Cu-catalyzed N-3-Arylation of Hydantoins Using Diaryliodonium salts

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General considerations

Commercially available reagents and solvents were purchased from Sigma-Aldrich and TCI and used without further purification unless stated otherwise. Thin layer chromatography was performed on 60 F254 silica coated aluminum plates from Merck and visualized using UV-light or KMnO4-stain.

Flash chromatography was performed on silica gel from Merck (Silicagel 60, 40-63 µm) using Isco Inc. CombiFlash Companion with PeakTrack software (v.1.4.10).

$^1$H- and $^{13}$C-NMR spectra were recorded either using Bruker AVI600, AVII600 or AVIIIHD800 spectrometers. $^{19}$F-NMR spectra were recorded using Bruker AVIIIHD400 spectrometer. Spectra were calibrated using the residual solvent peaks for CDCl$_3$ ($^1$H: 7.24 ppm; $^{13}$C: 77.00 ppm) and DMSO-$d_6$ ($^1$H: 2.50 ppm; $^{13}$C: 39.52 ppm). All spectra were recorded at 298 K.

FTIR spectra were recorded in ATR (Bruker ATR A225/Q) on a Vertex 80 Bruker infrared spectrophotometer, equipped with a DTGS detector; 32 interferograms (recorded at 4 cm$^{-1}$ resolution) were typically averaged for each spectrum.

High-resolution mass spectra (HRMS) were obtained by electron spray ionization (ESI) on Bruker Daltonik GmbH MAXIS II ETD spectrometer.

Optical rotations were measured on a polarimeter using a 10 cm cell with a Na 589 nm filter. The specific solvents and concentrations (in g/100 mL) are indicated.

Melting point for crystalline compounds were measured on a Stuart SMP10 melting point apparatus and are reported uncorrected.
Single crystal X-ray diffraction data for 3a

Single crystals of 3a were grown by recrystallization from hot toluene. Around 50 mg of 3a was added to a 4 mL screw cap vial equipped with a magnet together with a few drops of toluene and heated (on a heating block) to around 105°C. Upon heating, more toluene was added dropwise until full dissolution. The vial was fitted with a screw cap and left over night for slow cooling to room temperature.

Figure SI-1: ORTEP representation of two dimeric 3a. Ellipsoids are of 50% probability.
Table SI-1: Crystal data and structure refinement for two dimeric 3a

Crystal data

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<tr>
<td>Empirical formula</td>
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<td>Formula weight</td>
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<td>Crystal color, shape</td>
<td>Colorless, block</td>
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<td>Crystal size (mm^3)</td>
<td>0.8 × 0.15 × 0.15</td>
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<td>Crystal system, space group</td>
<td>Triclinic, P-1</td>
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<tr>
<td>a, b, c (Å)</td>
<td>9.3780(4), 14.1940(6), 14.3864(6)</td>
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<tr>
<td>α, β, γ (°)</td>
<td>115.6560(10), 106.5660(10), 91.210(2)</td>
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<td>Volume (Å³)</td>
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<td>Temperature (K)</td>
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<tr>
<td>ρ calc (g cm⁻³)</td>
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<tr>
<td>μ (mm⁻¹)</td>
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<tr>
<td>F(000)</td>
<td>728.0</td>
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<tr>
<td>Radiation</td>
<td>MoKα (λ = 0.71073)</td>
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Data collection and refinement

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<td>2Θ range for data collection (°)</td>
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<td>Index ranges</td>
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<td>R₁ = 0.1046, wR₂ = 0.2089</td>
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<td>Largest diff. peak/hole (e/Å³)</td>
<td>0.64/-0.72</td>
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Single crystal X-ray diffraction data for 3f
Single crystals of 3f were grown by slow evaporation from acetonitrile. In a 4 mL screw cap vial, 44 mg of 3f was dissolved in acetonitrile (1-2 mL). The vial was left open, and the solution was allowed to slowly evaporate over days at room temperature.

**Figure SI-2**: ORTEP representation of 3f. Ellipsoids are of 50% probability.
Table SI-2: Crystal data and structure refinement for 3f

**Crystal data**

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<td>Empirical formula</td>
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<td>a, b, c (Å)</td>
<td>8.3504(10), 9.5583(13), 10.1140(15)</td>
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<td>α, β, γ (°)</td>
<td>83.652(4), 87.334(3), 74.134(3)</td>
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<td>Volume (Å³)</td>
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**Data collection and refinement**

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Single crystal X-ray diffraction data for 3p

Single crystals of 3p were grown by slow evaporation from deuterated chloroform. In a 4 mL screw cap vial, 53 mg of 3p was dissolved in deuterated chloroform (0.5 mL). The vial was left open, and the solution was allowed to slowly evaporate over night at room temperature.

Figure SI-3: ORTEP representation of 3p. Ellipsoids are of 50% probability.
**Table SI-3: Crystal data and structure refinement for 3p**

**Crystal data**

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<td>α, β, γ (°)</td>
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**Data collection and refinement**

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Single crystal X-ray diffraction data for 3r

Single crystals of 3r were grown by slow evaporation from deuterated chloroform. In a 4 mL screw cap vial, 56 mg of 3r was dissolved in deuterated chloroform (0.5 mL). The vial was left open, and the solution was allowed to slowly evaporate over night at room temperature.

Figure SI-4: ORTEP representation of 3r. Ellipsoids are of 50% probability.
Table SI-4: Crystal data and structure refinement for 3r

**Crystal data**

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<td>Z</td>
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<tr>
<td>Temperature (K)</td>
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<td>F(000)</td>
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<td>Radiation</td>
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**Data collection and refinement**

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<td>Final R indexes [$</td>
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<td>Final R indexes [all data]</td>
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<td>Largest diff. peak/hole (e/Å$^3$)</td>
<td>0.35/-0.38</td>
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**Preparation of N-(4-iodobenzyl)phthalimide**

*N-(4-iodobenzyl)phthalimide* was prepared according as described in the literature\(^1\) with slight modifications as specified.

**N-(4-iodobenzyl)phthalimide [CAS: 411221-85-7]**

\[
\begin{align*}
\text{N} & \quad \text{O} \\
\text{O} & \quad \text{Br} \\
& \quad \text{DMF, rt, 12 h} \\
& \quad \text{Et}_2\text{O} \\
\end{align*}
\]

A 50 mL round-bottom flask was charged with 4-iodobenzyl bromide (2.227 g, 7.50 mmol, 1.0 eq), followed by phthalimide potassium salt (1.459 g, 7.88 mmol, 1.05 eq) and DMF (10 mL). The reaction mixture was stirred at room temperature for 12 h. The post reaction mixture was poured into water (100 mL) and extracted with Et\(_2\)O (5 x 50 mL). The combined organic layers were dried over Na\(_2\)SO\(_4\), filtered and concentrated under reduced pressure. The product was isolated as a colorless amorphous solid (2.42 g) in 89% yield.

\(^1\)H NMR (600 MHz, CDCl\(_3\)): \(\delta\) 7.82 (dd, \(J = 5.4, 3.1\) Hz, 2H), 7.69 (dd, \(J = 5.5, 3.0\) Hz, 2H), 7.62 (d, \(J = 8.4\) Hz, 2H), 7.16 (d, \(J = 8.3\) Hz, 2H), 4.75 (s, 2H).

\(^13\)C NMR (151 MHz, CDCl\(_3\)): \(\delta\) 167.9, 137.8, 135.9, 134.1, 132.0, 130.6, 123.4, 93.5, 41.1.

HRMS (ESI) \(m/z\) [M + Na]\(^+\): Calcd. for C\(_{15}\)H\(_{10}\)INNaO\(_2\): 385.9648, found: 385.9648.

The spectroscopic data is in accordance with the literature.\(^1\)
Preparation of chiral hydantoins 1e and 1g

Hydantoins 1e and 1g were prepared according to literature\textsuperscript{2} with slight modifications.

**General procedure**

Chiral amino acid (10.0 mmol, 1.0 eq), potassium cyanate (10.0 mmol, 1.0 eq) and water (3 mL) were added to a 10 mL round-bottom flask. The reaction mixture was refluxed for 1 h, and allowed to cool to room temp. Hydrochloric acid (35\% (v/v), 1.5 mL) was added and the reaction was refluxed for an additional 15 min. The mixture was cooled in an ice bath, affording colorless crystals, which were filtered off, washed with cold water (2-3 mL) and cold ethanol (0.5 mL).

**{(R)=5-Methylhydantoin (1e)} [CAS: 55147-68-7]**

1e was prepared from D-alanine according to the general procedure above, and obtained as colorless crystals (609.3 mg, 53%)

\[1^\text{H} \text{NMR (600 MHz, DMSO-d}_6\text{): } \delta 10.56 (s, 1H), 7.87 (s, 1H), 4.02 (qd, J = 6.9, 1.2 Hz, 1H), 1.21 (d, J = 7.0 Hz, 3H).\]

\[13^\text{C} \text{NMR (151 MHz, DMSO-d}_6\text{): } \delta 176.9, 157.2, 53.3, 17.3.\]

HRMS (ESI) m/z [M + Na]\textsuperscript{+}: Calcd. for C\textsubscript{4}H\textsubscript{6}N\textsubscript{2}NaO\textsubscript{2}\textsuperscript{+}: 137.0321, found: 137.0321.

\[ [\alpha]_{D}^{20} = + 40.8 \text{ (c = 0.50, ethanol)} \]

Melting point: 172 – 175\textdegree C

This compound has previously been reported.\textsuperscript{4}
(S)-Tetrahydro-1H-pyrrolo[1,2-c]imidazole-1,3(2H)-dione (1g) [CAS: 40856-87-9]

1g was prepared from L-proline according to the general procedure above, and obtained as colorless crystals (945.5 mg, 68%).

$^1$H NMR (600 MHz, DMSO-$d_6$): δ 10.74 (br s, 1H), 4.11 (dd, $J = 9.0, 7.6$ Hz, 1H), 3.46 (dt, $J = 10.8, 7.6$ Hz, 1H), 3.04 (ddd, $J = 10.8, 8.1, 4.7$ Hz, 1H), 2.09 – 2.02 (m, 1H), 2.01 – 1.87 (m, 2H), 1.71 – 1.58 (m, 1H).

$^{13}$C NMR (151 MHz, DMSO-$d_6$): δ 175.4, 161.0, 64.0, 44.9, 26.7, 26.7.


$[\alpha]_{D}^{20} = -127.5$ (c = 0.51, ethanol)

Melting point: 159 – 162°C

The spectroscopic data is in accordance with the literature.$^3$
Preparation of aryl(2,4,6-trimethoxyphenyl)iodonium salts

Aryl(TMP)iodonium salts 2a-s were prepared according to literature procedures.5,6

General procedure A: preparation of aryl(2,4,6-trimethoxyphenyl)iodonium tosylates

Aryl iodide (5.00 mmol, 1.00 eq) and acetonitrile (5 mL) were added to a 50 mL round-bottom flask and equipped with a magnetic stir bar. p-Toluenesulfonylic acid monohydrate (TsOH · H₂O) (5.05 mmol, 1.01 eq) was added in one portion at room temperature, followed by m-chloroperbenzoic acid (mCPBA) 5.05 mmol, 1.01 eq). The flask was lowered into a pre-heated heating block set at 77°C, and stirred for 30 minutes. 1,3,5-trimethoxybenzene (TMP-H) (5.05 mmol, 1.01 eq) was then added and the resulting solution was stirred for 5 minutes at 77 °C. The flask was cooled to ambient temperature before acetonitrile was removed under reduced pressure. The resulting crude oil was triturated with diethyl ether (40 mL). The precipitate was isolated by vacuum filtration with a fritted funnel and rinsed with diethyl ether (3 x 20 mL), then dried further under high vacuum to obtain the product in analytic purity.

General procedure B: preparation of aryl(2,4,6-trimethoxyphenyl)iodonium tosylates

Aryl iodide (2.50 mmol, 1.0 eq) and dichloromethane (19 mL) were added to a 50 mL round-bottom flask equipped with a magnetic stir bar. m-Chloroperbenzoic acid (mCPBA) (2.75 mmol, 1.1 eq) was added and the solution was stirred at rt for 4h. p-Toluenesulfonic acid monohydrate (TsOH · H₂O) (2.75 mmol, 1.1 eq) and 1,3,5-trimethoxybenzene (TMP-H) (3.75 mmol, 1.5 eq) were then added. The resulting solution was stirred at rt for 30 minutes, and evaporated under reduced pressure. The resulting crude oil was triturated with diethyl ether (20 mL). The precipitate was isolated by vacuum filtration with a fritted funnel and rinsed with diethyl ether (3 x 20 mL), then dried further under high vacuum to obtain the product in analytic purity.
Phenyl(2,4,6-trimethoxyphenyl)iodonium tosylate (2a) [CAS: 936326-60-2]

2a was prepared according to general procedure A on a 5 mmol scale and obtained as a pale, pink amorphous solid (2.48 g, 91%).

$^1$H NMR (600 MHz, DMSO-$d_6$): $\delta$ 7.91 (dd, $J = 8.4, 1.2$ Hz, 2H), 7.65 – 7.55 (m, 1H), 7.47 (ddd, $J = 10.1, 4.7, 2.7$ Hz, 4H), 7.10 (dd, $J = 8.4, 1.1$ Hz, 2H), 6.46 (s, 2H), 3.94 (s, 6H), 3.86 (s, 3H), 2.28 (s, 3H).

$^{13}$C NMR (151 MHz, DMSO-$d_6$): $\delta$ 166.2, 159.4, 145.7, 137.6, 134.3, 131.6, 131.5, 128.1, 125.5, 116.1, 92.1, 87.1, 57.3, 56.2, 20.8.

HRMS (ESI) m/z [M - OTs]$^+$: Calcd. for C$_{15}$H$_{16}$IO$_3$: 371.0139, found: 371.0139.

The spectroscopic data is in accordance with the literature.$^5$

4-Tolyl(2,4,6-trimethoxyphenyl)iodonium tosylate (2b) [CAS: 1868172-98-8]

2b was prepared according to general procedure A on a 5 mmol scale and obtained as a colorless amorphous solid (2.64 g, 95%).

$^1$H NMR (600 MHz, DMSO-$d_6$): $\delta$ 7.79 (d, $J = 8.4$ Hz, 2H), 7.47 (d, $J = 8.1$ Hz, 2H), 7.26 (d, $J = 8.1$ Hz, 2H), 7.10 (d, $J = 7.8$ Hz, 2H), 6.45 (s, 2H), 3.94 (s, 6H), 3.86 (s, 3H), 2.31 (s, 3H), 2.28 (s, 3H).

$^{13}$C NMR (151 MHz, DMSO-$d_6$): $\delta$ 166.1, 159.3, 145.8, 141.9, 137.5, 134.4, 132.1, 128.0, 125.5, 112.5, 92.0, 87.2, 57.3, 56.1, 20.8, 20.8.

HRMS (ESI) m/z [M - OTs]$^+$: Calcd. for C$_{16}$H$_{18}$IO$_3$: 385.0295, found: 385.0295.

The spectroscopic data is in accordance with the literature.$^5$
4-Nitro(2,4,6-trimethoxyphenyl)iodonium tosylate (2c) [CAS: 1868173-17-4]

2c was prepared according to general procedure A on a 5 mmol scale and obtained as a pale, orange amorphous solid (2.64 g, 95%).

$^1$H NMR (600 MHz, DMSO-$d_6$): δ 8.22 (d, $J = 9.1$ Hz, 2H), 8.14 (d, $J = 9.0$ Hz, 2H), 7.46 (d, $J = 8.1$ Hz, 2H), 7.10 (d, $J = 7.8$ Hz, 2H), 6.49 (s, 2H), 3.94 (s, 6H), 3.88 (s, 3H), 2.28 (s, 3H).

$^{13}$C NMR (151 MHz, DMSO-$d_6$): δ 166.5, 159.4, 149.1, 145.6, 137.7, 135.4, 128.1, 126.0, 125.5, 122.4, 92.2, 87.2, 57.4, 56.3, 20.8.

HRMS (ESI) m/z [M - OTs]$^+$: Calcd. for C$_{15}$H$_{15}$INO$^+$: 415.9989, found: 415.9989.

The spectroscopic data is in accordance with previously reported ones.$^5$

4-(Methoxycarbonyl)phenyl(2,4,6-trimethoxyphenyl)iodonium tosylate (2d) [CAS: 1868172-97-7]

2d was prepared according to general procedure A on a 5 mmol scale and obtained as a pale, pink amorphous solid (2.58 g, 86%).

$^1$H NMR (600 MHz, DMSO-$d_6$): δ 8.04 (d, $J = 8.6$ Hz, 2H), 7.96 (d, $J = 8.6$ Hz, 2H), 7.47 (d, $J = 8.1$ Hz, 2H), 7.10 (d, $J = 7.8$ Hz, 2H), 6.48 (s, 2H), 3.94 (s, 6H), 3.87 (s, 6H), 3.85 (s, 3H), 2.28 (s, 3H).

$^{13}$C NMR (151 MHz, DMSO-$d_6$): δ 166.4, 165.1, 159.4, 145.6, 137.6, 134.5, 132.1, 131.8, 128.0, 125.5, 120.9, 92.2, 86.9, 57.4, 56.2, 52.6, 20.7.

HRMS (ESI) m/z [M - OTs]$^+$: Calcd. for C$_{17}$H$_{18}$I$^+$O$^+$: 429.0193, found: 429.0193.

The spectroscopic data is in accordance with previously reported ones.$^5$
4-(Pentafluorosulfanyl)phenyl(2,4,6-trimethoxyphenyl)iodonium tosylate (2e) [NEW]

2e was prepared according to general procedure A on a 1.52 mmol scale and obtained as a colorless amorphous solid (816.0 mg, 80%).

\(^1\)H NMR (600 MHz, DMSO-\(d_6\)): \(\delta\) 8.11 (d, \(J = 8.7\) Hz, 2H), 7.99 (d, \(J = 9.1\) Hz, 2H), 7.46 (d, \(J = 8.1\) Hz, 2H), 7.10 (d, \(J = 7.8\) Hz, 2H), 6.49 (s, 2H), 3.95 (s, 6H), 3.88 (s, 3H), 2.28 (s, 3H).

\(^13\)C NMR (151 MHz, DMSO-\(d_6\)): \(\delta\) 166.5, 159.4, 154.4 (p, \(J_{CF} = 17.0\) Hz), 145.6, 137.7, 135.2, 128.7 (p, \(J_{CF} = 4.2\) Hz), 128.0, 125.5, 119.9, 92.2, 87.1, 57.4, 56.2, 20.8.

\(^19\)F NMR (376 MHz, DMSO-\(d_6\)): \(\delta\) 84.9 (p, \(J = 151.5\) Hz), 63.7 (d, \(J = 151.0\) Hz).

FTIR (neat, \(\nu_{\text{max}}\) cm\(^{-1}\)): 3097, 3062, 3027, 1584, 1343, 1230, 559

HRMS (ESI) m/z [M - OTs]: Calcd. for C\(_{15}\)H\(_{15}\)F\(_5\)IO\(_3\)S\(^+\): 496.9701, found: 496.9702.

(4-((N-phthalimidyl)benzyl))(2,4,6-trimethoxyphenyl)iodonium tosylate (2f) [CAS: 1868173-00-5]

2f was prepared according to general procedure A on a 5 mmol scale and obtained as a colorless amorphous solid (3.13 g, 89%).

\(^1\)H NMR (600 MHz, DMSO-\(d_6\)): \(\delta\) 7.90 – 7.82 (m, 6H), 7.47 (d, \(J = 8.1\) Hz, 2H), 7.39 (d, \(J = 8.6\) Hz, 2H), 7.10 (d, \(J = 7.7\) Hz, 2H), 6.44 (s, 2H), 4.80 (s, 2H), 3.93 (s, 6H), 3.85 (s, 3H), 2.27 (s, 3H).

\(^13\)C NMR (151 MHz, DMSO-\(d_6\)): \(\delta\) 167.7, 166.2, 159.3, 145.8, 140.4, 137.6, 134.6 (two overlapping signals), 131.5, 130.2, 128.0, 125.5, 123.3, 114.6, 92.0, 87.1, 57.3, 56.1, 40.3, 20.8.

HRMS (ESI) m/z [M - OTs]: Calcd. for C\(_{24}\)H\(_{21}\)INO\(_5\)S\(^+\): 530.0459, found: 530.0459.

The spectroscopic data is in accordance with previously reported ones.\(^\text{5}\)
3-Tolyl(2,4,6-trimethoxyphenyl)iodonium tosylate (2g) [CAS: 2293160-55-9]

2g was prepared according to general procedure A on a 5 mmol scale and obtained as a pale, pink amorphous solid (2.41 g, 87%).

$^1$H NMR (600 MHz, DMSO-$_d$6): $\delta$ 7.78 – 7.74 (m, 1H), 7.72 – 7.67 (m, 1H), 7.47 (d, $J = 7.9$ Hz, 3H), 7.41 (d, $J = 7.5$ Hz, 1H), 7.34 (t, $J = 7.8$ Hz, 1H), 7.10 (d, $J = 7.8$ Hz, 3H), 6.46 (s, 3H), 3.95 (s, 9H), 3.87 (s, 4H), 2.32 (s, 5H), 2.28 (s, 4H).

$^{13}$C NMR (151 MHz, DMSO-$_d$6): $\delta$ 134.2, 131.9, 131.1, 130.9, 127.7, 125.2, 91.8, 57.0, 55.9, 20.5, 20.4.

HRMS (ESI) m/z [M - OTs]$^+$: Calcd. for C$_{16}$H$_{18}$IO$_3$: 385.0295, found: 385.0295.

This compound has previously been reported.$^6$

(3-Bromophenyl)(2,4,6-trimethoxyphenyl)iodonium tosylate (2h) [CAS: 1799303-32-4]

2h was prepared according to general procedure A on a 5 mmol scale and obtained as a pale, pink amorphous solid (2.74 g, 88%).

$^1$H NMR (600 MHz, DMSO-$_d$6): $\delta$ 8.13 (t, $J = 1.8$ Hz, 1H), 7.86 (ddd, $J = 8.1$, 1.9, 0.9 Hz, 1H), 7.79 (ddd, $J = 8.1$, 1.9, 0.9 Hz, 1H), 7.47 (d, $J = 8.1$ Hz, 2H), 7.42 (d, $J = 8.1$ Hz, 1H), 7.10 (d, $J = 7.9$ Hz, 2H), 6.48 (s, 2H), 3.95 (s, 6H), 3.87 (s, 3H), 2.28 (s, 3H).

$^{13}$C NMR (151 MHz, DMSO-$_d$6): $\delta$ 166.4, 159.4, 145.6, 137.7, 136.0, 134.5, 133.4, 133.0, 128.1, 125.5, 123.0, 116.7, 92.2, 87.2, 67.4, 56.2, 20.8.

HRMS (ESI) m/z [M - OTs]$^+$: Calcd. for C$_{15}$H$_{13}$BrI0$_3$: 448.9244, found: 448.9244.

This compound has previously been reported.$^6$
2i was prepared according to general procedure A on a 2 mmol scale and obtained as a colorless amorphous solid (1.02 g, 82%).

$^1$H NMR (600 MHz, DMSO-\textit{d}_6): $\delta$ 8.23 (t, $J = 1.8$ Hz, 1H), 7.90 – 7.87 (m, 1H), 7.87 – 7.84 (m, 1H), 7.66 – 7.62 (m, 2H), 7.55 (t, $J = 7.9$ Hz, 1H), 7.51 (t, $J = 7.6$ Hz, 2H), 7.49 (d, $J = 8.1$ Hz, 2H), 7.46 – 7.40 (m, 1H), 7.09 (d, $J = 7.9$ Hz, 2H), 6.48 (s, 2H), 6.48 (s, 2H), 3.96 (s, 6H), 3.87 (s, 3H), 2.27 (s, 3H).

$^{13}$C NMR (151 MHz, DMSO-\textit{d}_6): $\delta$ 166.2, 159.4, 159.4 (d, $J_{C-F} = 249.9$ Hz), 145.6, 137.7, 137.2, 134.9 (d, $J_{C-F} = 8.1$ Hz), 128.1, 127.2 (d, $J_{C-F} = 3.1$ Hz), 125.5, 116.6 (d, $J_{C-F} = 22.2$ Hz), 103.6 (d, $J_{C-F} = 24.1$ Hz), 92.0, 87.5, 57.3, 56.2, 20.8.

HRMS (ESI) m/z [M - OTs]$^+$: Calcd. for C$_{21}$H$_{20}$IO$_3$: 447.0452, found: 447.0452.

This compound has previously been reported.$^7$

$\textbf{(2-Fluorophenyl)(2,4,6-trimethoxyphenyl)iodonium tosylate (2j) [NEW]}$

2j was prepared according to general procedure A on a 5 mmol scale and obtained as a colorless amorphous solid (2.45 g, 88%).

$^1$H NMR (600 MHz, DMSO-\textit{d}_6): $\delta$ 8.12 – 8.07 (m, 1H), 7.68 – 7.62 (m, 1H), 7.50 (dd, $J = 8.5$, 1.3 Hz, 1H), 7.47 (d, $J = 8.1$ Hz, 2H), 7.28 (td, $J = 7.7$, 1.4 Hz, 1H), 7.10 (d, $J = 7.9$ Hz, 2H), 6.44 (s, 2H), 6.44 (s, 2H), 3.94 (s, 6H), 3.85 (s, 3H), 3.85 (s, 3H), 2.28 (s, 3H).

$^{13}$C NMR (151 MHz, DMSO-\textit{d}_6): $\delta$ 166.2, 159.4, 159.4 (d, $J_{C-F} = 249.9$ Hz), 145.6, 137.7, 137.2, 134.9 (d, $J_{C-F} = 8.1$ Hz), 128.1, 127.2 (d, $J_{C-F} = 3.1$ Hz), 125.5, 116.6 (d, $J_{C-F} = 22.2$ Hz), 103.6 (d, $J_{C-F} = 24.1$ Hz), 92.0, 87.5, 57.3, 56.2, 20.8.

$^{19}$F NMR (376 MHz, DMSO-\textit{d}_6): $\delta$ -97.8.

FTIR (neat, $V_{\text{max}}$, cm$^{-1}$): 3090, 3074, 3041, 1583, 1345, 1234, 561

HRMS (ESI) m/z [M - OTs]$^+$: Calcd. for C$_{15}$H$_{15}$FIO$_3$: 389.0044, found: 389.0045.
2-Tolyl(2,4,6-trimethoxyphenyl)iodonium tosylate (2k) [CAS: 2293160-58-2]

2k was prepared according to general procedure A on a 5 mmol scale and obtained as a colorless amorphous solid (2.27 g, 82%).

$^1$H NMR (600 MHz, DMSO-$d_6$): $\delta$ 8.05 (dd, $J = 8.0, 1.1$ Hz, 1H), 7.54 – 7.49 (m, 2H), 7.47 (d, $J = 8.1$ Hz, 1H), 7.27 – 7.18 (m, 1H), 7.14 – 7.04 (m, 2H), 6.44 (s, 2H), 3.95 (s, 6H), 3.85 (s, 3H), 2.60 (s, 3H), 2.28 (s, 3H).

$^{13}$C NMR (151 MHz, DMSO-$d_6$): $\delta$ 166.0, 159.3, 145.8, 140.4, 137.5, 137.1, 132.3, 131.1, 128.8, 128.0, 125.5, 121.3, 92.0, 86.6, 57.2, 56.1, 24.8, 20.7.

HRMS (ESI) m/z [M - OTs]$^+$: Calcd. for C$_{16}$H$_{18}$IO$_3$: 385.0295, found: 385.0296.

The spectroscopic data is in accordance with the literature.

(3,4-Difluorophenyl)(2,4,6-trimethoxyphenyl)iodonium tosylate (2l) [NEW]

2l was prepared according to general procedure A on a 5 mmol scale and obtained as a colorless amorphous solid (2.55 g, 88%).

$^1$H NMR (600 MHz, DMSO-$d_6$): $\delta$ 8.11 (ddd, $J = 9.4, 7.3, 2.2$ Hz, 1H), 7.77 – 7.72 (m, 1H), 7.56 (dt, $J = 10.6, 8.5$ Hz, 1H), 7.47 (d, $J = 8.1$ Hz, 2H), 7.10 (d, $J = 7.9$ Hz, 2H), 6.47 (s, 2H), 3.95 (s, 6H), 3.87 (s, 3H), 2.28 (s, 3H).

$^{13}$C NMR (151 MHz, DMSO-$d_6$): $\delta$ 166.4, 159.4, 151.5 (dd, $J_{CF} = 282.4, 12.5$ Hz), 149.8 (dd, $J_{CF} = 284.3, 12.4$ Hz), 145.6, 137.7, 132.1 (dd, $J_{CF} = 7.3, 3.9$ Hz), 128.1, 125.5, 124.0 (d, $J_{CF} = 19.7$ Hz), 120.6 (d, $J_{CF} = 18.4$ Hz), 109.4 (dd, $J_{CF} = 4.8, 1.0$ Hz), 92.1, 87.6, 57.4, 56.2, 20.8.

$^{19}$F NMR (376 MHz, DMSO-$d_6$): $\delta$ -132.6 (d, $J = 21.6$ Hz), -133.2 (d, $J = 21.7$ Hz).

FTIR (neat, $\nu_{max}$, cm$^{-1}$): 3098, 3057, 3034, 1583, 1348, 1227, 559

HRMS (ESI) m/z [M - OTs]$^+$: Calcd. for C$_{15}$H$_{14}$F$_2$IO$_3$: 406.9950, found: 406.9950.
(3-Fluoro-5-methylphenyl)(2,4,6-trimethoxyphenyl)iodonium tosylate (2m) [NEW]

2m was prepared according to general procedure A on a 5 mmol scale and obtained as a colorless amorphous solid (2.07 g, 93%).

$^1$H NMR (600 MHz, DMSO-$d_6$): $\delta$ 7.62 (d, $J = 7.5$ Hz, 1H), 7.58 (s, 1H), 7.47 (d, $J = 7.7$ Hz, 2H), 7.30 (d, $J = 9.7$ Hz, 1H), 7.10 (d, $J = 7.7$ Hz, 2H), 6.47 (s, 2H), 3.95 (s, 6H), 3.87 (s, 3H), 2.32 (s, 3H), 2.28 (s, 3H).

$^{13}$C NMR (151 MHz, DMSO-$d_6$): $\delta$ 166.3, 161.59 (d, $J_{C-F} = 251.4$ Hz) 159.4, 145.8, 143.9 (d, $J_{C-F} = 7.6$ Hz), 137.5, 130.8 (d, $J_{C-F} = 2.8$ Hz), 127.9, 125.5, 119.4 (d, $J_{C-F} = 20.9$ Hz), 118.4 (d, $J_{C-F} = 24.7$ Hz), 115.2 (d, $J_{C-F} = 8.1$ Hz), 92.1, 87.0, 57.3, 56.2, 20.7, 20.6 (d, $J_{C-F} = 0.9$ Hz).

$^{19}$F NMR (376 MHz, DMSO-$d_6$): $\delta$ -109.2.

FTIR (neat, $\nu_{max}$ cm$^{-1}$): 3076, 3064, 3030, 1578, 1342, 1220, 556

HRMS (ESI) m/z [M - OTs]$^+$: Calcd. for C$_{16}$H$_{17}$FIO$_3$: 403.0201, found: 403.0201.

(3-Chloro-4-methylphenyl)(2,4,6-trimethoxyphenyl)iodonium tosylate (2n) [CAS: 1868173-27-6]

2n was prepared according to general procedure A on a 1.2 mmol scale and obtained as a colorless amorphous solid (541.9 mg, 75%).

$^1$H NMR (600 MHz, DMSO-$d_6$): $\delta$ 7.97 (d, $J = 1.9$ Hz, 1H), 7.73 (dd, $J = 8.2$, 1.9 Hz, 1H), 7.46 (d, $J = 8.1$ Hz, 2H), 7.43 (dd, $J = 8.2$, 0.8 Hz, 1H), 7.10 (d, $J = 7.8$ Hz, 2H), 6.47 (s, 2H), 3.95 (s, 6H), 3.87 (s, 3H), 2.33 (s, 3H), 2.28 (s, 3H).

$^{13}$C NMR (151 MHz, DMSO-$d_6$): $\delta$ 166.3, 159.3, 145.7, 139.9, 137.6, 134.9, 133.8, 133.7, 132.6, 128.0, 125.5, 112.8, 92.1, 87.3, 57.3, 56.2, 20.8, 19.5.

HRMS (ESI) m/z [M - OTs]$^+$: Calcd. for C$_{16}$H$_{17}$ClIO$_3$: 418.9905, found: 418.9906.

The spectroscopic data is in accordance with the literature.\(^5\)
(1-Naphthyl)(2,4,6-trimethoxyphenyl)iodonium tosylate (2o) [CAS: 2366159-97-7]

2o was prepared according to general procedure B on a 2.5 mmol scale and obtained as a pale yellow amorphous solid (900.4 mg, 60%).

$^1$H NMR (600 MHz, DMSO-$d_6$): $\delta$ 8.48 (d, $J = 7.4$ Hz, 1H), 8.22 (t, $J = 7.1$ Hz, 2H), 8.02 (d, $J = 8.2$ Hz, 1H), 7.84 (t, $J = 7.7$ Hz, 1H), 7.69 (t, $J = 7.5$ Hz, 1H), 7.56 (t, $J = 7.8$ Hz, 1H), 7.47 (d, $J = 7.7$ Hz, 2H), 7.09 (d, $J = 7.7$ Hz, 2H), 6.39 (s, 2H), 3.95 (s, 6H), 3.80 (s, 3H), 2.27 (s, 3H).

$^{13}$C NMR (151 MHz, DMSO-$d_6$): $\delta$ 165.9, 159.4, 145.8, 137.6, 137.5, 134.0, 133.0, 131.0, 129.2 (two overlapping signals), 128.0, 127.8, 127.4, 125.5, 119.3, 92.0, 87.1, 57.2, 56.1, 20.8.

HRMS (ESI) m/z [M - OTs]$^+$: Calcd. for C19H18IO3$^+$: 421.0295, found: 421.0295.

This compound has previously been reported.9

(7-Bromo-2-fluorenyl)(2,4,6-trimethoxyphenyl)iodonium tosylate (2p) [NEW]

2p was prepared according to general procedure B on a 2.5 mmol scale and obtained as a beige amorphous solid (1.31 g, 74%).

$^1$H NMR (600 MHz, DMSO-$d_6$): $\delta$ 8.16 (d, $J = 1.6$ Hz, 1H), 8.00 (d, $J = 8.2$ Hz, 1H), 7.96 (dd, $J = 8.2, 1.6$ Hz, 1H), 7.91 (d, $J = 8.2$ Hz, 1H), 7.82 (d, $J = 1.8$ Hz, 1H), 7.60 (dd, $J = 8.2, 1.9$ Hz, 1H), 7.47 (d, $J = 8.1$ Hz, 2H), 7.09 (d, $J = 7.8$ Hz, 2H), 6.47 (s, 2H), 4.00 (s, 2H), 3.97 (s, 6H), 3.86 (s, 3H), 2.27 (s, 3H).

$^{13}$C NMR (151 MHz, DMSO-$d_6$): $\delta$ 166.1, 159.4, 146.0, 145.8, 145.8, 143.1, 138.6, 137.5, 133.5, 131.4, 130.0, 128.3, 128.0, 125.5, 122.9, 122.8, 121.5, 113.8, 92.1, 87.2, 57.3, 56.2, 36.6, 20.8.

FTIR (neat, $v_{max}$ cm$^{-1}$): 3092, 3069, 3055, 3008, 1583, 1346, 1212, 680, 565

HRMS (ESI) m/z [M - OTs]$^+$: Calcd. for C22H19BrIO3$^+$: 536.9557, found: 536.9556.
(6-Chloro-3-pyridinyl)(2,4,6-trimethoxyphenyl)iodonium tosylate (2q) [CAS: 1868173-37-8]

2q was prepared according to general procedure A on a 5 mmol scale and obtained as a colorless amorphous solid (1.95 g, 79%).

$^1$H NMR (600 MHz, DMSO-d$_6$): $\delta$ 8.86 (dd, $J = 2.5$, 0.7 Hz, 1H), 8.36 (dd, $J = 8.5$, 2.4 Hz, 1H), 7.63 (dd, $J = 8.5$, 0.7 Hz, 1H), 7.46 (d, $J = 8.1$ Hz, 2H), 7.10 (d, $J = 7.8$ Hz, 2H), 6.46 (s, 2H), 3.95 (s, 6H), 3.87 (s, 3H), 2.28 (s, 3H).

$^{13}$C NMR (151 MHz, DMSO-d$_6$): $\delta$ 166.4, 159.3, 153.6, 152.7, 145.6, 145.1, 137.6, 128.0, 127.5, 125.5, 113.8, 92.1, 87.2, 57.4, 56.2, 20.8.

HRMS (ESI) m/z [M - OTs]$^+$: Calcd. for C$_{14}$H$_{14}$ClINO$_3$: 405.9701, found: 405.9701.

The spectroscopic data is in accordance with the literature.$^5$

(4-Cyano-3-(trifluoromethyl)phenyl(2,4,6-trimethoxyphenyl)iodonium tosylate (2r) [NEW]

2r was prepared according to general procedure A on a 5 mmol scale and obtained as an off-colorless amorphous solid (1.70 g, 82%).

$^1$H NMR (600 MHz, DMSO-d$_6$): $\delta$ 8.53 (s, 1H), 8.24 (d, $J = 7.8$ Hz, 1H), 8.18 (d, $J = 8.3$ Hz, 1H), 7.45 (d, $J = 7.7$ Hz, 2H), 7.09 (d, $J = 7.7$ Hz, 2H), 6.50 (s, 2H), 3.94 (s, 6H), 3.89 (s, 3H), 2.28 (s, 3H).

$^{13}$C NMR (151 MHz, DMSO-d$_6$): $\delta$ 166.8, 159.5, 145.6, 138.3, 137.6, 132.1 (q, $J_{CF} = 32.5$ Hz), 131.9 (q, $J_{CF} = 4.9$ Hz), 128.0, 125.4, 123.3 (q, $J_{CF} = 274.5$ Hz), 121.0, 114.5, 111.3 (q, $J_{CF} = 2.2$ Hz), 92.3, 86.9, 57.4, 56.3, 20.7.

$^{19}$F NMR (376 MHz, DMSO-d$_6$): $\delta$ -60.9.

FTIR (neat, $V_{max}$ cm$^{-1}$): 3098, 3096, 3066, 2236, 1586, 1346, 1230, 563

HRMS (ESI) m/z [M - OTs]$^+$: Calcd. for C$_{17}$H$_{14}$F$_{3}$INO$_3$: 463.9965, found: 463.9965.
(2,3-dihydrobenzofuran-5-yl)(2,4,6-trimethoxyphenyl)iodonium tosylate (2s) [NEW]

2s was prepared according to general procedure B on a 1.2 mmol scale and obtained as a brown-grey amorphous solid (434.9 mg, 62%).

$^1$H NMR (600 MHz, DMSO-$d_6$): $\delta$ 7.80 – 7.75 (m, 1H), 7.67 (dd, $J = 8.6$, 2.0 Hz, 1H), 7.47 (d, $J = 8.1$ Hz, 2H), 7.10 (d, $J = 8.0$ Hz, 2H), 6.83 (d, $J = 8.5$ Hz, 1H), 6.45 (s, 2H), 4.57 (t, $J = 8.8$ Hz, 2H), 3.95 (s, 6H), 3.86 (s, 3H), 3.20 (t, $J = 8.8$ Hz, 2H), 2.28 (s, 3H).

$^{13}$C NMR (151 MHz, DMSO-$d_6$): $\delta$ 166.0, 162.3, 159.3, 145.7, 137.6, 135.6, 131.8, 131.7, 128.0, 125.5, 112.0, 104.2, 92.0, 87.7, 72.1, 57.3, 56.1, 28.7, 20.8.

FTIR (neat, $V_{\text{max}}$ cm$^{-1}$): 3099, 3074, 3021, 1579, 1343, 1117, 564

HRMS (ESI) m/z [M - OTs]$^+$: Calcd. for C$_{17}$H$_{18}$IO$_4$: 413.0245, found: 413.0244.
Synthesis of N-3-arylhydantoins and N-aryl imides

General procedure

Hydantoin or imide (0.2 mmol, 1.0 eq), aryl(TMP)iodonium tosylate (0.6 mmol, 3.0 eq) and Cu(NO$_3$)$_2$·2.5 H$_2$O (4.7 mg, 0.02 mmol, 0.1 eq) were added to a 7 mL screw cap vial equipped with a magnetic stir bar. Toluene (2 mL) was added and the vial was placed in a pre-heated vial insert heating block set to 70 °C. Triethylamine (42 µL, 0.3 mmol, 1.5 eq) was added via a syringe, and the suspension was stirred for 24 hours at 70 °C. The resulting mixture (typically colored orange, brown or black) was allowed to cool to room temperature before it was concentrated under reduced pressure.

The crude mixture was purified by column chromatography using silica gel and eluent system as specified. In certain cases, co-elution of a strongly colored impurity during column chromatography was observed. This impurity was removed by trituration in a small amount of either toluene or methanol as specified. The desired product was dried under high vacuum for several hours.

Procedure using 1 mmol salt and 5 mol % catalyst (Scheme 3, compound 3a, footnote b)

Hydantoin (0.33 mmol, 1.0 eq), aryl(TMP)iodonium tosylate (1.0 mmol, 3.0 eq) and Cu(NO$_3$)$_2$·2.5 H$_2$O (3.9 mg, 0.017 mmol, 0.05 eq) were added to a 7 mL screw cap vial equipped with a magnetic stir bar. Toluene (3.3 mL) was added and the vial was placed in a pre-heated vial insert heating block set to 70 °C. Triethylamine (70 µL, 0.5 mmol, 1.5 eq) was added via a syringe, and the suspension was stirred for 31 hours at 70 °C.

Figure SI-5: Left: reaction vials after 1 min. Middle: reaction vials after 24h. Right: TLC (stained with KMNO$_4$) of the left reaction (LNB261, m-tolyl)
3-Phenylhydantoin (3a) [CAS: 2221-13-8]

Deviation from standard procedure: 5 mol% Cu(NO$_3$)$_2$·2.5 H$_2$O was used. 3a was obtained after column chromatography [hexane:acetone (90:10 % → 70:30 %)] as a colorless, amorphous solid (28.4 mg, 79%).

Using the procedure with 1 mmol salt: 3a was obtained after column chromatography [hexane:acetone (90:10 % → 70:30 %)] as a colorless, amorphous solid (45.5 mg, 78%).

$^1$H NMR (600 MHz, DMSO-$d_6$): δ 8.30 (s, 1H), 7.50 – 7.44 (m, 2H), 7.40 – 7.37 (m, 1H), 7.36 – 7.34 (m, 2H), 4.07 (d, $J = 1.3$ Hz, 2H).

$^{13}$C NMR (151 MHz, DMSO-$d_6$): δ 171.1, 156.5, 132.3, 128.7, 127.7, 126.7, 46.0.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for C$_9$H$_8$N$_2$O$_2$Na: 199.0478, found: 199.0477.

This compound has previously been reported.$^{10}$

3-(4-methylphenyl)hydantoin (3b) [CAS: 65119-75-7]

3b was obtained after column chromatography [hexane:acetone (90:10% → 75:25%)] as a colorless, amorphous solid (31.5 mg, 82%).

$^1$H NMR (600 MHz, CDCl$_3$): δ 7.29 – 7.25 (m, 2H), 7.25 – 7.21 (m, 2H), 6.91 (br s, 1H), 4.05 (d, $J = 1.1$ Hz, 2H), 2.37 (s, 3H).

$^{13}$C NMR (151 MHz, CDCl$_3$): δ 170.3, 157.9, 138.6, 129.8, 128.6, 126.1, 46.5, 21.1.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for C$_{10}$H$_{10}$N$_2$NaO$_2$$: 213.0634, found: 213.0635.

This compound has previously been reported.$^{11}$
3-(4-Nitrophenyl)hydantoin (3c) [CAS: 62101-57-9]

3c was obtained after column chromatography [hexane:acetone (80:20% → 70:30%)] as a pale yellow, amorphous solid (26.4 mg, 59%).

\[ ^1H \text{ NMR (600 MHz, DMSO-}d_6\text{): } \delta \ 8.52 \text{ (br s, 1H), 8.34 (d, } J = 9.1 \text{ Hz, 2H), 7.74 (d, } J = 9.1 \text{ Hz, 2H), 4.10 (d, } J = 1.1 \text{ Hz, 2H).} \]

\[ ^{13}C \text{ NMR (151 MHz, DMSO-}d_6\text{): } \delta \ 170.7, 155.6, 145.7, 138.2, 126.7, 124.1, 46.1. \]

HRMS (ESI) m/z [M + Na]^+: Calcd. for C_{9}H_{7}N_{3}O_{4}Na: 244.0329, found: 244.0329.

The spectroscopic data is in accordance with the literature.\(^{12}\)

3-(4-Methoxycarbonylphenyl)hydantoin (3d) [CAS: 2107864-97-9]

3d was obtained after column chromatography [hexane:acetone (90:10% → 70:30%)] as a colorless, amorphous solid (27.2 mg, 58%).

\[ ^1H \text{ NMR (600 MHz, DMSO-}d_6\text{): } \delta \ 8.43 \text{ (br s, 1H), 8.05 (d, } J = 8.6 \text{ Hz, 2H), 7.57 (d, } J = 8.6 \text{ Hz, 2H), 4.08 (d, } J = 1.1 \text{ Hz, 2H), 3.87 (s, 3H).} \]

\[ ^{13}C \text{ NMR (151 MHz, DMSO-}d_6\text{): } \delta \ 170.8, 165.7, 155.9, 136.6, 129.6, 128.3, 126.2, 52.3, 46.0. \]

HRMS (ESI) m/z [M + Na]^+: Calcd. for C_{11}H_{10}N_{2}O_{4}: 257.0533, found: 257.0532.

This compound has been reported in the literature, but without spectroscopic data.
3-(4-(Pentafluorosulfanyl)phenyl)hydantoin (3e) [NEW]

3e was obtained after column chromatography [hexane:chloroform:acetone (47.5:47.5:5 % → 42.5:42.5:15 %)] as a colorless, crystalline solid (30.1 mg, 50 %).

$^1$H NMR (600 MHz, CDCl$_3$): δ 7.85 (d, $J = 9.1$ Hz, 2H), 7.60 (d, $J = 8.7$ Hz, 2H), 6.62 (br s, 1H), 4.15 (d, $J = 1.1$ Hz, 2H).

$^{13}$C NMR (151 MHz, CDCl$_3$): δ 169.4, 156.6, 152.7 (p, $J_{C-F} = 18.2$ Hz), 134.1, 127.0, 125.7, 46.3.

$^{19}$F NMR (376 MHz, CDCl$_3$): δ 83.5 (p, $J = 150.3$ Hz), 62.9 (d, $J = 150.4$ Hz).

FTIR (neat, $v_{\text{max}}$ cm$^{-1}$): 3243, 3115, 1782, 1718, 1506

HRMS (ESI) m/z [M + Na$^+$]: Calcd. for C$_{27}$H$_{25}$F$_5$N$_2$NaO$_2$S$: 325.0041, found: 325.0041.

Melting point: 183 – 185°C

3-(4-((Phthalimidyl)benzyl)phenyl)hydantoin (3f) [NEW]

3f was obtained after column chromatography [hexane:chloroform:acetone (47.5:47.5:5 % → 42.5:42.5:15 %)] as an off-colorless, amorphous solid (43.9 mg, 65 %).

$^1$H NMR (600 MHz, DMSO-d$_6$): δ 8.29 (br s, 1H), 7.94 – 7.87 (m, 2H), 7.89 – 7.82 (m, 2H), 7.41 (d, $J = 8.0$ Hz, 2H), 7.30 (d, $J = 8.0$ Hz, 2H), 7.41 (d, $J = 8.0$ Hz, 2H), 7.30 (d, $J = 8.0$ Hz, 2H), 7.41 (d, $J = 8.0$ Hz, 2H), 7.30 (d, $J = 8.0$ Hz, 2H), 7.41 (s, 2H), 4.81 (s, 2H), 4.05 (s, 2H).

$^{13}$C NMR (151 MHz, DMSO-d$_6$): δ 171.0, 167.7, 156.4, 156.2, 134.6, 131.6, 131.4, 127.7, 126.9, 123.3, 46.0, 40.5.

FTIR (neat, $v_{\text{max}}$ cm$^{-1}$): 3234, 3147, 3109, 1778, 1767, 1708, 1520

HRMS (ESI) m/z [M + Na$^+$]: Calcd. for C$_{18}$H$_{13}$N$_3$NaO$_4$: 358.0798, found: 358.0798.
3-(3-methylphenyl)hydantoin (3g) [CAS: 71532-35-9]

3g was obtained after column chromatography [hexane:acetone (90:10% → 75:25%)] as a colorless, amorphous solid (26.6 mg, 70%).

\[ \text{H NMR (600 MHz, CDCl}_3): \delta 7.35 (t, J = 7.7 \text{ Hz}, 1\text{H}), 7.19 (d, J = 7.7 \text{ Hz}, 1\text{H}), 7.17 - 7.12 (m, 2\text{H}), 6.86 (br s, 1\text{H}), 4.06 (d, J = 1.1 \text{ Hz}, 2\text{H}), 2.38 (s, 3\text{H}). \]

\[ \text{C NMR (151 MHz, CDCl}_3): \delta 170.3, 157.9, 139.3, 131.1, 129.4, 129.0, 126.9, 123.4, 46.5, 21.3. \]


This compound has previously been reported.

3-(3-Bromophenyl)hydantoin (3h) [CAS: 1697381-00-2]

3h was obtained after column chromatography [hexane:EtOAc (60:40% → 40:60%)] as a colorless, amorphous solid (25.8 mg, 51%).

\[ \text{H NMR (800 MHz, CDCl}_3): \delta 7.59 (d, J = 1.4 \text{ Hz}, 1\text{H}), 7.50 (dd, J = 7.8, 1.0 \text{ Hz}, 1\text{H}), 7.37 (dd, J = 8.0, 1.0 \text{ Hz}, 1\text{H}), 7.33 (t, J = 7.9 \text{ Hz}, 1\text{H}), 6.70 (br s, 1\text{H}), 4.09 (s, 2\text{H}). \]

\[ \text{C NMR (201 MHz, CDCl}_3): \delta 169.7, 157.0, 132.5, 131.4, 130.3, 129.1, 124.7, 122.4, 46.4. \]


This compound has previously been reported.
3-(3-[1,1′-Biphen-3-yl])hydantoin (3i) [NEW]

![Chemical Structure](image)

3i was obtained after column chromatography [hexane:acetone (90:10% → 70:30%)]. The resulting crude was concentrated under reduced pressure and triturated with toluene. The product was obtained as a colorless, amorphous solid (28.6 mg, 57%).

$^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 7.62 – 7.56 (m, 4H), 7.55 – 7.51 (m, 1H), 7.45 – 7.41 (m, 2H), 7.37 – 7.33 (m, 2H), 6.80 (br s, 1H), 4.09 (d, $J = 1.2$ Hz, 2H).

$^{13}$C NMR (151 MHz, CDCl$_3$): $\delta$ 170.1, 157.6, 142.5, 140.0, 131.7, 129.5, 128.8, 127.7, 127.2, 125.1, 124.9, 46.5.

FTIR (neat, $V_{\text{max}}$ cm$^{-1}$): 3244, 3121, 1778, 1716, 1507

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for C$_{15}$H$_{12}$N$_2$NaO$_2$: 275.0791, found: 275.0790.

3-(2-Fluorophenyl)hydantoin (3j) [CAS: 1153758-51-0]

$\text{Chemical Structure}$

3j was obtained after column chromatography [hexane:acetone (90:10% → 70:30%)]. The resulting crude was concentrated under reduced pressure and triturated with toluene. The product was obtained as a colorless, amorphous solid (16.0 mg, 41%).

$^1$H NMR (800 MHz, DMSO-$d_6$): $\delta$ 8.38 (br s, 1H), 7.53 – 7.48 (m, 1H), 7.40 (q, $J = 9.9$, 8.5 Hz, 2H), 7.32 (t, $J = 7.7$ Hz, 1H), 4.15 (br s, 2H).

$^{13}$C NMR (201 MHz, DMSO-$d_6$): $\delta$ 170.61, 157.4 (d, $J_{C-F} = 250.3$ Hz), 155.7, 130.8 (d, $J_{C-F} = 7.8$ Hz), 130.5, 124.8 (d, $J_{C-F} = 3.7$ Hz), 119.7 (d, $J_{C-F} = 12.9$ Hz), 116.2 (d, $J_{C-F} = 19.3$ Hz), 46.3.

$^{19}$F NMR (376 MHz, DMSO-$d_6$): $\delta$ -120.6.


This compound has been reported in the literature, but no spectroscopic data was divulged.
3-(2-Fluorophenyl)-5,5-diphenylhydantoin (3k) [CAS: 1975145-65-3]

3k was obtained after column chromatography [hexane:EtOAc (95:15% → 70:30%)] as a colorless amorphous solid (34.0 mg, 49%).

$^1$H NMR (800 MHz, CDCl$_3$): δ 7.49 – 7.30 (m, 12H, overlap with NH peak at 7.43), 7.28 (t, $J = 7.5$ Hz, 1H), 7.23 – 7.17 (m, 2H).

$^{13}$C NMR (201 MHz, CDCl$_3$): δ 171.8, 157.9 (d, $J_{C,F} = 253.3$ Hz), 154.9, 138.9 (br), 131.0 (d, $J_{C,F} = 7.8$ Hz), 129.8, 128.9, 128.7, 126.9, 124.6 (d, $J_{C,F} = 3.7$ Hz), 119.1 (d, $J_{C,F} = 13.1$ Hz), 116.7 (d, $J_{C,F} = 19.6$ Hz), 70.7.

$^{19}$F NMR (376 MHz, CDCl$_3$): δ -118.9.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for C$_{21}$H$_{15}$FN$_2$NaO$_2$$^+$: 369.1010, found: 369.1010.

This compound has previously been reported.$^{15}$

1-Methyl-3-phenylhydantoin (3l) [CAS: 2221-12-7]

3l was obtained after column chromatography [hexane:acetone (80:20)]. The resulting crude was concentrated under reduced pressure and washed with toluene. The product was obtained as a brown amorphous solid (31.3 mg, 82%).

$^1$H NMR (600 MHz, CDCl$_3$): δ 7.46 – 7.41 (m, 2H), 7.39 – 7.32 (m, 3H), 4.00 (s, 2H), 3.05 (s, 3H).

$^{13}$C NMR (151 MHz, CDCl$_3$): δ 168.6, 155.7, 131.8, 129.0, 128.1, 126.0, 51.6, 29.8.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for C$_{10}$H$_{10}$N$_2$NaO$_2$$^+$: 213.0634, found: 213.0634.

The spectroscopic data is in accordance with the literature.$^{16}$

531
5-Methyl-3-phenylhydantoin (rac-3m) [CAS: 33558-00-8]

*rac*-3m was obtained after column chromatography [hexane:acetone (90:10% → 70:30%)] as a colorless, amorphous solid (28.8 mg, 76%).

$^1$H NMR (600 MHz, CDCl$_3$): δ 7.49 – 7.43 (m, 2H), 7.40 – 7.33 (m, 3H), 6.63 (br s, 1H), 4.20 (qd, $J = 6.9, 1.3$ Hz, 1H), 1.51 (d, $J = 7.0$ Hz, 3H).

$^{13}$C NMR (151 MHz, CDCl$_3$): δ 173.5, 156.6, 131.4, 129.1, 128.3, 126.1, 52.8, 17.8.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for C$_{10}$H$_{10}$N$_2$NaO$_2$: 213.0634, found: 213.0634.

The spectroscopic data is in accordance with the literature.

(R)-5-Methyl-3-phenylhydantoin ((R)-3m) [CAS: 113890-05-4]

*(R)-3m* was obtained after column chromatography [hexane:EtOAc (70:30% → 40:60%)] as a colorless, crystalline solid (28.7 mg, 75%).

$^1$H NMR (600 MHz, Chloroform-$d$): δ 7.48 – 7.43 (m, 2H), 7.40 – 7.34 (m, 3H), 6.93 (br s, 1H), 4.18 (qd, $J = 7.0$, 1.3 Hz, 1H), 1.49 (d, $J = 7.0$ Hz, 3H).

$^{13}$C NMR (151 MHz, CDCl$_3$): δ 173.6, 156.8, 131.4, 129.1, 128.3, 126.1, 52.8, 17.7.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for C$_{10}$H$_{10}$N$_2$NaO$_2$: 213.0634, found: 213.0634.

$\left[\alpha\right]_{D}^{20} = +31.6$ (c = 0.43, CHCl$_3$)

Melting point: 150 – 152 °C

This compound has previously been reported.
5,5-Dimethyl-3-phenylhydantoin (3n) [CAS: 70974-10-06]

3n was obtained after column chromatography [hexane:acetone (90:10% → 70:30%)] as a colorless, amorphous solid (29.3 mg, 72%).

$^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 7.48 – 7.42 (m, 2H), 7.42 – 7.38 (m, 2H), 7.36 (td, $J = 7.2$, 1.3 Hz, 1H), 6.15 (br s, 1H), 1.52 (s, 6H).

$^{13}$C NMR (151 MHz, CDCl$_3$): $\delta$ 176.1, 155.4, 131.6, 129.1, 128.2, 126.1, 58.6, 25.3.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for C$_{11}$H$_{12}$N$_2$NaO$_2$: 227.0791, found: 227.0791.

The spectroscopic data is in accordance with the literature.$^{17}$

(S)-2-phenyltetrahydro-1H-pyrrolo[1,2-c]imidazole-1,3(2H)-dione ([S]-3o) [CAS: 70741-88-7]

(S)-3o was obtained after column chromatography [hexane:EtOAc (80:20% → 70:30%)] as a colorless, crystalline solid (33.7 mg, 78%).

$^1$H NMR (600 MHz CDCl$_3$): $\delta$ 7.45 – 7.40 (m, 2H), 7.39 – 7.35 (m, 2H), 7.35 – 7.31 (m, 1H), 4.21 (dd, $J = 9.3$, 7.4 Hz, 1H), 3.77 (dt, $J = 11.3$, 7.7 Hz, 1H), 3.32 (ddd, $J = 11.3$, 8.4, 4.5 Hz, 1H), 2.32 (ddt, $J = 12.7$, 7.2, 3.6 Hz, 1H), 2.20 – 2.12 (m, 1H), 2.12 – 2.04 (m, 1H), 1.87 – 1.79 (m, 1H).

$^{13}$C NMR (151 MHz, CDCl$_3$): $\delta$ 172.5, 159.3, 131.8, 129.0, 128.1, 125.8, 63.2, 45.7, 27.7, 26.8.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for C$_{12}$H$_{12}$N$_2$NaO$_2$: 239.0791, found: 239.0790.

$[\alpha]_{D}^{20}$ = -38.8 (c = 0.51, CHCl$_3$)

Melting point: 141 – 143°C

The spectroscopic data is in accordance with the literature.$^{19}$
3,5,5-Triphenylhydantoin (3p) [CAS: 52461-02-6]

3p was obtained after column chromatography [hexane:acetone (100:0% → 90:10%)] as a colorless, crystalline solid (53.0 mg, 80%).

$^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 7.51 (br s, 1H), 7.46 – 7.41 (m, 6H), 7.41 – 7.33 (m, 9H).

$^{13}$C NMR (151 MHz, CDCl$_3$): $\delta$ 172.2, 155.7, 139.0, 131.4, 129.0, 128.8, 128.6, 128.3, 126.9, 126.3, 69.9.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for C$_{21}$H$_{16}$N$_2$NaO$_2$: 351.1104, found: 351.1103.

Melting point: 206 – 207°C

The spectroscopic data is in accordance with the literature.$^{17}$

5,5-Diphenyl-3-(3-methylphenyl)hydantoin (3r) [NEW]

3r was obtained after column chromatography [hexane:acetone (90:10%)] as a colorless, crystalline solid (56.1 mg, 82%).

$^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 7.46 – 7.41 (m, 5H), 7.38 – 7.33 (m, 6H), 7.32 (t, $J$ = 7.9 Hz, 1H), 7.18 – 7.17 (m, 2H), 7.16 (br s, 1H), 2.36 (s, 3H).

$^{13}$C NMR (151 MHz, CDCl$_3$): $\delta$ 172.2, 155.8, 139.1, 131.2, 129.3, 128.9, 128.8, 128.6, 126.9, 126.9, 123.5, 69.9, 21.3.

FTIR (neat, $\nu_{\text{max}}$ cm$^{-1}$): 3205, 3097, 1775, 1713, 1490, 1449.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for C$_{22}$H$_{18}$N$_2$NaO$_2$: 365.1260, found: 365.1260.

Melting point: 228 – 230°C
5,5-Diphenyl-3-(4-methylphenyl)hydantoin (3s) [CAS: 137606-46-3]

![Chemical Structure](image)

3s was obtained after column chromatography [hexane:acetone (90:10%)] as colorless, crystalline solid (55.4 mg, 81%).

\(^1\)H NMR (600 MHz, CDCl\textsubscript{3}): \(\delta\) 7.66 (br s, 1H), 7.45 – 7.42 (m, 4H), 7.37 – 7.33 (m, 6H), 7.25 – 7.21 (m, 4H), 2.36 (s, 3H).

\(^{13}\)C NMR (151 MHz, CDCl\textsubscript{3}): \(\delta\) 172.3, 156.0, 139.1, 138.4, 129.7, 128.8, 128.7, 128.5, 126.9, 126.2, 69.9, 21.1.

HRMS (ESI) m/z [M + Na]^+: Calcd. for C\textsubscript{22}H\textsubscript{18}N\textsubscript{2}NaO\textsubscript{2}: 365.1260, found: 365.1260.

Melting point: 222 – 224°C

The spectroscopic data is in accordance with previously reported data.\(^20\)

3-(3,4-Difluorophenyl)hydantoin (3t) [CAS: 1637409-87-0]

![Chemical Structure](image)

3t was obtained after column chromatography [hexane:chloroform:acetone (47.5:47.5:5% \(\rightarrow\) 42.5:42.5:15%)] as a colorless, amorphous solid (25.9 mg, 61%).

\(^1\)H NMR (600 MHz, DMSO-\textsubscript{d}\textsubscript{6}): \(\delta\) 8.39 (br s, 1H), 7.58 – 7.53 (m, 1H), 7.53 – 7.48 (m, 1H), 7.30 – 7.23 (m, 1H), 4.06 (d, \(J = 1.2\) Hz, 2H).

\(^{13}\)C NMR (151 MHz, DMSO-\textsubscript{d}\textsubscript{6}): \(\delta\) 170.9, 156.0, 148.9 (dd, \(J_{C - F} = 246.0, 13.5\) Hz), 148.6 (dd, \(J_{C - F} = 246.7, 12.4\) Hz), 129.0 (dd, \(J_{C - F} = 8.8, 3.4\) Hz), 123.8 (dd, \(J_{C - F} = 6.8, 3.4\) Hz), 117.5 (d, \(J_{C - F} = 18.2\) Hz), 116.1 (d, \(J_{C - F} = 19.4\) Hz), 46.0.

\(^{19}\)F NMR (376 MHz, DMSO-\textsubscript{d}\textsubscript{6}): \(\delta\) -137.3 (d, \(J = 22.9\) Hz), -139.1 (d, \(J = 23.0\) Hz).

HRMS (ESI) m/z [M + Na]^+: Calcd. for C\textsubscript{9}H\textsubscript{6}F\textsubscript{2}N\textsubscript{2}NaO\textsubscript{2}: 235.0290, found: 235.0289.

This compound has previously been reported in the patent literature.\(^21\)
3-(3-Fluoro-5-methylphenyl)hydantoin (3u) [CAS: 1692273-32-7]

![Chemical structure of 3u]

3u was obtained after column chromatography [hexane:chloroform:acetone (47.5:47.5:5% → 42.5:42.5:15%)] as a colorless, amorphous solid (31.6 mg, 76%).

^{1}H NMR (600 MHz, CDCl₃): δ 7.01 (s, 1H), 6.95 (dt, J = 9.3, 2.2 Hz, 1H), 6.90 (d, J = 9.3 Hz, 1H), 6.88 (br s, 1H), 4.08 (d, J = 1.2 Hz, 2H), 2.37 (s, 3H).

^{13}C NMR (151 MHz, CDCl₃): δ 169.9, 162.5 (d, J_{C-F} = 246.5 Hz), 157.4, 141.2 (d, J_{C-F} = 8.7 Hz), 132.1 (d, J_{C-F} = 11.1 Hz), 122.4 (d, J_{C-F} = 3.0 Hz), 116.2 (d, J_{C-F} = 20.8 Hz), 110.8 (d, J_{C-F} = 24.7 Hz), 46.4, 21.4 (d, J_{C-F} = 2.0 Hz).

^{19}F NMR (376 MHz, CDCl₃): δ -112.1.

HRMS (ESI) m/z [M + Na]⁺: Calcd. for C₁₀H₉FN₂NaO₂⁺: 231.0540, found: 231.0540.

This compound has previously been reported in the literature, but characterization data has not been disclosed.

3-(3-Chloro-4-methylphenyl)hydantoin (3v) [CAS: 56011-97-3]

![Chemical structure of 3v]

3v was obtained after column [hexane:chloroform:acetone (47.5:47.5:5% → 42.5:42.5:15%)] as a colorless, amorphous solid (31.7 mg, 70%).

^{1}H NMR (600 MHz, CDCl₃): δ 7.40 (d, J = 2.2 Hz, 1H), 7.31 (d, J = 8.2 Hz, 1H), 7.19 (dd, J = 8.1, 2.2 Hz, 1H), 6.91 (br s, 1H), 4.07 (d, J = 1.1 Hz, 2H), 2.38 (s, 3H).

^{13}C NMR (151 MHz, CDCl₃): δ 169.9, 157.4, 136.6, 134.6, 131.2, 129.8, 126.7, 124.3, 46.4, 19.8.


This compound has been reported in the patent literature.²²
3-(1-Naphthalenyl)hydantoin (3w) [CAS: 64521-32-0]

3w was obtained after column chromatography [hexane:EtOAc (60:40% → 40:60%)] as a colorless, amorphous solid (23.7 mg, 52%).

$^1$H NMR (600 MHz, DMSO-$d_6$): $\delta$ 8.39 (br s, 1H), 8.07 – 8.02 (m, 2H), 7.77 – 7.73 (m, 1H), 7.64 – 7.55 (m, 3H), 7.47 (dd, $J = 7.2$, 1.2 Hz, 1H), 4.32 (dd, $J = 17.8$, 1.1 Hz, 1H), 4.17 (dd, $J = 17.8$, 1.2 Hz, 1H).

$^{13}$C NMR (151 MHz, DMSO-$d_6$): $\delta$ 171.8, 156.9, 133.7, 130.2, 129.2, 128.9, 128.2, 127.1, 126.9, 126.5, 125.6, 122.8, 46.5.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for C$_{13}$H$_{10}$N$_2$O$_2$Na$: 249.0634, found: 249.0634.

This compound has previously been reported in the patent literature.$^{23}$

3-(7-Bromo-9H-2-fluorenyl)-1-methylhydantoin (3x) [NEW]

3x was obtained after column chromatography [hexane:acetone (90:10% → 75:25%)] as a colorless, amorphous solid (48.4 mg, 70%).

$^1$H NMR (800 MHz, CDCl$_3$): $\delta$ 7.76 (d, $J = 8.1$ Hz, 1H), 7.64 (d, $J = 1.9$ Hz, 1H), 7.59 (d, $J = 8.1$ Hz, 1H), 7.52 (d, $J = 2.0$ Hz, 1H), 7.47 (dd, $J = 8.0$, 1.2 Hz, 1H), 7.37 (dd, $J = 8.2$, 1.9 Hz, 1H), 4.03 (s, 2H), 3.87 (s, 2H), 3.07 (s, 3H).

$^{13}$C NMR (201 MHz, CDCl$_3$): $\delta$ 168.7, 155.8, 145.5, 143.6, 140.6, 139.8, 130.6, 130.0, 128.3, 125.0, 122.9, 121.4, 120.9, 120.2, 51.6, 36.7, 29.9.

FTIR (neat, $\nu_{\text{max}}$ cm$^{-1}$): 3091, 3068, 1767, 1707, 1486, 1458, 628.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for C$_{17}$H$_{13}$BrN$_2$NaO$_2$$: 379.0053, found: 379.0052.
3-(6-Chloro-3-pyridinyl)hydantoin (3y) [NEW]

3y was obtained after column chromatography [hexane:chloroform:acetone (47.5:47.5:5% → 42.5:42.5:15%)]. The resulting crude was concentrated under reduced pressure and washed with toluene. The product was obtained as a colorless amorphous solid (16.1 mg, 38%).

$^1$H NMR (600 MHz, DMSO-d$_6$): δ 8.49 (br s, 1H), 8.47 (d, $J = 2.7$ Hz, 1H), 7.92 (dd, $J = 8.5$, 2.7 Hz, 1H), 7.67 (d, $J = 8.5$ Hz, 1H), 4.09 (d, $J = 1.3$ Hz, 2H).

$^{13}$C NMR (151 MHz, DMSO-d$_6$): δ 170.8, 155.7, 148.4, 147.1, 137.3, 128.7, 124.4, 46.2.

FTIR (neat, $\nu_{\text{max}}$ cm$^{-1}$): 3230, 3101, 1786, 1715, 1462, 569.


This compound has previously been reported in the literature, but without characterization data.

3-(3-Trifluoromethyl-4-cyanophenyl)hydantoin (3z) [CAS: 1961891-89-3]

3z was obtained after column chromatography [hexane:acetone (90:10% → 70:30%)] as a pale yellow, amorphous solid (17.1 mg, 32%).

$^1$H NMR (600 MHz, DMSO-d$_6$): δ 8.61 (br s, 1H), 8.30 (d, $J = 8.4$ Hz, 1H), 8.12 (d, $J = 2.0$ Hz, 1H), 8.00 (dd, $J = 8.4$, 2.0 Hz, 1H), 4.10 (d, $J = 1.0$ Hz, 2H).

$^{13}$C NMR (151 MHz, DMSO-d$_6$): δ 170.6, 155.2, 137.0, 136.2, 131.1 (q, $J_{C-F} = 32.2$ Hz), 129.8, 123.7 (q, $J_{C-F} = 4.9$ Hz), 122.2 (q, $J_{C-F} = 273.5$ Hz), 115.2, 106.5 (q, $J_{C-F} = 2.2$ Hz), 46.1.

$^{19}$F NMR (376 MHz, DMSO-d$_6$): δ -61.0.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for C$_{11}$H$_6$F$_3$N$_3$NaO$_2$: 292.0304, found: 292.0304.

This compound has previously been reported in the literature, but without characterization data.
3-(2,3-Dihydrobenzofuran-5-yl)hydantoin (3aa) [1856678-74-4]

3aa was obtained after column chromatography [hexane:EtOAc (80:20% → 60:40%)]. The resulting crude was concentrated under reduced pressure and washed with MeOH. The product was obtained as a colorless amorphous solid (13.0 mg, 30%).

$^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 7.16 – 7.11 (m, 1H), 7.06 (dd, $J = 8.5$, 2.2 Hz, 1H), 6.84 (d, $J = 8.4$ Hz, 1H), 6.03 (br s, 1H), 4.59 (t, $J = 8.7$ Hz, 2H), 4.10 (d, $J = 1.1$ Hz, 2H), 3.23 (t, $J = 8.7$ Hz, 2H).

$^{13}$C NMR (151 MHz, CDCl$_3$): $\delta$ 170.5, 160.3, 157.7, 128.3, 126.7, 123.4, 109.8, 71.8, 46.4, 29.7.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for C$_{11}$H$_{10}$N$_2$O$_3$: 241.0584, found: 241.0583.

This compound has previously been reported in the literature, but without characteristic data.

N-phenylsuccinimide (5a) [CAS: 83-25-0]

5a was obtained after column chromatography [hexane:acetone (90:10% → 80:20%)] as a colorless, amorphous solid (28.6 mg, 81%).

$^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 7.49 – 7.43 (m, 2H), 7.41 – 7.35 (m, 1H), 7.29 – 7.25 (m, 2H), 2.88 (s, 4H).

$^{13}$C NMR (151 MHz, CDCl$_3$): $\delta$ 176.1, 131.9, 129.2, 128.7, 126.5, 28.4.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for C$_{10}$H$_9$NNaO$_2$: 198.0525, found: 198.0526.

The spectroscopic data is in accordance with the literature.$^{24}$
**N-phenylphthalimide (5b) [CAS: 520-03-6]**

![Structure of N-phenylphthalimide (5b)](image)

5b was obtained after column chromatography [hexane:Et<sub>2</sub>O (90:10% → 85:15%) as a colorless, amorphous solid (39.1 mg, 87%).

\[\text{\(^1^H\) NMR (800 MHz, CDCl}_3\): } \delta 7.93 (dd, \(J = 5.4, 3.1\) Hz, 2H), 7.76 (dd, \(J = 5.5, 3.1\) Hz, 2H), 7.49 (t, \(J = 7.8\) Hz, 2H), 7.43 (d, \(J = 7.0\) Hz, 2H), 7.39 (t, \(J = 7.5\) Hz, 1H).

\[\text{\(^{13}C\) NMR (201 MHz, CDCl}_3\): } \delta 167.2, 134.3, 131.7 (2C), 129.1, 128.0, 126.5, 123.7.

HRMS (ESI) m/z [M + Na]+: Calcd. for C\textsubscript{14}H\textsubscript{9}NNaO\textsubscript{2}+: 246.0525, found: 246.0525.

The spectroscopic data is in accordance with the literature.\(^{25}\)

**N-phenyl-1,8-naphthalimide (5c) [CAS: 6914-98-3]**

![Structure of N-phenyl-1,8-naphthalimide (5c)](image)

5c was obtained after column chromatography [hexane:EtOAc (90:10% → 70:30%) as a colorless amorphous solid (22.6 mg, 40%).

\[\text{\(^1^H\) NMR (800 MHz, CDCl}_3\): } \delta 8.63 (dd, \(J = 7.2, 1.2\) Hz, 2H), 8.25 (dd, \(J = 8.2, 1.2\) Hz, 2H), 7.77 (t, \(J = 7.7\) Hz, 2H), 7.54 (t, \(J = 7.8\) Hz, 2H), 7.47 (t, \(J = 7.6\) Hz, 1H), 7.31 (d, \(J = 7.2\) Hz, 2H).

\[\text{\(^{13}C\) NMR (201 MHz, CDCl}_3\): } \delta 164.3, 135.4, 134.2, 131.7, 131.5, 129.3, 128.6, 128.5, 127.0, 122.8.

HRMS (ESI) m/z [M + Na]+: Calcd. for C\textsubscript{18}H\textsubscript{12}NNaO\textsubscript{2}+: 296.0682, found: 296.0681.

The spectroscopic data is in accordance with the literature.\(^{26}\)
1-(5-Nitrofurfurylideneamino)-3-phenylhydantoin (7a) [CAS: 20167-49-1]

7a was obtained after column chromatography [hexane:chloroform:acetone (47.5:47.5:5% → 42.5:42.5:15%)]. The resulting crude was concentrated under reduced pressure and triturated with methanol (ca. 0.5 mL). The product was obtained as a yellow-orange, amorphous solid (48.9 mg, 78%).

$^1$H NMR (600 MHz, DMSO-$d_6$): $\delta$ 7.97 (s, 1H), 7.81 (d, $J = 3.9$ Hz, 1H), 7.53 (t, $J = 7.8$ Hz, 2H), 7.45 (t, $J = 7.5$ Hz, 1H), 7.42 (d, $J = 7.1$ Hz, 2H), 7.21 (d, $J = 3.9$ Hz, 1H), 4.55 (s, 2H).

$^{13}$C NMR (151 MHz, DMSO-$d_6$): $\delta$ 166.5, 152.1, 151.9, 151.7, 132.1, 131.5, 128.9, 128.4, 127.0, 114.8, 114.7, 48.5.

FTIR (neat, $\nu_{\text{max}}$ cm$^{-1}$): 3133, 3114, 1800, 1734, 1721, 1558, 1508, 1338, 1200.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for C$_{14}$H$_{10}$N$_4$O$_5$: 337.0543, found: 337.0543.

This compound has previously been reported in the literature.$^{27}$

1-(5-Nitrofurfurylideneamino)-3-(4-methoxycarbonyl)phenylhydantoin (7b) [NEW]

7b was obtained after column chromatography [hexane:EtOAc (70:30%)]. The resulting crude was concentrated under reduced pressure and triturated with methanol (ca. 0.5 mL). The product was obtained as a yellow-orange, amorphous solid (48.8 mg, 65%).

$^1$H NMR (600 MHz, DMSO-$d_6$): $\delta$ 8.11 (d, $J = 8.3$ Hz, 2H), 7.99 (s, 1H), 7.81 (d, $J = 3.9$ Hz, 1H), 7.61 (d, $J = 8.2$ Hz, 2H), 7.21 (d, $J = 3.9$ Hz, 1H), 4.56 (s, 2H), 3.89 (s, 3H).

$^{13}$C NMR (201 MHz, DMSO-$d_6$): $\delta$ 166.2, 165.6, 151.9, 151.6, 151.5, 135.6, 132.4, 129.8, 129.1, 126.7, 114.9, 114.6, 52.3, 48.5.

FTIR (neat, $\nu_{\text{max}}$ cm$^{-1}$): 3133, 3114, 1800, 1734, 1721, 1558, 1508, 1338, 1200.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for C$_{16}$H$_{12}$N$_4$O$_7$: 395.0598, found: 395.0598.

1-(5-Nitrofurfurylideneamino)-3-((4-Phthalimidyl)benzyl)hydantoin (7c) [NEW]
was obtained after column chromatography [hexane:chloroform:acetone (47.5:47.5:5% → 40:40:20%)]. The resulting crude was concentrated under reduced pressure and triturated with methanol (ca. 0.5 mL). The product was obtained as a yellow-orange, amorphous solid (71.2 mg, 75%).

\[ 1^H \text{NMR} (600 \text{ MHz, DMSO-d}_6): \delta 7.96 (s, 1H), 7.92 – 7.88 (m, 2H), 7.88 – 7.84 (m, 2H), 7.79 (d, J = 4.1 Hz, 1H), 7.47 (d, J = 8.1 Hz, 2H), 7.38 (d, J = 8.0 Hz, 2H), 7.20 (d, J = 4.0 Hz, 1H), 4.84 (s, 2H), 4.53 (s, 2H). \]

\[ 1^{3}C \text{NMR} (201 \text{ MHz, DMSO-d}_6): \delta 167.7, 166.4, 151.9, 151.8, 151.6, 136.9, 134.5, 132.1, 131.6, 130.6, 127.9, 127.1, 123.2, 114.7, 114.6, 48.4, 40.5 \]

FTIR (neat, \( V_{\text{max}} \text{ cm}^{-1} \)): 3152, 3138, 3122, 1786, 1772, 1719, 1512, 1194, 1349.

HRMS (ESI) \( m/z \ [M + Na]^+ \): Calcd. for C\(_{23}\)H\(_{15}\)N\(_5\)O\(_7\): 496.0864, found: 496.0864.

**Rac-6-fluoro-1'-phenylspiro[chromane-4,4'-imidazolidine]-2',5'-dione (9) [NEW]**

\[ 19^{F} \text{NMR} (376 \text{ MHz, CDCl}_3): \delta -121.5 \]

Deviation from standard procedure: The reaction was performed on a 0.18 mmol scale. 9 was obtained after column chromatography [hexane:acetone (90:10% → 70:30%)] as a colorless, amorphous solid (42.7 mg, 75%).

\[ 1^H \text{NMR} (800 \text{ MHz, CDCl}_3): \delta 7.44 (t, J = 7.7 Hz, 2H), 7.37 (t, J = 8.2 Hz, 3H), 7.08 (s, 1H), 6.95 (td, J = 8.3, 3.1 Hz, 1H), 6.84 (dd, J = 8.9, 3.9 Hz, 2H), 4.65 (ddd, J = 11.9, 9.3, 2.9 Hz, 1H), 4.16 (ddd, J = 11.8, 6.3, 3.6 Hz, 1H), 2.37 (ddd, J = 14.1, 6.3, 2.9 Hz, 1H), 2.19 (ddd, J = 13.5, 9.3, 3.6 Hz, 1H). \]

\[ 1^{3}C \text{NMR} (201 \text{ MHz, CDCl}_3): \delta 173.5, 156.9 (d, J_{CF} = 240.6 \text{ Hz}), 155.7, 151.6 (d, J_{CF} = 2.0 \text{ Hz}), 131.0, 129.2, 128.5, 126.1, 119.5 (d, J_{CF} = 6.3 \text{ Hz}), 119.3 (d, J_{CF} = 7.8 \text{ Hz}), 117.9 (d, J_{CF} = 2.3 \text{ Hz}), 112.4 (d, J_{CF} = 23.7 \text{ Hz}), 62.5, 58.8, 32.1. \]

\[ 19^{F} \text{NMR} (376 \text{ MHz, CDCl}_3): \delta -121.5 \]

FTIR (neat, \( V_{\text{max}} \text{ cm}^{-1} \)): 3276, 3116, 3068, 1779, 1716, 1491, 1262.

HRMS (ESI) \( m/z \ [M + Na]^+ \): Calcd. for C\(_{17}\)H\(_{13}\)FN\(_2\)O\(_3\): 335.0802, found: 335.0802.
References

[21] Kapui, Z; Vasas, A; Buzder-Lantos, P; Batori, S; Szabo, T; Urban-Szabo, K; Ferencyz, G; Balogh, M. Prepartion of hydantoin derivatives as CD38 inhibitors for treatment of inflammatory disease, EP 2801573 A1, 2014
[22] Hubele, A. Imidazolidin-2,4-dion-derivate, DE 2441601 A1, 1975
$^1$H NMR spectrum of N-(4-iodobenzyl)phthalimide (600 MHz, CDCl$_3$)
$^{13}$C NMR spectrum of $N$-(4-iodobenzyl)phthalimide (151 MHz, CDCl$_3$)
$^1$H NMR spectrum of 1g (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 1g (151 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 1e (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 1e (151 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 2a (600 MHz, DMSO-$_d_6$)
$^{13}$C NMR spectrum of 2a (151 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 2b (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 2b (151 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 2c (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 2c (151 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 2d (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 2d (151 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 2e (600 MHz, DMSO-$d_6$)
$\text{13}^C \text{NMR spectrum of 2e (151 MHz, DMSO-$d_6$)}$
$^{19}$F NMR spectrum of 2e (376 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 2f (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 2f (151 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 2g (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 2g (151 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 2h (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 2h (151 MHz, DMSO-$d_6$)
\(^1\)H NMR spectrum of 2i (600 MHz, DMSO-\(d_6\))
$^{13}$C NMR spectrum of 2i (151 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 2j (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 2j (151 MHz, DMSO-d$_6$)
$^{19}\text{F} \text{ NMR spectrum of 2j (376 MHz, DMSO-$d_6$)}$
$^1$H NMR spectrum of 2k (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 2k (151 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 2l (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 2l (151 MHz, DMSO-$d_6$)
$^{19}$F NMR spectrum of 2l (376 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 2m (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 2m (151 MHz, DMSO-$d_6$)
$^{19}$F NMR spectrum of 2m (376 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 2n (600 MHz, DMSO-d$_6$)
$^{13}$C NMR spectrum of 2n (151 MHz, DMSO-$d_6$)
\(^1\)H NMR spectrum of 2o (600 MHz, DMSO-\(d_6\))
$^{13}$C NMR spectrum of 2o (151 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 2p (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 2p (151 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 2q (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 2q (151 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 2r (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 2r (151 MHz, DMSO-$d_6$)
$^{19}$F NMR spectrum of 2r (376 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 2s (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 2s (151 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 3a (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 3a (151 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 3b (600 MHz, CDCl$_3$)
$^{13}$C NMR spectrum of 3b (151 MHz, CDCl$_3$)
$^1$H NMR spectrum of 3c (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 3c (151 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 3d (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 3d (151 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 3e (600 MHz, CDCl$_3$)
$^{13}$C NMR spectrum of 3e (151 MHz, CDCl$_3$)
$^{19}$F NMR spectrum of 3e (376 MHz, CDCl$_3$)
$^1$H NMR spectrum of 3f (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 3f (151 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 3g (600 MHz, CDCl$_3$)
$^{13}$C NMR spectrum of 3g (151 MHz, CDCl₃)
$^1$H NMR spectrum of 3h (800 MHz, CDCl$_3$)
$^{13}$C NMR spectrum of 3h (201 MHz, CDCl$_3$)
$^1$H NMR spectrum of 3i (600 MHz, CDCl$_3$)
${^{13}C}$ NMR spectrum of 3i (151 MHz, CDCl$_3$)
$^1$H NMR spectrum of 3j (800 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 3j (201 MHz, DMSO-$_{d6}$)
$^{19}$F NMR spectrum of 3j (376 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 3k (800 MHz, CDCl$_3$)
$^{13}$C NMR spectrum of 3k (201 MHz, CDCl$_3$)
$^{19}$F NMR spectrum of 3k (376 MHz, CDCl$_3$)
$^1$H NMR spectrum of 3l (600 MHz, CDCl$_3$)
$^{13}$C NMR spectrum of 3l (151 MHz, CDCl$_3$)
$^1$H NMR spectrum of rac-3m (600 MHz, CDCl$_3$)
$^{13}$C NMR spectrum of rac-3m (151 MHz, CDCl$_3$)
$^1$H NMR spectrum of (R)-3m (600 MHz, CDCl$_3$)
$^{13}$C NMR spectrum of (R)-3m (151 MHz, CDCl$_3$)
\(^1\)H NMR spectrum of 3n (600 MHz, CDCl\(_3\))
$^{13}$C NMR spectrum of 3n (151 MHz, CDCl$_3$)
$^1$H NMR spectrum of (S)-3o (600 MHz, CDCl$_3$)
$^{13}$C NMR spectrum of (S)-3o (151 MHz, CDCl$_3$)
$^1$H NMR spectrum of 3p (600 MHz, CDCl$_3$)
$^{13}$C NMR spectrum of 3p (151 MHz, CDCl$_3$)
$^1$H NMR spectrum of 3r (600 MHz, CDCl$_3$)
$^{13}$C NMR spectrum of 3r (151 MHz, CDCl$_3$)
$^1$H NMR spectrum of 3s (600 MHz, CDCl$_3$)
$^{13}$C NMR spectrum of 3s (151 MHz, CDCl$_3$)
$^1$H NMR spectrum of 3t (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 3t (151 MHz, DMSO-$d_6$)
$^{19}$F NMR spectrum of 3t (376 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 3u (600 MHz, CDCl$_3$)
$^{13}$C NMR spectrum of 3u (151 MHz, CDCl$_3$)
$^{19}\text{F NMR spectrum of 3u (376 MHz, CDCl}_3$)
$^1$H NMR spectrum of 3v (600 MHz, CDCl$_3$)
$^{13}$C NMR spectrum of 3v (151 MHz, CDCl$_3$)
$^1$H NMR spectrum of 3w (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 3w (151 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 3x (800 MHz, CDCl$_3$)
$^{13}$C NMR spectrum of 3x (201 MHz, CDCl$_3$)
$^1\text{H NMR}$ spectrum of 3y (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 3y (151 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 3z (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 3z (151 MHz, DMSO-$d_6$)
$^{19}$F NMR spectrum of 3z (376 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 3aa (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 3aa (151 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 5a (600 MHz, CDCl$_3$)
$^{13}$C NMR spectrum of 5a (151 MHz, CDCl$_3$)
$^1$H NMR spectrum of 5b (800 MHz, CDCl$_3$)
$^{13}$C NMR spectrum of 5b (201 MHz, CDCl$_3$)
$^1$NMR spectrum of 5c (800 MHz, CDCl$_3$)
$^{13}$C NMR spectrum of 5c (201 MHz, CDCl$_3$)
$^1$H NMR spectrum of 7a (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 7a (151 MHz, DMSO-$d_6$)
$^{1}$H NMR spectrum of 7b (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 7b (201 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 7c (600 MHz, DMSO-$d_6$)
$^{13}$C NMR spectrum of 7c (201 MHz, DMSO-$d_6$)
$^1$H NMR spectrum of 9 (800 MHz, CDCl$_3$)
$^{13}$C NMR spectrum of 9 (201 MHz, CDCl$_3$)
19F NMR spectrum of 9 (376 MHz, CDCl₃)