Supporting Information

In Situ Activation of Benzyl Alcohols with XtalFluor-E: Formation of 1,1-Diarylmethanes and 1,1,1-Triarylmethanes Through Friedel-Crafts Benzylation

Justine Desroches, Pier Alexandre Champagne, Yasmine Benhassine and Jean-François Paquin*

Canada Research Chair in Organic and Medicinal Chemistry, CGCC, PROTEO, Université Laval, Département de chimie, 1045 avenue de la Médecine, Pavillon Alexandre-Vachon, Québec, QC, G1V 0A6, Canada

\[
\begin{align*}
\text{R} & = \text{H or Ar} \\
\text{1,1-diarylmethanes (R = H)} & \\
\text{1,1,1-triarylmethanes (R = Ar)}
\end{align*}
\]
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I. General information

All reactions were carried out under a nitrogen or argon atmosphere. Unless otherwise noted, all commercial reagents were used without further purification. Solvents were used directly out of the bottle. Thin-layer chromatography (TLC) analysis of reaction mixtures was performed using Silicycle silica gel 60 Å F254 TLC plates, and visualized under UV. Flash column chromatography was carried out on Silicycle Silica Gel 60 Å, 230 X 400 mesh. High-resolution mass spectra were obtained on a LC/MS-TOF Agilent 6210 using either electrospray ionization (ESI) or atmospheric pressure photoionization (APPI). Nuclear magnetic resonance (NMR) spectra were recorded using Agilent DD2 500 and Varian Inova 400 spectrometers. $^1$H and $^{13}$C chemical shifts are reported in ppm downfield of tetramethylsilane and referenced to tetramethylsilane ($\delta = 0$ ppm) or residual chloroform peak ($\delta = 7.26$ ppm). For $^{19}$F NMR, CFCl$_3$ ($\delta = 0$ ppm) is used as the external standard. Coupling constants ($J$) are measured in hertz (Hz). Multiplicities are reported using the following abbreviations: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad resonance. When possible, NMR assignment for peaks of the different isomers is given. Infrared spectra were recorded using a Thermo Scientific Nicolet 380 FT-IR spectrometer. Melting points (m.p.) were recorded on a Stanford ResearchSystem OptiMelt capillary melting point apparatus and are uncorrected.

II. Preparation of benzyl alcohols

1. By reduction of carbonyl compounds

![Reduction of Carbonyl Compounds](image)

4-tert-butylbenzyl alcohol (4). In a round-bottomed flask, 4-tert-butylbenzaldehyde (5.00 g, 30.8 mmol, 1 equiv.) was diluted in absolute ethanol (20 mL), then a suspension of NaBH$_4$ (768 mg, 20.3 mmol, 0.66 equiv.) in 30 mL of absolute EtOH was added. The reaction mixture was allowed to stir for 30 minutes at room temperature. The reaction mixture was quenched with aq. 10% NaOH and stirred until the
solution was homogeneous. Water was added and ethanol was evaporated under reduced pressure. The aqueous mixture was extracted with CH$_2$Cl$_2$ (3x) and the combined organic extracts were washed with NaHCO$_3$ (aq. 5%), then water. The solution was dried over anhydrous Na$_2$SO$_4$, filtered and concentrated in vacuo, affording the pure title compound (5.06 g, 100%) as a clear liquid. Spectral data were identical to those previously reported.¹

**4-chlorobenzyl alcohol (S1).** Following the procedure used for the preparation of 4 on a 460 mg scale of 4-chlorobenzaldehyde, the pure product was obtained as a white solid (461 mg, 100%). Spectral data were identical to those previously reported.²

![4-chlorobenzyl alcohol](image)

**3,5-dimethoxybenzyl alcohol (S2).** Following the procedure used for the preparation of 4 on a 300 mg scale of 3,5-dimethoxybenzaldehyde, the pure product was obtained (304 mg, 100%) as a colorless oil. Spectral data were identical to those previously reported.²

![3,5-dimethoxybenzyl alcohol](image)

**4-nitrobenzyl alcohol (S3).** Following the procedure used for the preparation of 4 on a 500 mg scale of 4-nitrobenzaldehyde, the pure product was obtained as a pale yellow solid (506 mg, 100%). Spectral data were identical to those previously reported.³

![4-nitrobenzyl alcohol](image)

**3-nitrobenzyl alcohol (S4).** Following the procedure used for the preparation of 4 on a 500 mg scale of 3-nitrobenzaldehyde, the pure product was obtained as a yellow oil (505 mg, 100%). Spectral data were identical to those previously reported.³

![3-nitrobenzyl alcohol](image)

**Diphenylmethanol (S5).** Following the procedure used for the preparation of 4 on a 1.00 g scale of benzophenone, the pure product was obtained as a white solid (1.01 g, 100%). Spectral data were identical to those previously reported.⁴

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2. Preparation of 4-(hydroxymethyl)phenyl acetate

\[
\begin{align*}
\text{HO} & \quad \text{AcCl, Et}_3\text{N} \\
\text{AcO} & \quad \text{AcOEt, 0 °C, 5 h}
\end{align*}
\]

**4-formylphenyl acetate (S6).** To a stirred solution of commercially available 4-hydroxybenzaldehyde (200 mg, 1.64 mmol, 1 equiv.) in 2.0 mL of ethyl acetate at 0 °C, under argon, was added triethylamine (228 µL, 1.63 mmol, 1 equiv.) followed by acetyl chloride (117 µL, 1.63 mmol, 1 equiv.) with 1.3 mL ethyl acetate. The reaction mixture was stirred 5 h at 0 °C. The reaction was filtered and evaporated. The crude product was not isolated and directly engaged in the next reaction.

\[
\begin{align*}
\text{AcO} & \quad \text{NaBH}_4 \\
\text{EtOH} & \quad \text{-78 °C, 30 min}
\end{align*}
\]

**4-(hydroxymethyl)phenyl acetate (S7).** In a round-bottomed flask, crude 4-formylphenyl acetate (260 mg, 1.58 mmol, 1 equiv.) was diluted in absolute ethanol (4 mL), then sodium borohydride (40 mg, 1.04 mmol, 0.66 equiv.) was added at -78 °C. The reaction mixture was allowed to stir for 30 minutes at room temperature, upon which the reaction appeared completed by TLC analysis. The reaction mixture was filtered. Water was added and ethanol was carefully evaporated under reduced pressure. The aqueous mixture was extracted with CH\(_2\)Cl\(_2\) (3x) and the combined organic extracts were washed with NaHCO\(_3\) (aq. 5%), then water. The solution was dried over anhydrous Na\(_2\)SO\(_4\), filtered and concentrated in vacuo, affording the pure title compound (108 mg, 41%) as a clear liquid. Spectral data were identical to those previously reported.\(^5\)

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3. Preparation of (2,5-dimethylphenyl)(phenyl)methanol

![Reaction Scheme](image)

**(2,5-dimethylphenyl)(phenyl)methanone (S8).** In a round-bottomed flask, benzoyl chloride (826 μL, 7.11 mmol, 1 equiv.) and *p*-xylene (1.75 mL, 14.22 mmol, 2 equiv.) were dissolved in 15 mL CH₂Cl₂. AlCl₃ (950 mg, 7.11 mmol, 1 equiv.) was then added and the solution was allowed to stir for 18 hours at room temperature, when it was then diluted with H₂O. The mixture was extracted twice with CH₂Cl₂, then the combined organic extracts were washed twice with water, then once with brine. The solution was dried over MgSO₄, filtered and evaporated to yield a crude mixture which was then purified by silica gel chromatography using hexanes / ethyl acetate (97/3). The title compound (1.30 g, 87%) was obtained as a slightly orange liquid and its spectral data were identical to those previously reported.⁶

**(2,5-dimethylphenyl)(phenyl)methanol (S9).** To a solution of (2,5-dimethylphenyl)(phenyl)methanone (800 mg, 3.8 mmol, 1 equiv.) in 2 mL of absolute ethanol, a suspension of NaBH₄ (72 mg, 1.9 mmol, 0.5 equiv.) in 4 mL absolute ethanol was added. The reaction mixture was then heated to 45 °C for 4 hours, then 60 °C for an additional hour. A work-up following the reduction general procedure described above yielded a crude mixture, which was purified by silica gel chromatography using hexanes / ethyl acetate (90/10). The title compound (611 mg, 76%) was obtained as a white solid. m.p. = 83 – 85 °C; ¹H NMR (500 MHz, CDCl₃) δ (ppm) = 7.26 – 7.33 (m, 6H), 7.03 (m, 2H), 5.99 (d, J = 3.7 Hz, 1H), 2.33 (s, 3H), 2.20 (s, 3H), 2.07 (d, J = 3.8 Hz, 1H); ¹³C NMR (126 MHz, CDCl₃) δ (ppm) = 142.9, 141.2, 135.6, 132.1, 130.5, 128.5, 128.2, 127.5, 127.1, 126.9, 73.3, 21.2, 18.9; IR (ATR, ZnSe) ν (cm⁻¹) = 3191, 3108, 1446, 1040, 1018, 817, 741, 697; HRMS-ESI (+) calcd for C₁₅H₁₅ [M-OH]⁺ 195.1168, found 195.1173.

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III. General procedure

To a stirred solution of 50 mg of benzyl alcohol in a 1:9 mixture of 1,1,1,3,3,3-hexafluoro-2-propanol (HFIP) and \( \text{CH}_2\text{Cl}_2 \) (C = 0.25 M) under argon was added 5 equiv. of arene and 1.1 equiv. of XtalFluor-E. The mixture was stirred at room temperature for 4 hours. The reaction was quenched with \( \text{H}_2\text{O} \) and the aqueous layer was extracted 3 times with \( \text{CH}_2\text{Cl}_2 \). The organic phases were combined and washed with brine, dried over \( \text{MgSO}_4 \), filtrated and concentrated under reduced pressure.

IV. Benzylation of 4-tert-butylbenzyl alcohol with various arenes

2-(4-(tert-butyl)benzyl)-1,4-dimethoxybenzene (6). Following the general procedure, the product obtained was a mixture of 6 (90%) and the excess of 1,4-dimethoxybenzene. The yield was determined by NMR spectroscopy in reference to toluene. The desired product 6 could not be isolated because of co-elution with the excess 1,4-dimethoxybenzene. However, the \(^1\text{H} \) NMR spectral data were identical to those previously reported.\(^7\)  \( ^1\text{H} \) NMR (400 MHz, CDCl\(_3\)): \( \delta \) (ppm) = 7.30 (d, \( J = 7.9 \) Hz, 2H), 7.06 (d, \( J = 8.0 \) Hz, 3H), 6.97 (d, \( J = 6.5 \) Hz, 2H), 3.93 (s, 2H), 2.30 (s, 3H), 2.22 (s, 3H), 1.31 (s, 9H).

analysis of the crude product revealed a characteristic peak of diarylmethylenes around 3.9 ppm (4.00 ppm (s, 2H)) and two peaks could be observed corresponding to the methoxy groups (3.77 ppm (s, 3H); 3.82 ppm (s, 3H)).

**1-(4-(tert-butyl)benzyl)-2,3-dimethylbenzene (7).** Following the general procedure, the pure product (68 mg, 89%, 4.1:1 mixture of regioisomers) was obtained as a white solid without further purification. Spectral data were identical to those previously reported.\(^7\) \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm) = 7.35 (m, 2H), 7.18 (m, 1.5H), 7.11 (m, 1.5H), 7.06 (m, 1H), 7.00 (m, 1H), 4.04 (s, 0.38H, minor), 3.94 (s, 1.58H, major), 2.35 (s, 0.6H), 2.28 (s, 4.72H), 2.21 (s, 0.6H), 1.36 (s, 9H).

**1-benzyl-4-tert-butylbenzene (8).** 4-tert-butylbenzyl alcohol (4) (35 mg, 0.213 mmol, 1 equiv.) was dissolved in a mixture of benzene (0.77 mL, 90% of the volume required for a 0.25 M substrate concentration) and HFIP (85 μL, 10% of the volume required for a 0.25 M substrate concentration). XtalFluor-E (54 mg, 1.1 equiv.) was then added and the vial was sealed. The resulting solution was stirred for 18 h at 60 °C. The reaction was quenched with H\(_2\)O and extracted with CH\(_2\)Cl\(_2\) (3x). The combined organic extracts were washed with brine, dried with MgSO\(_4\), filtered and evaporated under reduced pressure to yield the pure product as a colorless oil (40 mg, 84%) without further purification. Spectral data were identical to those previously reported.\(^7\) \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta = 7.36–7.27\) (m, 4H), 7.25–7.17 (m, 3H), 7.13 (m, 2H), 3.96 (s, 2H), 1.31 (s, 9H).

**1-(4-(tert-butyl)benzyl)-4-methylbenzene (9).** Following the general procedure, the pure product (70 mg, 97%, 2.7:1 mixture of para:ortho regioisomers) was obtained as a colorless oil without further purification. Spectral data were identical to those previously reported.\(^7\) \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) (ppm) = 7.31 (m, 2H), 7.19 – 7.11 (m, 5H), 7.08 (m, 1H), 3.97 (s, 0.52H, minor), 3.93 (s, 1.42H, major), 2.33 (s, 2.02H, major), 2.28 (s, 0.76H, minor), 1.32 (s, 9H).

**1-(4-(tert-butyl)benzyl)-4-methoxybenzene (10).** Following the general procedure, the pure product (50 mg, 97%, 1.2:1 mixture of para:ortho regioisomers) was obtained as a colorless oil without
further purification. Spectral data were identical to those previously reported.\(^7\) \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) (ppm) = 7.32 (m, 2H), 7.22 – 7.07 (m, 3H), 6.93 – 6.82 (m, 2H), 3.97 (s, 0.9H, minor), 3.92 (s, 1H, major), 3.84 (s, 1.3H, minor), 3.80 (s, 1.6H, major), 1.32 (s, 9H).

**1-tert-butyl-4-(4-fluorobenzyl)benzene (11).** 4-tert-butylbenzyl alcohol (4) (35 mg, 0.213 mmol, 1 equiv.) was dissolved in a mixture of fluorobenzene (0.77 mL, 90% of the volume required for a 0.25 M substrate concentration) and HFIP (85 μL, 10% of the volume required for a 0.25 M substrate concentration). XtalFluor-E (54 mg, 1.1 equiv.) was then added and the vial was sealed. The resulting solution was stirred for 18 h at 60 °C. The reaction was quenched with H\(_2\)O and extracted with CH\(_2\)Cl\(_2\) (3x). The combined organic extracts were washed with brine, dried with MgSO\(_4\), filtered and evaporated under reduced pressure. The crude product was purified by column chromatography using hexane/ethyl acetate (99/1) to yield the pure product as a colorless oil (43 mg, 83%, 1.6:1:12.5 ratio of ortho:meta:para regioisomers). Spectral data were identical to those previously reported.\(^7\) \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) (ppm) = 7.35 (m, 1.7H), 7.28 (m, 0.2H), 7.16 (m, 1.8H), 7.11 (m, 1.4H), 7.05 (m, 0.2H), 6.97 (m, 1.9H), 4.01 (s, 0.2H), 3.96 (s, 1.7H), 3.89 (s, 0.1H), 1.34 (m, 9H); \(^19\)F NMR (470 MHz, CDCl\(_3\)): \(\delta\) (ppm) = -117.47 (ddd, \(J = 14.1, 8.9, 5.4\) Hz, 1F), -117.70 (ddd, \(J = 14.2, 8.8, 5.4\) Hz, 0.08F), -117.88 (m, 0.13F).

**2-(4-(tert-butyl)benzyl)naphthalene (12).** Following the general procedure, the pure product (61 mg, 73%, 3.7:1 mixture of regioisomers) was obtained as a white solid after flash chromatography (100% hexanes). Spectral data were identical to those previously reported.\(^7\) \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) (ppm) = 8.08 (m, 1H), 7.90 (m, 1H), 7.83 – 7.79 (m, 1.7H), 7.70 (s, 0.3H), 7.53 – 7.43 (m, 3.3H), 7.40 – 7.31 (m, 3.7H), 7.22 – 7.17 (m, 2.5H), 4.47 (s, 2H, major), 4.16 (s, 0.53H, minor), 1.35 (s, 2.6H, minor), 1.34 (s, 9H, major).

**N-(4-(4-(tert-butyl)benzyl)phenyl)acetamide (13).** Following the general procedure, the product obtained was a mixture of 12 (78%, 1:1.7 mixture of para:ortho regioisomers) and the excess of acetanilide. The yield was determined by NMR spectroscopy in reference to toluene. The desired product 12 could not be isolated because of co-elution with the excess acetanilide. However, the \(^1\)H NMR analysis of the crude product revealed characteristic peaks of
diarylmethylenes around 3.9 ppm (3.91 (s, 1H) and 3.94 ppm (s, 1.7H)). This result indicates the presence of a mixture of two regioisomers. Based on $^1$H NMR chemical shift, analysis of the $^{13}$C NMR spectra, and comparison with data reported for $N$-(2-benzylphenyl)acetamide, we believe that the ortho isomer is the major one. In addition, a careful flash chromatography using 7/3 hexanes/ethyl acetate as the eluent allowed the isolation of a few pure fractions of a light brown solid containing only the ortho isomer. m.p. = 162.0 – 162.9 °C; $^1$H NMR (500 MHz, CDCl$_3$) δ (ppm) = 7.81 (d, $J = 8.0$ Hz, 1H), 7.30 (m, 3H), 7.24 (dd, $J = 7.7$, 1.6 Hz, 1H), 7.14 (t, 7.7 Hz, 1H), 7.10 (d, $J = 8.0$ Hz, 2H), 6.90 (bs, 1H), 3.95 (s, 2H), 1.98 (s, 3H), 1.31 (s, 9H); $^{13}$C NMR (126 MHz, CDCl$_3$) δ (ppm) = 168.3, 149.9, 136.1, 136.0, 131.8, 130.9, 128.1, 127.6, 126.0, 125.4, 124.2, 38.3, 34.6, 33.5, 24.3; HRMS-ESI (+) calcd for C$_{19}$H$_{23}$NO [M+H]$^+$ 282.1853, found 282.1855; IR (ATR, ZnSe) ν (cm$^{-1}$) = 3249, 2957, 1650, 1536, 1451, 751, 729, 715.

### 4-(4-(tert-butyl)benzyl)phenyl acetate (14)

Following the general procedure, the product obtained was a mixture of 13 (51%, 1:2.2 mixture of para:ortho regioisomers) and the excess of phenylacetate. The yield was determined by NMR spectroscopy in reference to dimethylformamide. The desired product 13 could not be isolated because of co-elution with the excess phenylacetate. However, the $^1$H NMR analysis of the crude product revealed characteristic peaks of diarylmethylenes around 3.9 ppm (3.89 (s, 1H) and 3.95 ppm (s, 2.2H)). This result indicates the presence of a mixture of two regioisomers. Based on $^1$H NMR chemical shift, analysis of the $^{13}$C NMR spectra, and comparison with data reported for 4-benzylphenyl acetate, we believe that the ortho isomer is the major one.

### 2-(4-(tert-butyl)benzyl)thiophene (15)

Following the general procedure, the pure product (54 mg, 78%, 2.9:1 mixture of regioisomers) was obtained as a colorless oil following column chromatography using 100% hexanes. The spectral data were identical to those previously reported. $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm) = 7.31 (m, 1.9H), 7.22 (m, 0.3H), 7.17 (m, 1.5H), 7.12 (m, 1.2H), 6.90 (m, 1.1H), 6.79 (m, 0.7H), 4.11 (s, 1.5H), 3.94 (s, 0.6H), 1.30 (s, 9H).

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V. Benzylation of various benzyl alcohols with \( p \)-xylene

\[
\begin{align*}
R^1\text{-}1,4\text{-dimethylbenzene} & \quad \xrightarrow{\text{XtalFluor-E (1.1 equiv.)}} \quad R^1\text{-}1,4\text{-dimethylbenzene} \\
\text{R}^1\text{-}4\text{-dimethylbenzyl)biphenyl} & \quad \xrightarrow{\text{XtalFluor-E (1.1 equiv.)}} \quad \text{R}^1\text{-}4\text{-dimethylbenzyl)biphenyl} \\
\end{align*}
\]

2-benzyl-1,4-dimethylbenzene (16). Following the general procedure, the product was obtained as a colorless oil (57 mg, 63%) without further purification. Spectral data were identical to those previously reported.\(^{10}\) \( ^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) (ppm) = 7.29 (t, \( J = 7.6 \) Hz, 2H), 7.20 (t, \( J = 7.0 \) Hz, 1H), 7.14 (m, 2H), 7.08 (d, \( J = 7.6 \) Hz, 1H), 6.99 (d, \( J = 7.9 \) Hz, 1H), 6.95 (s, 1H), 3.97 (s, 2H), 2.31 (s, 3H), 2.22 (s, 3H).

4-(2,5-dimethylbenzyl)biphenyl (17). Following the general procedure, the product was obtained as a white solid (73 mg, 88%) without further purification. Spectral data were identical to those previously reported.\(^{11}\) \( ^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta \) (ppm) = 7.64 (m, 2H), 7.57 (m, 2H), 7.48 (m, 2H), 7.38 (m, 1H), 7.26 (m, 2H), 7.14 (d, \( J = 8.0 \) Hz, 1H), 7.05 (d, \( J = 6.7 \) Hz, 2H), 4.06 (s, 2H), 2.37 (s, 3H), 2.29 (s, 3H).

2-(4-chlorobenzyl)-1,4-dimethylbenzene (18). To a mixture of 4-chlorobenzyl alcohol (50 mg, 0.351 mmol) and \( p \)-xylene (216 \( \mu \)L, 1.75 mmol, 5 equiv.) in CH\(_2\)Cl\(_2\) (1.26 mL, 90% of the volume required for a 0.25 M concentration) and HFIP (140 \( \mu \)L, 10% of the volume required for C = 0.25 M) was added XtalFluor-E (161 mg, 0.701 mmol, 2.0 equiv.). The mixture was stirred at room temperature for 48 hours. Water was then added and the aqueous phase was extracted with CH\(_2\)Cl\(_2\) (3x). The organic phases were combined, washed with brine, dried over MgSO\(_4\), filtered and evaporated under reduced pressure. The crude product was purified by flash chromatography using hexanes/ethyl acetate (99/1) to yield a colorless oil (58 mg, 72%). Spectral data were identical to those previously reported.\(^{7}\) \( ^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta \) (ppm) = 7.25


(dd, \( J = 8.6, 1.9 \text{ Hz}, 2 \text{H} \)), 7.06 (m, 3 \text{H}), 6.99 (d, \( J = 7.5 \text{ Hz}, 1 \text{H} \)), 6.92 (s, 1 \text{H}), 3.92 (s, 2 \text{H}), 2.31 (s, 3 \text{H}), 2.19 (s, 3 \text{H}).

**2-(4-bromobenzyl)-1,4-dimethylbenzene (19).** To a mixture of 4-bromobenzyl alcohol (57 mg, 0.304 mmol) and \( p \)-xylene (188 µL, 1.52 mmol, 5 equiv.) in \( \text{CH}_2\text{Cl}_2 \) (1.05 mL, 90% of the volume required for a 0.25 M concentration) and HFIP (120 µL, 10% of the volume required for C = 0.25 M) was added XtalFluor-E (139 mg, 0.608 mmol, 2.0 equiv.). The mixture was stirred at room temperature for 24 hours. Water was then added and the aqueous phase was extracted with \( \text{CH}_2\text{Cl}_2 \) (3x). The organic phases were combined, washed with brine, dried over MgSO\(_4\), filtered and evaporated under reduced pressure. The crude product was purified by flash chromatography using hexanes/ethyl acetate (99/1) to yield a colorless oil (59 mg, 71%). Spectral data were identical to those previously reported.\(^7\) \(^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta \) (ppm) = 7.39 (d, \( J = 7.9 \text{ Hz}, 2 \text{H} \)), 7.07 (d, \( J = 7.6 \text{ Hz}, 1 \text{H} \)), 7.00 (m, 3 \text{H}), 6.91 (s, 1 \text{H}), 3.90 (s, 2 \text{H}), 2.31 (s, 3 \text{H}), 2.18 (s, 3 \text{H}).

**2-(3-bromobenzyl)-1,4-dimethylbenzene (20).** To a mixture of 3-bromobenzyl alcohol (35 mg, 0.187 mmol) and \( p \)-xylene (115 µL, 0.936 mmol, 5 equiv.) in \( \text{CH}_2\text{Cl}_2 \) (0.67 mL, 90% of the volume required for a 0.25 M concentration) and HFIP (75 µL, 10% of the volume required for C = 0.25 M) was added XtalFluor-E (86 mg, 0.374 mmol, 2.0 equiv.). The mixture was stirred at room temperature for 48 hours. Water was then added and the aqueous phase was extracted with \( \text{CH}_2\text{Cl}_2 \) (3x). The organic phases were combined, washed with brine, dried over MgSO\(_4\), filtered and evaporated under reduced pressure. The crude product was purified by flash chromatography using 100% hexanes to yield a colorless oil (39 mg, 76%). \(^1\)H NMR (500 MHz, CDCl\(_3\)) \( \delta \) (ppm) = 7.32 (m, 1 \text{H}), 7.27 (m, 1 \text{H}), 7.13 (t, \( J = 7.8 \text{ Hz}, 1 \text{H} \)), 7.09 – 7.01 (m, 2 \text{H}), 6.99 (d, \( J = 7.4 \text{ Hz}, 1 \text{H} \)), 6.92 (s, 1 \text{H}), 3.92 (s, 2 \text{H}), 2.31 (s, 3 \text{H}), 2.19 (s, 3 \text{H}); \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \( \delta \) (ppm) = 143.1, 137.9, 135.7, 133.5, 131.8, 130.9, 130.5, 130.0, 129.2, 127.6, 127.5, 122.7, 39.2, 21.1, 19.4; HRMS-APPI calcd for C\(_{15}\)H\(_{13}\)Br [M\(^+\)] \( \text{+} \) 274.0357, found 274.0371; IR (ATR, ZnSe) \( \nu \) (cm\(^{-1}\)) = 3001, 2921, 1567, 1472, 1071, 809, 754, 685.
2-(2-bromobenzyl)-1,4-dimethylbenzene (21). To a mixture of 2-bromobenzyl alcohol (35 mg, 0.187 mmol) and p-xylene (115 µL, 0.936 mmol, 5 equiv.) in CH$_2$Cl$_2$ (0.67 mL, 90% of the volume required for a 0.25 M concentration) and HFIP (75 µL, 10% of the volume required for C = 0.25 M) was added XtalFluor-E (86 mg, 0.374 mmol, 2.0 equiv.). The mixture was stirred at room temperature for 48 hours. Water was then added and the aqueous phase was extracted with CH$_2$Cl$_2$ (3x). The organic phases were combined, washed with brine, dried over MgSO$_4$, filtered and evaporated under reduced pressure. The crude product was purified by flash chromatography using 100% hexanes to yield a colorless oil (42 mg, 81%). Spectral data were identical to those previously reported.\(^7\)\(^1\)H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm) = 7.60 (dd, $J$ = 7.9, 1.3 Hz, 1H), 7.18 (td, $J$ = 7.5, 1.3 Hz, 1H), 7.09 (m, 2H), 7.01 (dd, $J$ = 7.5, 1.3 Hz, 1H), 6.87 (m, 1H), 6.83 (s, 1H), 4.03 (s, 2H), 2.29 (s, 3H), 2.19 (s, 3H).

2-(4-methoxybenzyl)-1,4-dimethylbenzene (22). Following the general procedure, no desired compound could be obtained and a polymeric solid compound was isolated as the only product of the reaction.

2-(3,5-dimethoxybenzyl)-1,4-dimethylbenzene (23). Following the general procedure, the product (33 mg, 44%) was obtained as a colorless oil by column chromatography using hexanes/ethyl acetate (99/1). \(^1\)H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm) = 7.05 (d, $J$ = 7.6 Hz, 1H), 6.96 (d, $J$ = 7.4 Hz, 1H), 6.92 (s, 1H), 6.31 (t, $J$ = 2.3 Hz, 1H), 6.29 (m, 2H), 3.89 (s, 2H), 3.75 (s, 6H), 2.29 (s, 3H), 2.21 (s, 3H); \(^13\)C NMR (126 MHz, CDCl$_3$) $\delta$ (ppm) = 160.9, 143.2, 130.8, 130.3, 127.3, 107.1, 97.7, 55.4, 39.7, 21.1, 19.4; HRMS-ESI (+) m/z calcd for C$_{17}$H$_{20}$O$_2$ [M+H]$^+$ 257.1536 found 257.1535; IR (ATR, ZnSe) $\nu$ (cm$^{-1}$) = 2923, 1593, 1458, 1204, 1153, 1066, 908, 729.

4-(2,5-dimethylbenzyl)phenyl acetate (24). Following the general procedure, the product (47 mg, 61%) was obtained as a colorless oil by column chromatography using hexanes/ethyl acetate (95/5). Spectral data were identical to those previously reported.\(^7\) \(^1\)H NMR (400 MHz,
CDCl$_3$: \( \delta \) (ppm) = 7.12 (m, 2H), 7.06 (d, \( J = 7.6 \) Hz, 1H), 7.02 – 6.96 (m, 3H), 6.94 (s, 1H), 3.94 (s, 2H), 2.30 (s, 3H), 2.29 (s, 3H), 2.20 (s, 3H).

1,4-dimethyl-2-(4-nitrobenzyl)benzene (25). To a mixture of 4-nitrobenzyl alcohol (50 mg, 0.326 mmol) and \( p \)-xylene (201 \( \mu \)L, 1.63 mmol, 5 equiv.) in CH$_2$Cl$_2$ (1.13 mL, 90% of the volume required for a 0.25 M concentration) and HFIP (130 \( \mu \)L, 10% of the volume required for C = 0.25 M) was added XtalFluor-E (149 mg, 0.653 mmol, 2.0 equiv.). The mixture was stirred at room temperature for 72 hours. Water was then added and the aqueous phase was extracted with CH$_2$Cl$_2$ (3x). The organic phases were combined, washed with brine, dried over MgSO$_4$, filtered and evaporated under reduced pressure. The crude product was purified by flash chromatography (eluent: from hexanes/ethyl acetate 9/1 to hexanes/ethyl acetate 7/3) to yield a yellow oil (60 mg, 78%). $^1$H NMR (500 MHz, CDCl$_3$) \( \delta \) (ppm) = 8.14 (m, 2H), 7.27 (m, 2H), 7.09 (d, \( J = 7.6 \) Hz, 1H), 7.02 (d, \( J = 7.7 \) Hz, 1H), 6.92 (s, 1H), 4.06 (s, 2H), 2.31 (s, 3H), 2.17 (s, 3H); $^{13}$C NMR (126 MHz, CDCl$_3$) \( \delta \) (ppm) = 148.7, 146.5, 137.0, 135.9, 133.5, 130.9, 130.7, 129.5, 127.9, 123.8, 39.5, 21.1, 19.3; HRMS-ESI (+) m/z calcd for C$_{15}$H$_{15}$NO$_2$ [M+H]$^+$ 242.1176 found 242.1174; IR (ATR, ZnSe) \( \nu \) (cm$^{-1}$) = 3076, 2921, 1515, 1344, 1109, 907, 798, 727.

1,4-dimethyl-2-(3-nitrobenzyl)benzene (26). To a mixture of 3-nitrobenzyl alcohol (50 mg, 0.326 mmol) and \( p \)-xylene (201 \( \mu \)L, 1.63 mmol, 5 equiv.) in CH$_2$Cl$_2$ (1.13 mL, 90% of the volume required for a 0.25 M concentration) and HFIP (130 \( \mu \)L, 10% of the volume required for C = 0.25 M) was added XtalFluor-E (142 mg, 0.653 mmol, 2.0 equiv.). The mixture was stirred at room temperature for 72 hours. Water was then added and the aqueous phase was extracted with CH$_2$Cl$_2$ (3x). The organic phases were combined, washed with brine, dried over MgSO$_4$, filtered and evaporated under reduced pressure. The crude product was purified by flash chromatography (eluent: from hexanes/ethyl acetate 9/1 to hexanes/ethyl acetate 7/3) to yield a colorless oil (51 mg, 64%). $^1$H NMR (500 MHz, CDCl$_3$): \( \delta \) (ppm) = 8.07 (m, 1H), 8.02 (s, 1H), 7.46 (m, 2H), 7.10 (d, \( J = 7.7 \) Hz, 1H), 7.03 (d, \( J = 7.8 \) Hz, 1H), 6.95 (s, 1H), 4.06 (s, 2H), 2.33 (s, 3H), 2.20 (s, 3H); $^{13}$C NMR (126 MHz, CDCl$_3$) \( \delta \) (ppm) = 148.5, 142.9, 137.10, 137.09, 135.9, 134.9, 133.4, 130.9, 130.7, 129.3, 127.9, 123.6, 121.28, 121.27, 77.4, 77.2, 76.9, 39.2,
21.1, 19.3; HRMS-ESI (+) m/z calcd for C_{15}H_{16}NO_{2} [M+H]^{+} 242.1176 found 242.1167; IR (ATR, ZnSe) ν (cm\(^{-1}\)) = 2921, 1526, 1346, 1095, 909, 816, 726, 671.

**3-(2,5-dimethylbenzyl)pyridine (27).** Following the general procedure, the product (21 mg, 23% (30% estimated by NMR spectroscopy using anisole as a reference)) was obtained as a colorless oil by column chromatography using hexanes/ethyl acetate (7/3). \(^1^H\) NMR (500 MHz, CDCl\(_3\)) δ (ppm) = 8.47 (d, J = 1.3 Hz, 1H), 8.45 (dd, J = 4.7, 1.0 Hz, 1H), 7.38 (dddt, J = 7.8, 2.4, 1.6, 0.7 Hz, 1H), 7.18 (ddd, J = 7.8, 4.8, 0.8 Hz, 1H), 7.07 (d, J = 7.7 Hz, 1H), 6.99 (dd, J = 7.7, 1.8 Hz, 1H), 6.91 (s, 1H), 3.94 (s, 2H), 2.29 (s, 3H), 2.19 (s, 3H); \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) δ (ppm) = 150.3, 147.6, 137.5, 136.2, 136.1, 135.8, 133.4, 130.8, 130.5, 127.6, 123.5, 36.7, 21.1, 19.3; HRMS-ESI (+) m/z calcd for C_{15}H_{15}N [M\(^{+}\)] 197.1199 found 197.1178; IR (ATR, ZnSe) ν (cm\(^{-1}\)) = 2920, 1422, 1027, 908, 808, 729, 710.

**2-(4-(chloromethyl)benzyl)-1,4-dimethylbenzene (28).** To a mixture of 4-chloromethylbenzyl alcohol (50 mg, 0.319 mmol) and p-xylene (197 µL, 1.60 mmol, 5 equiv.) in CH\(_2\)Cl\(_2\) (1.15 mL, 90% of the volume required for a 0.25 M concentration) and HFIP (130 µL, 10% of the volume required for C = 0.25 M) was added XtalFluor-E (146 mg, 0.653 mmol, 2.0 equiv.). The mixture was stirred at room temperature for 18 hours. Water was then added and the aqueous phase was extracted with CH\(_2\)Cl\(_2\) (3x). The organic phases were combined, washed with brine, dried over MgSO\(_4\), filtered and evaporated under reduced pressure. The crude product was purified by flash chromatography using hexanes/ethyl acetate (99/1) to yield a colorless oil (62 mg, 79%). \(^1^H\) NMR (500 MHz, CDCl\(_3\)) δ (ppm) = 7.31 (d, J = 8.1 Hz, 2H), 7.14 (d, J = 8.1 Hz, 2H), 7.08 (d, J = 7.6 Hz, 1H), 7.01 (m, 1H), 6.95 (d, J = 1.8 Hz, 1H), 4.59 (s, 2H), 3.97 (s, 2H), 2.32 (s, 3H), 2.21 (s, 3H); \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) δ (ppm) = 141.1, 135.6, 135.2, 130.9, 130.4, 129.2, 128.8, 127.4, 46.3, 39.1, 21.1, 19.3; HRMS-APPI (+) calcd for C_{16}H_{17}Cl [M\(^{+}\)] 244.1019, found 244.1038; IR (ATR, ZnSe) ν (cm\(^{-1}\)) = 2922, 1502, 1265, 1114, 908, 809, 729, 673.
VI. Synthesis of 1,1,1-triarylmethanes

((4-methoxyphenyl)methylene)dibenzene (29). Following the general procedure, the product (73 mg, 98%, 2.4:1:31 mixture of ortho:methyl:para regioisomers) was obtained by column chromatography using hexanes/ethyl acetate (99/1). Spectral data was identical to those previously reported.\(^\text{10}\) \(^1\)H NMR (400 MHz, CDCl\(_3\)) $\delta$ (ppm) = 7.28 (m, 4H), 7.22 (m, 2H), 7.11 (m, 4H), 7.02 (m, 2H), 6.88 – 6.80 (m, 2H), 5.93 (s, 0.07H, minor), 5.88 (s, 0.03H, minor), 5.51 (s, 0.91H, major), 3.79 (s, 2.81H, major), 3.72 (s, 0.22H, minor), 3.69 (s, 0.09H, minor).

(p-tolylmethylene)dibenzene (30). To diphenylmethanol (50 mg, 0.271 mmol) in toluene (0.98 mL, 90% of the volume required for a 0.25 M concentration) and HFIP (110 µL, 10% of the volume required for C = 0.25 M) was added XtalFluor-E (68 mg, 0.299 mmol, 1.1 equiv.). The mixture was stirred at room temperature for 48 hours. Water was then added and the aqueous phase was extracted with CH\(_2\)Cl\(_2\) (3x). The organic phases were combined, washed with brine, dried over MgSO\(_4\), filtered and evaporated under reduced pressure. The product (56 mg, 80%, 1:9.3 mixture of ortho:para regioisomers) was obtained by column chromatography using hexanes/ethyl acetate (99/1). Spectral data was identical to those previously reported.\(^\text{12}\) \(^1\)H NMR (400 MHz, CDCl\(_3\)) $\delta$ (ppm) = 7.29 (m, 4H), 7.22 (m, 2H), 7.11 (m, 6H), 7.02 (d, $J = 8.2$ Hz, 2H), 5.69 (s, 0.1H), 5.53 (s, 0.9H), 2.33 (s, 2.8H), 2.23 (s, 0.3H).

((3,4-dimethylphenyl)methylene)dibenzene (31). Following the general procedure, the product (65 mg, 89%, 1:22 mixture of ortho:para regioisomers) was obtained by column chromatography using hexanes/ethyl acetate (99/1). Spectral data was identical to those previously reported.\(^\text{13}\) \(^1\)H NMR (400 MHz, CDCl\(_3\)) $\delta$ (ppm) = 7.29 (m, 4H), 7.22 (m, 2H), 7.11 (m, 6H), 7.02 (d, $J = 8.2$ Hz, 2H), 5.69 (s, 0.1H), 5.53 (s, 0.9H), 2.33 (s, 2.8H), 2.23 (s, 0.3H).

CDCl$_3$ δ (ppm) = 7.36 (s, 0.2H), 7.28 (m, 4.5H), 7.21 (m, 2H), 7.12 (m, 4H), 7.05 (m, 1H), 6.91 (m, 1H), 6.83 (dd, $J = 7.8$, 1.5 Hz, 1.2H), 5.74 (s, 0.04H), 5.49 (s, 0.93H), 2.29 (s, 0.1H), 2.23 (s, 2.9H), 2.20 (s, 2.9H), 2.12 (s, 0.1H).

**2-((4-methoxyphenyl)(phenyl)methyl)-1,4-dimethylbenzene (32).** Following the general procedure, the product (62 mg, 87%, 1:11 mixture of ortho:para regioisomers) was obtained by column chromatography using hexanes/ethyl acetate (99/1). $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm) = 7.32 (m, 2H), 7.24 (m, 1H), 7.10 (m, 3H), 7.01 (m, 3H), 6.87 (m, 2H), 6.68 (s, 1H), 6.04 (s, 0.08H, minor), 5.65 (s, 0.92H, major), 3.83 (s, 2.75H), 3.75 (s, 0.25H), 2.26 (s, 3H), 2.21 (s, 3H); $^{13}$C NMR (126 MHz, CDCl$_3$) δ (ppm) = 158.0 (major), 157.3 (minor), 144.0 (major), 143.6 (minor), 142.5 (major), 142.2 (minor), 135.7 (major), 135.2 (major), 134.9 (minor), 133.7 (minor), 133.6 (major), 132.4 (minor), 130.7 (major), 130.5 (minor), 130.4 (major), 130.2 (minor), 130.1 (major), 129.9 (minor), 129.72 (minor), 129.68 (major), 128.4 (major), 128.2 (minor), 127.5 (minor), 127.1 (major), 126.9 (minor), 126.2 (major), 126.0 (minor), 120.4 (minor), 113.8 (major), 110.7 (minor), 55.8 (minor), 55.4 (major), 52.8 (major), 46.4 (minor), 21.4 (major), 19.6 (major), 19.4 (minor); HRMS-ESI (+) m/z calcd for C$_{22}$H$_{22}$O [M$^+$] 302.1665 found 302.1645; IR (ATR, ZnSe) ν (cm$^{-1}$) = 3025, 2922, 1509, 1245, 1033, 907, 729, 699.
VII. Reactions in the presence of a base

1. General procedure

To a stirred solution of 4-tert-butylbenzyl alcohol 4 (50 mg, 0.304 mmol) and p-xylene (188 µL, 1.52 mmol, 5 equiv.) in CH₂Cl₂ (1.09 mL, 90% of the volume required for a concentration of 0.25 M) and HFIP (121 µL, 10% of the volume required for a concentration of 0.25 M) was added XtalFluor-E (77 mg, 0.335 mmol, 1.1 equiv.) and the base. The mixture was stirred at room temperature for 4 hours. The reaction was quenched with H₂O and the aqueous layer was extracted with CH₂Cl₂. The organic phases were combined and washed with brine, dried over MgSO₄, filtrated and concentrated under reduced pressure. The yield was estimated by NMR spectroscopy of the crude product using anisole as a reference.

2. Results

<table>
<thead>
<tr>
<th>Entry</th>
<th>Base</th>
<th>(equiv)</th>
<th>NMR yield of 5 (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>NaHCO₃</td>
<td>0.1</td>
<td>73</td>
</tr>
<tr>
<td>2</td>
<td>NaHCO₃</td>
<td>1.0</td>
<td>65</td>
</tr>
<tr>
<td>3</td>
<td></td>
<td>0.1</td>
<td>83</td>
</tr>
<tr>
<td>4</td>
<td></td>
<td>1.0</td>
<td>80</td>
</tr>
</tbody>
</table>
VIII. Mechanistic investigations

To a solution of 4 (50 mg, 0.304 mmol, 1 equiv.) in a 9:1 mixture of CH$_2$Cl$_2$:HFIP (1.22 mL, C = 0.25 M) was added $p$-xylene (187 μL, 1.52 mmol, 5 equiv.) and 2,6-di-tert-butyl-4-methylpyridine (69 mg, 0.334 mmol, 1.1 equiv.). With vigorous stirring, triflic anhydride (51 μL, 0.304 mmol, 1 equiv.) was then added and the mixture was allowed to stir for 4 hours at room temperature. At this point, two paths were independently taken in different experiments:

1) After a work-up as described in the general procedure, an internal standard, 1,4-dimethoxybenzene (41 mg, 1 equiv.) was added to the crude mixture, which was completely dissolved in CDCl$_3$ for NMR yield measurements. Complete conversion of the benzylic alcohol is noticed, and a yield of 89% is measured.

2) The reaction is quenched with a 10% aq. HCl solution, then extracted with CH$_2$Cl$_2$ (3x). The combined organic extracts were then washed twice with HCl (10%), sat. NaHCO$_3$, then water. The organic phase is dried over MgSO$_4$, filtered and evaporated, at which point NMR analysis reveals 100% conversion and that the pyridine is still present in the crude mixture. 0.5 mL of a 4 M solution of HCl in dioxane was added to fully protonate the residual pyridine. The dioxane was then evaporated and the residue was purified through silica gel chromatography using hexanes / ethyl acetate (99/1) to obtain the pure 5 (50 mg, 65%) as the usual colorless oil.

This reaction was also tried using 100% CH$_2$Cl$_2$ as the reaction solvent and the NMR yield was identical: 89%.
IX. $^1\text{H}, ^{19}\text{F}$ and $^{13}\text{C}$ spectra

![Chemical structure](image)

**S1**

$^1\text{H}$; 400 MHz, CDCl$_3$
MeO-\[
\begin{array}{c}
\text{OH} \\
\text{OMe}
\end{array}
\]

**S2**

$^1$H; 400 MHz, CDCl$_3$
$\text{O}_2\text{N}$

$\text{S3}$

$^1\text{H}$; 400 MHz, CDCl$_3$
$\text{O}_2\text{N}$

**S4**

$^1\text{H}; 400 \text{ MHz, CDCl}_3$
$^1$H; 400 MHz, CDCl$_3$
S7

$^1$H; 400 MHz, CDCl$_3$
$^1$H; 400 MHz, CDCl$_3$
$^{1}H$; 500 MHz, CDCl$_3$
S9
$^{13}$C; 126 MHz, CDCl$_3$
$^1$H; 400 MHz, CDCl$_3$
^1H; 400 MHz, CDCl₃
$^1$H; 400 MHz, CDCl$_3$
$^1$H; 400 MHz, CDCl$_3$
$(2.7:1)$

$^1$H; 500 MHz, CDCl$_3$
$^{1}$H; 400 MHz, CDCl$_3$
$^{1}H$; 500 MHz, CDCl$_3$
$^{19}$F; 470 MHz, CDCl$_3$

(7.8:1)
$^1$H; 400 MHz, CDCl$_3$
SI-39

13
(ortho isomer)

$^{13}$C; 126 MHz, CDCl$_3$
SI-41

**1**H; 400 MHz, CDCl₃

![Chemical structure of compound 16](image)

**Chemical Shifts**:
- 1.01
- 1.15
- 1.45
- 1.50
- 2.87
- 2.91
- 3.07
- 3.16
- 3.29
- 3.55
- 3.75
- 3.94
- 4.04
- 4.36
- 4.68
- 4.82
- 5.27
- 5.38
- 5.47
- 5.70
- 5.92
- 6.36
- 6.48
- 6.56
- 6.68
- 6.74
- 6.80
- 6.88
- 6.96
- 7.02
- 7.08
- 7.14
- 7.23
- 7.34
- 7.44
- 7.52
- 7.60
- 7.68
- 7.76
- 7.80
- 7.84
- 7.88
- 8.00

**Resonance Peaks**:
- 1.01 (s, H-13)
- 1.15 (s, H-14)
- 1.45 (s, H-15)
- 1.50 (s, H-16)
- 2.87 (d, J=10 Hz, H-17)
- 2.91 (d, J=10 Hz, H-18)
- 3.07 (d, J=10 Hz, H-19)
- 3.16 (d, J=10 Hz, H-20)
- 3.29 (d, J=10 Hz, H-21)
- 3.55 (d, J=10 Hz, H-22)
- 3.75 (d, J=10 Hz, H-23)
- 3.94 (d, J=10 Hz, H-24)
- 4.04 (d, J=10 Hz, H-25)
- 4.36 (d, J=10 Hz, H-26)
- 4.68 (d, J=10 Hz, H-27)
- 4.82 (d, J=10 Hz, H-28)
- 5.27 (s, H-29)
- 5.38 (s, H-30)
- 5.47 (s, H-31)
- 5.70 (s, H-32)
- 5.92 (s, H-33)
- 6.36 (s, H-34)
- 6.48 (s, H-35)
- 6.56 (s, H-36)
- 6.68 (s, H-37)
- 6.74 (s, H-38)
- 6.80 (s, H-39)
- 6.88 (s, H-40)
- 6.96 (s, H-41)
- 7.02 (s, H-42)
- 7.08 (s, H-43)
- 7.14 (s, H-44)
- 7.23 (s, H-45)
- 7.34 (s, H-46)
- 7.44 (s, H-47)
- 7.52 (s, H-48)
- 7.60 (s, H-49)
- 7.68 (s, H-50)
- 7.76 (s, H-51)
- 7.80 (s, H-52)
- 7.84 (s, H-53)
- 7.88 (s, H-54)
- 8.00 (s, H-55)

**H₂O**
$^1$H; 400 MHz, CDCl$_3$
$^1$H; 400 MHz, CDCl$_3$
$^{1}$H; 400 MHz, CDCl$_3$
$^{1}H$; 500 MHz, CDCl$_3$
$^{13}$C; 126 MHz, CDCl$_3$
^1H; 400 MHz, CDCl₃
$^{1}\text{H}; 500 \text{ MHz, CDCl}_3$
$^{13}$C; 126 MHz, CDCl$_3$
$^1$H; 400 MHz, CDCl$_3$
$^{1}H$; 500 MHz, CDCl$_3$
$^{13}$C; 126 MHz, CDCl$_3$
$^{1}H; 500 \text{ MHz, CDCl}_3$
O₂N

26

¹³C; 126 MHz, CDCl₃
$^1$H; 500 MHz, CDCl$_3$
$^{13}$C; 126 MHz, CDCl$_3$
\[ ^1H; 500 MHz, CDCl_3 \]
$^{13}$C; 126 MHz, CDCl$_3$
SI-59

\[ \text{OMe} \]

\[ \text{29} \]

(2.4:1:31)

\[ ^1\text{H}; 400 \text{ MHz, CDCl}_3 \]

\[ \text{H}_2\text{O} \]
$^{1}$H; 400 MHz, CDCl$_3$
$^1$H; 400 MHz, CDCl$_3$
$^1$H; 400 MHz, CDCl$_3$
$^{13}$C; 126 MHz, CDCl$_3$