol), were dissolved in 25w/w% NH₃ (aq) solution-toluene 1:1 (20 mL) stirred at RT for 2 h. Yellowish-white solid (172.1mg, 0.49 mmol, yield: 98%). Rᶠ: 0.29 (in hexane:EtOAc 10:1) m.p. 86-88 °C. ¹H NMR (250 MHz, CDCl₃) δ 7.84 (dd, J = 8.2, 1.2 Hz, 1H), 7.44 (d, J = 7.6 Hz, 1H), 7.39 – 7.31 (m, 2H), 7.30 – 7.04 (m, 4H), 6.81 (s, 1H), 4.33 (s, 1H) ppm. ¹³C NMR (63 MHz, CDCl₃) δ 152.49, 145.95, 138.78, 133.27, 132.96, 129.89, 129.30, 128.92, 128.82, 128.71, 128.59, 128.55, 128.34, 128.30, 125.98, 104.13, 51.04 ppm. MS (EI, 70 eV): m/z (%): 323 (100, [M⁺]), 295 (90), 282 (40), 267 (15), 246 (80), 220 (70), 205 (15), 193 (25), 165 (40), 104, (30), 91 (20), 77 (55). IR (ATR), 2956, 2920, 2850, 2103, 1545, 1494, 1480, 1464, 1458, 1436, 1429, 1409, 1359, 1345, 1294, 1285, 1260, 1217, 1180, 1157, 1084, 1074, 1059, 1026, 971, 954, 913, 807, 769, 758, 736, 721, 694, 688, 667, 639, 619, 602, 538, 499 cm⁻¹. HRMS calcd for C₂₂H₁₈N₅ [M+H]⁺ 352.1562 found 352.1563
The general procedure was followed. 3,5-diphenyl-1H-pyrazol 1a (110 mg, 0.5 mmol), 4-anisyl(3-pyridinium)iodonium bistriflate 2m (336 mg, 0.55 mmol), were dissolved in 25w/w% NH₃ (aq) solution-toluene 1:1 (20 mL) stirred at RT for 2 h.

Yellowish-white solid (119.3 mg, 0.40 mmol, yield: 80%). R₆: 0.43 (in hexane:EtOAc 1.5:1) m.p. 83-86 °C. ¹H NMR (250 MHz, CDCl₃) δ 8.50 (d, J = 2.2 Hz, 1H), 8.35 (d, J = 4.7 Hz, 1H), 7.85 – 7.74 (m, 2H), 7.54 (dd, J = 8.2, 1.5 Hz, 1H), 7.28 (t, J = 7.2 Hz, 2H), 7.23 – 7.03 (m, 7H), 6.68 (s, 1H) ppm. ¹³C NMR (63 MHz, CDCl₃) δ 152.79, 147.96, 145.85, 144.69, 136.60, 132.56, 131.86, 129.86, 128.72, 128.69, 128.66, 128.24, 125.76, 123.30, 105.89 ppm. MS (EI, 70 eV): m/z (%): 297 (90, [M⁺]+), 296 (100), 269 (10), 220 (10), 193 (30), 166 (15), 139 (5), 102 (5), 89 (5), 79 (20), 78 (20). IR (ATR), 2920, 2852, 1631, 1602, 1590, 1575, 1546, 1485, 1478, 1460, 1443, 1422, 1405, 1358, 1321, 1261, 1217, 1191, 1175, 1159, 1107, 1084, 1077, 1062, 1030, 1022, 917, 953, 924, 855, 847, 815, 762, 713, 690, 672, 667, 621, 614, 605, 524, 493, 448, 412, 400 cm⁻¹. HRMS calcd for C₂₀H₁₆N₃ [M+H]+ 298.1344 found 298.1348

The general procedure was followed. 3,5-diphenyl-1H-pyrazol 1a (110 mg, 0.5 mmol), 4-anisyl(6-quinolinium)iodonium bistriflate 2n (364 mg, 0.55 mmol), were dissolved in 25w/w% NH₃ (aq) solution-toluene 1:1 (20 mL) stirred at RT for 2 h.

Yellow oil (104.7 mg, 0.30 mmol, yield: 60%). R₆: 0.34 (in hexane:EtOAc 1:1). ¹H NMR (250 MHz, CDCl₃) δ 8.78 (dd, J = 4.2, 1.5 Hz, 1H), 7.99 – 7.90 (m, 2H), 7.88 – 7.81 (m, 2H), 7.77 (d, J = 2.2 Hz, 1H), 7.56 (dd, J = 9.0, 2.3 Hz, 1H), 7.33 (t, J = 7.3 Hz, 2H), 7.29 – 7.22 (m, 2H), 7.19 (s, 5H), 6.76 (s, 1H) ppm. ¹³C NMR (63 MHz, CDCl₃) δ 152.54, 150.72, 146.89, 144.70, 138.12, 136.31, 132.89, 130.40, 130.15, 128.33, 128.77, 128.71, 128.64, 128.25, 128.21, 127.13, 125.91, 122.82, 121.81, 105.87 ppm. MS (EI, 70 eV): m/z (%): 347 (100, [M⁺]+), 319 (5), 270 (10), 242 (20), 216 (10), 189 (10), 140 (5), 128 (20), 115 (10), 101 (15), 89 (5), 77 (25). IR (ATR), 3040, 2923, 1624, 1596, 1570, 1549, 1500, 1484, 1458, 1429, 1408, 1357, 1321, 1303, 1285, 1229, 1215, 1177, 1156, 1120, 1075, 1063, 1027, 1000, 983, 955, 941, 909, 881, 862, 835, 794, 757, 728, 690, 668, 644, 637, 619, 610, 604, 592, 583, 541, 525, 518, 496, 475, 448 cm⁻¹. HRMS calcd for C₂₄H₁₈N₃ [M+H]+ 348.1501 found 348.1500
The general procedure was followed. 3,5-diphenyl-1\(H\)-pyrazole 1a (110 mg, 0.5 mmol), 4-tolyl(2-thienyl)iodonium bromide 2a (210 mg, 0.55 mmol), were dissolved in 25 wt% NH\(_3\) solution-toluene 1:1 (20 mL) stirred at RT for 4 h. Yellowish-white solid (142.5 mg, 0.46 mmol, yield: 92%). R\(_f\): 0.35 (in hexane:EtOAc 10:1) m.p. 104-108 °C (Lit.: 106-108 °C). \(^1\)H NMR (250 MHz, CDCl\(_3\)) \(\delta\) 7.84 – 7.77 (m, 2H), 7.28 (t, \(J = 7.4\) Hz, 2H), 7.19 (d, \(J = 7.2\) Hz, 1H), 7.16 – 7.09 (m, 7H), 6.98 (d, \(J = 8.2\) Hz, 2H), 6.67 (s, 1H), 2.19 (s, 3H) ppm. \(^{13}\)C NMR (63 MHz, CDCl\(_3\)) \(\delta\) 151.73, 144.32, 137.74, 137.34, 133.14, 130.65, 129.52, 128.74, 128.66, 128.47, 128.23, 127.96, 125.84, 125.19, 104.97, 21.13 ppm. MS (EI, 70 eV): \(m/z\) (%): 310 (100, [M\(^+\)]), 295 (5), 282 (5), 267 (5), 206 (10), 191 (10), 178 (5), 165 (10), 154 (10), 104, (5), 91 (20), 77 (15). IR (ATR), 3038, 2917, 1603, 1545, 1511, 1479, 1462, 1435, 1412, 1380, 1361, 1308, 1286, 1263, 1211, 1177, 1156, 1108, 1087, 1077, 1065, 1044, 1027, 1020, 1000, 971, 956, 943, 917, 848, 822, 809, 805, 761, 721, 710, 691, 676, 667, 624, 617, 593, 524, 503, 493, 452, 440, 422, 418, 401 cm\(^{-1}\). HRMS calcd for C\(_{24}\)H\(_{23}\)N\(_2\)O\(_3\) [M+H\(^+\)] 387.1709 found 387.1710

The general procedure was followed. 3,5-bis(p-anisyl)-1\(H\)-pyrazole 1e (140 mg, 0.5 mmol), 4-anisyl(2-thienyl)iodonium bromide 2u (210 mg, 0.55 mmol), were dissolved in 25 wt% NH\(_3\) solution-toluene 1:1 (20 mL) stirred at RT for 4 h. Drabbb solid (21.3 mg, 0.06 mmol, yield: 11%). R\(_f\): 0.32 (in hexane:EtOAc 10:1) m.p. 132-137 °C. \(^1\)H NMR (250 MHz, CDCl\(_3\)) \(\delta\) 7.86 (d, \(J = 8.7\) Hz, 2H), 7.29 (d, \(J = 8.9\) Hz, 2H), 6.96 (d, \(J = 8.6\) Hz, 2H), 6.86 (t, \(J = 8.8\) Hz, 4H), 6.69 (s, 1H), 3.85 (s, 3H), 3.82 (s, 3H), 3.80 (s, 3H) ppm. \(^{13}\)C NMR (63 MHz, CDCl\(_3\)) \(\delta\) 159.74, 159.67, 158.97, 151.23, 144.46, 133.14, 130.08, 128.65, 127.32, 127.01, 125.52, 122.83, 114.23, 114.15, 114.02, 103.76, 55.61, 55.42, 55.38 ppm. MS (EI, 70 eV): \(m/z\) (%): 386 (100, [M\(^+\)]), 371 (15), 193 (10), 77 (10). IR (ATR), 3001, 2955, 2926, 2853, 2834, 1612, 1575, 1555, 1512, 1494, 1463, 1445, 1432, 1397, 1360, 1349, 1302, 1291, 1243, 1168, 1106, 1072, 1066, 1027, 971, 955, 939, 834, 818, 808, 793, 750, 734, 720, 695, 656, 640, 611, 590, 534, 515, 488, 456, 422, 417 cm\(^{-1}\). HRMS calcd for C\(_{24}\)H\(_{23}\)N\(_2\)O\(_3\) [M+H\(^+\)] 387.1709 found 387.1710
The general procedure was followed. 4-jodo-2,5-bisp-tolyl-1H-pyrazol 1i (187 mg, 0.5 mmol), 4-tolyl(2-thienyl)iodonium bromide 2as (180 mg, 0.55 mmol), were dissolved in 25w/w\% NH₃ (aq) solution-toluene 1:1 (20 mL) stirred at RT for 20 min. Yellowish-white solid (190.5 mg, 0.41 mmol, yield: 82%). Rf.: 0.53 (in hexane:EtOAc 10:1) m.p. 131-135 °C. ¹H NMR (250 MHz, CDCl₃) δ 7.75 (d, J = 8.1 Hz, 2H), 7.18 (d, J = 8.0 Hz, 2H), 7.15 – 7.03 (m, 6H), 6.97 (d, J = 8.4 Hz, 2H), 2.31 (s, 3H), 2.28 (s, 3H), 2.21 (s, 3H) ppm. ¹³C NMR (63 MHz, CDCl₃) δ 152.83, 145.37, 138.97, 138.21, 137.79, 137.37, 130.46, 130.20, 129.43, 129.27, 129.03, 128.56, 127.51, 63.35, 21.54, 21.49, 21.15 ppm. MS (EI, 70 eV): m/z (%): 464(100, [M⁺]), 337 (20), 231 (5), 218 (10), 178 (5), 153, (10), 115 (5), 91 (35), 65 (20). IR (ATR), 3040, 3020, 2915, 2857, 1614, 1513, 1484, 1444, 1418, 1390, 1346, 1313, 1259, 1210, 1180, 1152, 1111, 1079, 1043, 1024, 1019, 977, 963, 943, 909, 845, 839, 815, 794, 728, 709, 691, 672, 648, 639, 622, 608, 586, 529, 515, 485, 475, 434, 420 cm⁻¹. HRMS calcd for C₂₄H₂₂IN₂ [M+H⁺] 465.0828 found 465.0829

The general procedure was followed. 3,5-diphenyl-1H-pyrazol 1a (110 mg, 0.5 mmol), 4-anisyl(2-thienyl)iodonium bromide 2u (210 mg, 0.55 mmol), were dissolved in 25w/w\% NH₃ (aq) solution-toluene 1:1 (20 mL) stirred at RT for 4 h. Yellowish-white solid (46.6 mg, 0.14 mmol, yield: 29%). Rf.: 0.34 (in hexane:EtOAc 10:1) m.p. 115-117 °C (Lit.: 128-130 °C). ¹H NMR (250 MHz, CDCl₃) δ 7.88 – 7.78 (m, 2H), 7.33 (t, J = 7.3 Hz, 2H), 7.25 – 7.14 (m, 8H), 6.77 (d, J = 8.9 Hz, 2H), 6.72 (s, 1H), 3.70 (s, 3H) ppm. ¹³C NMR (63 MHz, CDCl₃) δ 158.97, 151.64, 144.49, 133.36, 133.09, 130.58, 128.78, 128.73, 128.55, 128.31, 128.05, 126.87, 125.91, 114.20, 104.73, 55.57 ppm. MS (EI, 70 eV): m/z (%): 310 (100, [M⁺]), 295 (5), 282 (5), 267 (5), 206 (10), 191 (10), 178 (5), 165 (10), 154 (10), 104, (5), 91 (20), 77 (15). IR (ATR), 3065, 3012, 2961, 2932, 2836, 1604, 1585, 1545, 1518, 1511, 1481, 1461, 1450, 1441, 1413, 1362, 1300, 1252, 1215, 1181, 1169, 1155, 1105, 1086, 1068, 1029, 998, 982, 973, 955, 934, 919, 846, 818, 808, 804, 770, 760, 729, 712, 691, 678, 667, 641, 623, 616, 593, 532, 503, 483 cm⁻¹.
References

4-Iodo-3,5-bis-(4-chlorophenyl)-1H-pyrazole (1h)
4-Iodo-3,5-di-p-tolyl-1H-pyrazole (1i)
Mesityl(2,4,6-trimethoxyphenyl)iodonium triflate (2f)
4-Tolyl(3-pyridinium)iodonium bistriflate (2l)
4-Anisyl(7-quinolinium)iodonium bistriflate (2n)
3,5-Xylyl(phenyl)iodonium triflate (2y)

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Chemical shifts for peaks:
A (d) 8.21
B (s) 7.90
C (t) 7.66
D (d) 7.52
E (s) 7.29
F (s) 7.29
4-Chlorophenyl(4-fluorophenyl)iodonium triflate (2ah)

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GZ51241_1Hdmsa
C13CFO
4-Bromophenyl(4-fluorophenyl)iodonium triflate (2ai)
2,4-Difluorophenyl(phenyl)iodonium triflate (2an)
3-Chloro-2-toly(phenyl)iodonium triflate (2ap)
Mesityl(2,4,6-trimethoxyphenyl)iodonium tosilate (2ax)
1-Mesityl-3,5-diphenyl-pirazole (3a)
3,5-Bis(4-chlorophenyl)-1-mesityl-1H-pyrazole (3b)
3,5-Bis(4-bromophenyl)-1-mesityl-1H-pyrazole (3c)
3,5-Bis(4-tolyl)-1-mesityl-1H-pyrazole (3d)
3,5-bis(4-anisyl)-1-mesityl-1H-pyrazole (3e)
3,5-Bis(4-nitrophenyl)-1-mesityl-1H-pyrazole (3f)
4-Iodo-1-mesityl-3,5-diphenyl-1H-pyrazole (3g)
3,5-Bis(4-chlorophenyl)-4-iodo-1-mesityl-1H-pyrazole (3h)
4-Iodo-1-mesityl-3,5-di(4-tolyl)-1H-pyrazole (3i)
4-Iodo-3,5-dimethyl-1-mesityl-1H-pyrazole (3j)
4-Iodo-1-mesityl-1H-pyrazole (3k)
1,2,5-Tri(4-bromophenyl)-1H-pyrazole (3l)

**S** - 86
1,3,5-Tri(4-chlorophenyl)-4-iodo-1H-pyrazole (3m)
1,3,5-Triphenyl-1H-pyrazole (3n)
4-Iodo-1,3,5-triphenyl-1H-pyrazole (3o)
1-(4-Fluorophenyl)-3,5-diphenyl-1H-pyrazole (3p)
1-(4-Chlorophenyl)-3,5-diphenyl-1H-pyrazole (3q)
1-(4-Bromophenyl)-3,5-diphenyl-1H-pyrazole (3r)
1-(4-Nitrophenyl)-3,5-diphenyl-1H-pyrazole (3s)
1-(2-Tolyl)-3,5-diphenyl-1H-pyrazole (3t)
1-(2,6-Xylyl)-3,5-diphenyl-1H-pyrazole (3u)
1-(2-Trifluoromethylphenyl)-3,5-diphenyl-1H-pyrazole (3v)

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1-(3-Chloro-2-tolyl)-3,5-diphenyl-1H-pyrazole (3w)
1,2,5-Tri-(4-nitrophenyl)-1H-pyrazole (3x)
1-(2-(Azidomethyl)phenyl)-4-iodo-3,5-di-p-tolyl-1H-pyrazole (3y)
1-(2-(Azidomethyl)phenyl)-3,5-diphenyl-1H-pyrazole (3z)
1-(3-Pyridyl)-3,5-diphenyl-1H-pyrazole (3aa)
6-(3,5-Diphenyl-1H-pyrazol-1-yl)quinoline (3ab)
1-(4-Tolyl)-3,5-diphenyl-1H-pyrazole (3ac)
1,2,5-Tri-(4-anisyl)-1H-pyrazole (3ad)
4-Iodo-1,2,5-tri-(4-tolyl)-1H-pyrazole (3ae)
1-(4-Anisyl)-3,5-diphenyl-1H-pyrazole (3af)