Supporting Information for:

“Direct, metal-free synthesis of benzyl alcohols and deuterated benzyl alcohols from \( p \)-toluenesulfonylhydrazones using water as solvent”

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1. General Considerations

The starting materials were purchased from Aldrich Chemical Co. and were used without further purification. \( p \)-Toluenesulfonylhydrazones were prepared according to the literature.1-3 Solvents were distilled before use. Water was deionized from a Millipore, System Direct-Q equipment. Silica gel (230–400 mesh) was purchased from Merck. Silica plates of 0.20 mm thickness were used for thin layer chromatography. Melting points were determined with a Fischer-Johns Scientific melting point apparatus and they are uncorrected.

\(^1\)H and \(^{13}\)C NMR spectra were recorded using a Bruker Avance 300 MHz, and a Varian 500 MHz, The chemical shifts (δ) are given in ppm relative to TMS as internal standard (0.00). For analytical purposes the mass spectra were recorded on a Shimadzu GCMS-QP2010 Plus and on a JEOL JMS-5X 10217 in the EI mode, 70 eV, 200 °C via direct inlet probe. Only the molecular and parent ions (m/z). IR spectra were recorded on a Nicolet Magna 55-X FT instrument. The microwave-assisted reactions were performed using a focused microwave unit Anton-Paar Synthos 300, constant factor of the microwave 1.214. The temperature was monitored with an infrared temperature sensor. In all experiments the microwave temperature was held constant. Reactions were carried out in 5 mL glass vessels, which were sealed with a cap septum. The specific reaction time corresponds to the total irradiation time.

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2. General procedure for the synthesis of alcohols from reductive couplings of tosylhydrazones to Water

2.1 Reactions performed under conventional heating

CAUTION! Although we did not have any incidents by handling, it is known that diazoalkanes are presumed to be highly toxic and potentially explosive.4 All manipulations should be carried out in a hood.

Typical procedure. A solution of the appropriate tosylhydrazone (0.3mmol) with potassium carbonate (145.1mg, 3.5 equiv.) and water ID (10 mL). The reaction
mixture was heated at 100 °C with stirring for 12 h. The reaction was monitored by TLC. The mixture was cooled to room temperature and the characteristic colored organic layer was separated. The aqueous phase was extracted with dichloromethane (3 X 10 mL). The mixture was dried over Na$_2$SO$_4$ and filtered. The solvent was removed under reduced pressure and the final product was purified by column chromatography (SiO$_2$, hexane/AcOEt 95:5).

2.2 Reactions performed under microwave irradiation

Typical procedure. A 5 mL microwave vial was charged with potassium carbonate (145.1 mg, 3.5 equiv, 1.05 mmol), the corresponding tosylhydrazone (0.5 mmol), H$_2$O (5 mL), and a cylindrical magnetic stirring bar. The vessel was sealed with a septum, placed into the microwave cavity and irradiated to heat the reaction mixture at 130°C in an Anton-Paar microwave equipment. The total heating time was 10 minutes with 130°C. When the reaction was completed, the vial was cooled down to room temperature. The vial was then opened and poured into a separating funnel. The layers were separated and the aqueous phase was extracted with dichloromethane (3 X 10 mL). The combined organic layers were washed with brine, dried over Na$_2$SO$_4$ and filtered. The solvent was removed under reduced pressure and the final product was purified by column chromatography (SiO$_2$, hexane/AcOEt 95:5).

3. - General procedure for the synthesis of deuterium labelled alcohols from reductive couplings of tosylhydrazones to deuterium oxide

3.1 Reactions performed under conventional heating

CAUTION! Although we did not have any incidents by handling, it is known that diazoalkanes are presumed to be highly toxic and potentially explosive. All manipulations should be carried out in a hood.

Typical procedure. The appropriate tosylhydrazone (0.3 mmol) was added to a stirred solution of K$_2$CO$_3$ (145.1 mg, 3.5 equiv, 1.05 mmol) in H$_2$O (10 mL). The resulting reaction mixture was heated at reflux temperature for 24 h. The reaction mixture was cooled to room temperature and organic layer was separated. The aqueous phase was extracted with dichloromethane (3 X 10 mL). The organic
phases were joined, dried over Na\textsubscript{2}SO\textsubscript{4} and filtered. The solvent was removed under reduced pressure and the final product was purified by column chromatography (SiO\textsubscript{2}, hexane/AcOEt 95:5).

### 3.2 Reactions performed under microwave irradiation

*Typical procedure.* A 5 mL microwave vial was charged with potassium carbonate (145.1 mg, 3.5 equiv), the tosylhydrazone (0.3 mmol), water deuterium (5 mL) and a triangular magnetic stirring bar. The vessel was sealed with a septum, placed into the microwave cavity and irradiated to heat the reaction mixture at the desired temperature in 130°C microwave apparatus. The total time was 30 minutes. When the reaction was completed, the vial was cooled down to room temperature. It was then opened and poured into a separating funnel. The vial was rinsed with a dichloromethane (15 mL). The layers were separated and the aqueous phase was extracted three times with dichloromethane. The combined organic layers were washed with two portions of brine and the dried over Na\textsubscript{2}SO\textsubscript{4} and filtered. Solvent was removed under reduced pressure. If necessary were purified by chromatography on silica gel.

### 3.3 Reactions performed under conventional heating

*CAUTION! Although we did not have any incidents by handling, it is known that diazoalkanes are presumed to be highly toxic and potentially explosive.*\textsuperscript{4} All manipulations should be carried out in a hood.

*Typical procedure.* The appropriate tosylhydrazone (0.3 mmol) was added to a stirred solution of K\textsubscript{2}CO\textsubscript{3} (145.1 mg, 3.5 equiv, 1.05 mmol) in H\textsubscript{2}O (10 mL). The resulting reaction mixture was heated at reflux temperature for 24 h. The reaction mixture was cooled to room temperature and organic layer was separated. The aqueous phase was extracted with dichloromethane (3 X 10 mL). The organic phases were joined, dried over Na\textsubscript{2}SO\textsubscript{4} and filtered. The solvent was removed under reduced pressure and the final product was purified by column chromatography (SiO\textsubscript{2}, hexane/AcOEt 95:5)
4. - References.


5. Spectroscopic data of compounds

*p*-tolylmethanol (entry 1, Tables 2 and 4):

![OH](image)

4-Methylbenzaldehyde *p*-toluenesulfonylhydrazone afforded *p*-tolylmethanol as a white solid m.p. 61 °C. Yields: 31 mg (85%, from 0.3 mmol of tosylhydrazone under reflux conditions) and 60 mg (99%, from 0.5 mmol of tosylhydrazone under microwave conditions).

**IR (ATR, cm⁻¹):** 3267, 3049, 1618.

**HRMS (EI):** calcd. For C₈H₁₀O: 122.0732; found: 122.0735.

**¹H NMR:** (500 MHz, CDCl₃) δ = 2.26 (s, 2H), 4.54 (s, 3H), 7.08 (d, J = 7.5 Hz, 2H), 7.16 (d, J = 7.0 Hz, 2H).

**¹³C NMR:** (125 MHz, CDCl₃) δ = 21.30 (CH₃), 65.36 (CH₂), 127.27 (2xCH), 129.38 (2xCH), 137.51 (C), 138.07 (C).
Benzyl alcohol (entry 2, Tables 2 and 4):

Benzaldehyde p-toluenesulfonylhydrazone afforded benzyl alcohol as a colorless oil. Yields: 14.6 mg (45%, from 0.3 mmol of tosylhydrazone under reflux conditions) and 50.7 mg (94%, from 0.5 mmol of tosylhydrazone under microwave conditions).

IR (ATR, cm⁻¹): 3326, 3088, 1611.

HRMS (EI): calcd. For C₇H₈O: 108.0575; found: 108.0579.

¹H NMR: (500 MHz, CDCl₃) δ = 4.60 (s, 2H), 7.22 (m, 3H), 7.29 (m, 2H).

¹³C NMR: (125 MHz, CDCl₃) δ = 65.49 (CH₂), 127.15 (2xCH), 127.81 (2xCH), 128.72. (CH), 141.02 (C).

(4-chlorophenyl)methanol (entry 3, Tables 2 and 4):

4-Chlorobenzaldehyde p-toluenesulfonylhydrazone afforded (4-chlorophenyl)methanol as a white solid m.p. 72 °C. Yields: 17 mg (40%, from 0.3 mmol of tosylhydrazone under reflux conditions) and 67.5 mg (95%, from 0.5 mmol of tosylhydrazone under microwave conditions).

IR (ATR, cm⁻¹): 3270, 3259, 2955, 1592.

HRMS (EI): calcd. For C₇H₇ClO: 142.0185; found: 142.0187.

¹H NMR: (500 MHz, CDCl₃) δ = 4.64 (s, 2H), 7.27 (d, J= 8 Hz, 2H), 7.31 (d, J= 8 Hz, 2H).

¹³C NMR: (125 MHz, CDCl₃) δ = 64.64 (CH₂), 128.48 (2xCH), 128.85 (2xCH), 133.49 (C), 139.54 (C).
(3-chlorophenyl)methanol (entry 4, Tables 2 and 4):

3-Chlorobenzaldehyde p-toluenesulfonylhydrazone afforded (3-chlorophenyl)methanol as a colorless oil. Yields: 28.1 mg (66%, from 0.3 mmol of tosylhydrazone under reflux conditions) and 65.3 mg (92%, from 0.5 mmol of tosylhydrazone under microwave conditions).

IR (ATR, cm⁻¹): 3321, 3067.

HRMS (EI): calcd. For C₇H₇ClO: 142.0185; found: 142.0188.

¹H NMR: (500 MHz, CDCl₃) δ = 4.59 (s, 2H), 7.15 (m, 1H), 7.19 (m, 1H), 7.21 (m, 1H), 7.42 (m, 1H).

¹³C NMR: (125 MHz, CDCl₃) δ = 64.67 (CH₂), 125.01 (CH), 127.12 (CH), 127.86 (CH), 129.98 (CH), 134.60 (C), 143.02 (C).

(4-dimethylaminophenyl)methanol (entry 5, Tables 2 and 4):

4-dimethylaminobenzaldehyde p-toluenesulfonylhydrazone afforded (4-dimethylaminophenyl)methanol as a colorless oil. Yields: 28.5 mg (63%, from 0.3 mmol of tosylhydrazone under reflux conditions) and 67.2 mg (89%, from 0.5 mmol of tosylhydrazone under microwave conditions).

IR (ATR, cm⁻¹): 3239, 3093, 1614.

HRMS (EI): calcd. For C₉H₁₃NO: 151.0997; found: 151.0999.

¹H NMR: (500 MHz, CDCl₃) δ = 2.90 (s, 3H), 2.98 (s, 3H), 4.48 (s, 2H), 7.16 (d, J = 8.9 Hz, 2H), 7.31 (d, J = 8.9 Hz, 2H).

¹³C NMR: (125 MHz, CDCl₃) δ = 40.43 (CH₃), 40.87 (CH₃), 53.90 (CH₂), 111.76 (C), 128.82 (2xCH), 134.43 (2xCH), 152.26 (C).
1-phenylethanol (entry 6, Tables 2 and 4):

Acetophenone \( p \)-toluenesulfonylhydrazone afforded 1-phenylethanol as a colorless oil. Yields: 21.9 mg (60%, from 0.3 mmol of tosylhydrazone under reflux conditions) and 57.9 mg (95%, from 0.5 mmol of tosylhydrazone under microwave conditions).

**IR (ATR, cm\(^{-1}\))**: 3364, 3086, 2973, 1601.

**HRMS (EI)**: calcd. For C\(_8\)H\(_{10}\)O: 122.0732; found: 122.0735.

**\(^1\)H NMR**: (500 MHz, CDCl\(_3\)) \( \delta = 1.50 \) (d, 3H), 4.89 (q, 2H), 7.29-7.62 (m, 5H).

**\(^{13}\)C NMR**: (125 MHz, CDCl\(_3\)) \( \delta = 21.81 \) (CH\(_3\)), 65.40 (CH\(_2\)), 127.19 (2xCH), 127.79 (CH), 128.73 (2xCH), 141.30 (C).

1-(4-methoxyphenyl)ethanol (entry 7, Tables 2 and 4):

4-Methoxyacetophenone \( p \)-toluenesulfonylhydrazone afforded 1-(4-methoxyphenyl)ethanol as a colorless oil. Yields: 28.3 mg (62%, from 0.3 mmol of tosylhydrazone under reflux conditions) and 75.3 mg (99%, from 0.5 mmol of tosylhydrazone under microwave conditions).

**IR (ATR, cm\(^{-1}\))**: 3385, 3066, 1601.

**HRMS (EI)**: calcd. For C\(_9\)H\(_{12}\)O\(_2\): 152.0837; found: 152.0839.

**\(^1\)H NMR**: (500 MHz, CDCl\(_3\)) \( \delta = 1.38 \) (d, 3H), 3.70 (s, 3H), 4.75 (m, 1H), 6.78 (d, \( J = 8.6 \) Hz, 2H), 7.20 (d, \( J = 8.7 \) Hz, 2H).

**\(^{13}\)C NMR**: (125 MHz, CDCl\(_3\)) \( \delta = 25.18 \) (CH\(_3\)), 55.43 (CH\(_3\)), 70.09 (CH), 113.97 (CH), 126.81 (CH), 138.18 (C), 159.10 (C).
(2,6-dichlorophenyl)methanol (entry 8, Table 2):

2,6-Dichlorobenzaldehyde $p$-toluenesulfonylhydrazone afforded (2,6-dichlorophenyl)methanol as white solid m.p. 98ºC. Yield: 32.7 mg (62%).

IR (ATR, cm$^{-1}$): 3321, 3067.

HRMS (EI): calcd. For C$_7$H$_6$Cl$_2$O: 175.9796; found: 175.9799.

$^1$H NMR: (500 MHz, CDCl$_3$) $\delta$ = 4.97 (s, 2H), 7.32 (m, 1H), 7.35 (m, 2H).

$^{13}$C NMR: (125 MHz, CDCl$_3$) $\delta$ =60.47 (CH$_2$), 128.73 (2xCH), 128.84 (CH), 136.22 (2xC), 142.28 (C).

6-Benzyl-2-hydroxymethyl-6H-thieno[2,3-b]pyrrole-5-carboxylic acid methyl ester (entry 1, Table 3): Obtained as Colorless oil. Yield: 53.4 mg (59%).

IR (ATR, cm$^{-1}$): 3600, 3022, 2953, 1696, 1517.

HRMS (EI): calcd. For C$_{16}$H$_{15}$NO$_3$S: 301.0773; found: 301.0776.

$^1$H NMR: (300 MHz, CDCl$_3$) $\delta$ = 3.83 (s, 3H), 4.72 (s, 2H), 5.29 (s, 2H), 6. 55 (s, 1H), 7.22 - 7.32 (m, 5H), 7.36 (s, 1H).

$^{13}$C NMR: (75 MHz, CDCl$_3$) $\delta$ = 50.4 (CH$_2$), 51.8 (CH$_3$), 57.3 (CH$_2$), 102.8 (CH), 125.5 (CH), 127.5 (2xCH), 128.2 (CH), 128.4 (C), 128.8 (2xCH), 135.2. (C), 139.8 (2xC), 141.6 (C), 164.0 (C).
1-(6-Benzyl-5-hydroxymethyl-6H-thieno[2,3-b]pyrrol-2-yl)-ethanol (entry 2, Table 3): Obtained as Colorless oil. Yield: 38.8 mg (45%).

**IR (ATR, cm⁻¹):** 3600, 3321, 3067.

**HRMS (EI):** calcd. For C₁₆H₁₇NO₂S: 287.0980; found: 287.0984.

**¹H NMR:** (300 MHz, CDCl₃) δ = 1.52 (d, 3H), 4.60 (s, 2H), 4.95 (q, 1H), 5.21 (s, 2H), 6.29 (s, 1H), 6.81 (s, 1H), 7.17 (m, 2H), 7.27-7.30 (m, 3H).

**¹³C NMR:** (75 MHz, CDCl₃) δ = 24.9 (CH₃), 50.4 (CH₂), 57.6 (CH₂), 67.3 (CH), 101.9 (CH), 114.4 (CH), 127.5 (2xCH), 127.6 (C), 128.1 (CH), 129.0 (2xCH), 136.3 (C), 136.5 (C), 137.4 (C), 142.0 (C).

1-Thieno[2,3-b]pyridin-5-yl-ethanol (entry 3, Table 3): Obtained as Colorless oil. Yield: 30.1 mg (56%).

**IR (ATR, cm⁻¹):** 3321, 3067, 1597.

**HRMS (EI):** calcd. For C₉H₉NOS: 179.0405; found: 179.0408.

**¹H NMR:** (300 MHz, CDCl₃) δ = 1.54 (d, 3H), 5.04 (q, 1H), 7.19 (d, J = 5.7, Hz 1H) 7.50 (d, J = 6.0, Hz 1H), 8.07 (d, J = 2.1 Hz, 1H), 8.46 (d, J = 2.1 Hz 1H).

**¹³C NMR:** (75 MHz, CDCl₃) δ = 25.7 (CH₃), 68.2 (CH), 121.6 (CH), 127.6 (CH), 128.2 (CH), 132.6 (C), 137.6 (C), 145.1 (CH), 160.6 (C).
(2-methoxyphenyl) methanol (entry 8, Table 4):

4-Methoxybenzaldehyde p-toluenesulfonylhydrazone afforded (2-methoxyphenyl)methanol as a colorless oil. Yield: 68.3 mg (99%).

**IR (ATR, cm\(^{-1}\))**: 3366, 3066, 1690.

**HRMS (EI)**: calcd. For C\(_8\)H\(_{10}\)O\(_2\): 138.0681; found: 138.0684.

**\(^1\)H NMR**: (500 MHz, CDCl\(_3\)) \(\delta = 3.33\) (s, 3H), 4.16 (s, 3H), 6.33 (m, 1H), 6.37 (m, 1H), 6.44 (m, 1H), 6.76 (m, 1H).

**\(^{13}\)C NMR**: (125 MHz, CDCl\(_3\)) \(\delta = 55.36\) (CH\(_2\)), 62.12(CH\(_3\)), 126.47 (CH), 128.68 (CH), 129.04 (CH), 129.60 (CH), 131.05 (C), 157.53 (C).

(4-fluorophenyl)methanol (entry 9, Table 4):

4-Fluorobenzaldehyde p-toluenesulfonylhydrazone afforded (4-fluorophenyl)methanol as a colorless oil. Yield: 50.4 mg 80%.

**IR (ATR, cm\(^{-1}\))**: 3220, 3011, 1605.

**HRMS (EI)**: calcd. For C\(_7\)H\(_7\)FO: 126.0481; found: 126.0485.

**\(^1\)H NMR**: (500 MHz, CDCl\(_3\)) \(\delta = 4.56\) (s, 2H), 6.96 (d, \(J = 8.5\) Hz, 2H), 7.25 (d, \(J = 8.2\) Hz, 2H).

**\(^{13}\)C NMR**: (125 MHz, CDCl\(_3\)) \(\delta = 64.76\) (CH\(_2\)), 115.68 (2xCH), 128.97 (2xCH), 136.72 (C), 164.09 (C).
(4-bromophenyl)methanol (entry 10, Table 4):

4-Bromobenzaldehyde p-toluenesulfonylhydrazone afforded (4-bromophenyl)methanol as white solid, m.p. 82°C. Yield: 82.7 mg (89%).

**IR (ATR, cm\(^{-1}\))**: 3311, 3075, 1592.

**HRMS (EI)**: calcd. For C\(_7\)H\(_7\)BrO: 185.9680; found: 185.9682.

**\(^1\)H NMR**: (500 MHz, CDCl\(_3\)) \(\delta =4.53\) (s, 2H), 7.12 (d, \(J = 8.2\) Hz, 2H), 7.37 (d, \(J = 8.2\) Hz, 2H).

**\(^13\)C NMR**: (125 MHz, CDCl\(_3\)) \(\delta =64.66(\text{CH}_2), 121.56\) (C), 128.73 (2xCH), 131.75 (2xCH), 139.93 (C).

(2-bromophenyl)methanol (entry 11, Table 4):

2-Bromobenzaldehyde p-toluenesulfonylhydrazone afforded (2-bromophenyl)methanol as white solid, m.p. 78°C. Yield: 82.9 mg (89%).

**IR (ATR, cm\(^{-1}\))**: 3297, 3207, 2955, 1966.

**HRMS (EI)**: calcd. For C\(_7\)H\(_7\)BrO: 185.9680; found: 185.9687.

**\(^1\)H NMR**: (500 MHz, CDCl\(_3\)) \(\delta =4.67\) (s, 2H), 7.39 (m, 1H), 7.45 (m, 1H), 7.5 (m, 1H), 7.65 (dd, \(J = 7.8\) Hz, 1H).

**\(^13\)C NMR**: (125 MHz, CDCl\(_3\)) \(\delta =65.25\) (CH\(_2\)), 127.33 (C), 127.84 (CH), 129.40 (CH), 130.29 (CH), 136.98 (CH), 139.90 (C).
1-p-tolylethanol (entry 12, Table 4):

4-Methylacetophenone p-toluenesulfonylhydrazone afforded 1-p-tolylethanol as a colorless oil. Yield: 65.9 mg (97%).

**IR (ATR, cm⁻¹):** 3346, 3096,2920.

**HRMS (EI):** calcd. For C₉H₁₂O: 136.0888; found: 137.0891.

**¹H NMR:** (500 MHz, CDCl₃) δ =1.40 (d, J = 6.5 Hz, 3H), 2.26 (s, 3H), 4.78 (q, J = 6.4 Hz, 1H), 7.08 (d, J = 7.6 Hz, 2H), 7.18 (d, J = 7.6 Hz, 2H).

**¹³C NMR:** (125 MHz, CDCl₃) δ =21.25 (CH₃), 25.24 (CH₃), 70.39 (CH), 125.51 (2xCH), 129.31 (2xCH), 137.28 (C), 143.04 (C).

1-(4-chlorophenyl)ethanol (entry 13, Table 4):

4-Chloroacetophenone p-toluenesulfonylhydrazone afforded 1-(4-chlorophenyl)ethanol as a colorless oil. Yield: 77.2 mg (99%).

**IR (ATR, cm⁻¹):** 3346, 3086,1652.

**HRMS (EI):** calcd. For C₈H₉ClO: 156.0342; found: 158.0342.

**¹H NMR:** (500 MHz, CDCl₃) δ =1.34 (d, J = 6.5 Hz, 3H), 4.74 (q, J = 6.4 Hz, 1H), 7.27 (d, J = 8.6 Hz, 2H), 7.72 (d, J = 8.6 Hz, 2H).

**¹³C NMR:** (125 MHz, CDCl₃) δ =25.40 (CH₃), 69.83 (CH), 126.94(2xCH), 128.71 (2xCH), 133.15 (C), 144.42 (C).
Phenyl (O,1-\(^2\)H\(_2\)) methanol (entry 1, Tables 5 and 6):

Benzaldehyde \(p\)-toluenesulfonylhydrazone and deuterium oxide afforded Phenyl (O,1-\(^2\)H\(_2\)) methanol as a colorless oil. Yields: 27.1 mg (82%, from 0.3 mmol of tosylhydrazone under reflux conditions) and 53.9 mg (98%, from 0.5 mmol of tosylhydrazone under microwave conditions).

IR (ATR, cm\(^{-1}\)): 2327 (O-D).

HRMS (EI): calcd. For C\(_7\)H\(_6\)D\(_2\)O: 110.0699; found: 110.0702.

\(^1\)H NMR: (500 MHz, CDCl\(_3\)) \(\delta = 4.67\) (s, 1H), 7.36 (m, 2H), 7.37 (m, 3H).

\(^{13}\)C NMR: (125 MHz, CDCl\(_3\)) \(\delta = 64.97\) (CDH), 127.30 (2xCH), 127.95 (CH), 128.84 (2xCH), 141.03 (C).

\(p\)-tolyl (O,1-\(^2\)H\(_2\)) methanol (entry 2, Tables 5 and 6):

4-Methylbenzaldehyde \(p\)-toluenesulfonylhydrazone and deuterium oxide afforded \(p\)-tolyl (O,1-\(^2\)H\(_2\)) methanol as a colorless oil. Yields: 33.5 mg (90%, from 0.3 mmol of tosylhydrazone under reflux conditions) and 60.1 mg (97%, from 0.5 mmol of tosylhydrazone under microwave conditions).

IR (ATR, cm\(^{-1}\)): 2325 (O-D).

HRMS (EI): calcd. For C\(_8\)H\(_8\)D\(_2\)O: 124.0855; found: 124.0854.

\(^1\)H NMR: (500 MHz, CDCl\(_3\)) \(\delta = 2.35\) (s, 3H), 4.62 (s, 1H), 7.17 (d, 2H, \(J = 7.1\) Hz), 7.25 (d, \(J = 7.1\) 2H).

\(^{13}\)C NMR: (125 MHz, CDCl\(_3\)) \(\delta = 21.42\) (CH\(_3\)), 65.03 (CDH), 127.42 (2xCH), 129.51 (2xCH), 137.69 (C), 138.07 (C).
4-dimethylaminophenyl (O,1-^2^H) methanol (entry 3, Tables 5 and 6):

4-dimethylaminobenzaldehyde p-toluenesulfonylhydrazone and deuterium oxide afforded as 4-dimethylaminophenyl (O,1-^2^H) methanol as a colorless oil. Yields: 25.3 mg (55%, from 0.3 mmol of tosylhydrazone under reflux conditions) and 57.4 mg (75%, from 0.5 mmol of tosylhy-drazone under microwave conditions).

IR (ATR, cm⁻¹): 2330 (O-D).

HRMS (EI): calcd. For C₉H₁₁D₂NO: 153.1121; found: 153.1125.

¹H NMR: (500 MHz, CDCl₃) δ = 2.90 (s, 3H), 4.50 (s, 1H), 7.18 (d, J = 8.9 Hz, 2H), 7.33 (d, J = 8.9 Hz, 2H).

¹³C NMR: (125 MHz, CDCl₃) δ = 40.43 (2xCH₃), 53.90 (CDH), 111.76 (C), 128.82 (2xCH), 134.43 (2xCH), 152.26 (C).

4-chlorophenyl (O,1-^2^H) methanol (entry 4, Tables 5 and 6):

4-Chlorobenzaldehyde p-toluenesulfonylhydrazone and deuterium oxide afforded 4-chlorophenyl (O,1-^2^H) methanol as a colorless oil. Yields: 28.1 mg (65%, from 0.3 mmol of tosylhydrazone under reflux conditions) and 69.1 mg (96%, from 0.5 mmol of tosylhy-drazone under microwave conditions).

IR (ATR, cm⁻¹): 2325(O-D).

HRMS (EI): calcd. For C₇H₅D₂ClO: 144.0309; found: 144.03010.

¹H NMR: (500 MHz, CDCl₃) δ = 4.64 (s, 1H), 7.29 (d, J = 8.3 Hz, 2H), 7.32 (d, J = 8.3 Hz, 2H).

¹³C NMR: (125 MHz, CDCl₃) δ = 64.64 (CDH), 128.48 (2xCH), 128.85 (2xCH), 133.49 (C), 139.54 (C).
2,6-dichlorophenyl (O,1-^2H_2) methanol (entry 5, Tables 5 and 6):

2,6-Dichlorobenzaldehyde p-toluenesulfonylhydrazone and deuterium oxide afforded 2,6-dichlorophenyl (O,1-^2H_2) methanol as a colorless oil. Yields: 32.0 mg (60%, from 0.3 mmol of tosylhydrazone under reflux conditions) and 82.8 mg (93%, from 0.5 mmol of tosylhy-drazozone under microwave conditions).

IR (ATR, cm\(^{-1}\)): 2324 (O-D).

HRMS (EI): calcd. For C\(_7\)H\(_4\)D\(_2\)ClO: 177.9919; found: 177.9921.

\(^1\)H NMR: (500 MHz, CDCl\(_3\)) \(\delta = 4.91\) (s, 1H), 7.32 (m, 2H), 7.34 (m, 1H).

\(^13\)C NMR: (125 MHz, CDCl\(_3\)) \(\delta = 60.50\) (CDH), 128.76 (2xCH), 129.11 (CH), 131.34 (2xC), 142.31 (C).

1-phenyl (O,1-^2H_2) ethanol (entry 6 Tables 5 and 6):

Acetophenone p-toluenesulfonylhydrazone afforded 1-phenyl (O,1-^2H_2) ethanol as a colorless oil. Yields: 26.0 mg (70%, from 0.3 mmol of tosylhydrazone under reflux conditions) and 60.1 mg (97%, from 0.5 mmol of tosylhy-drazozone under microwave conditions).

IR (ATR, cm\(^{-1}\)): 2325 (O-D).

HRMS (EI): calcd. For C\(_8\)H\(_8\)D\(_2\)O: 124.0855; found: 125.0858.

\(^1\)H NMR: (500 MHz, CDCl\(_3\)) \(\delta = 1.51\) (s, 3H), 7.36 (m, 2H), 7.38 (m, 3H).

\(^13\)C NMR: (125 MHz, CDCl\(_3\)) \(\delta = 25.30\) (CH\(_3\)), 70.27 (CD), 125.64 (2xCH), 127.74 (CH), 128.77 (2xCH), 146.01 (C).
6. - $^1$H NMR and $^{13}$C NMR spectra for compounds

$p$-tolylmethanol:
Benzyl Alcohol:
(4-chlorophenyl)methanol:
(3-chlorophenyl)methanol:
(4-(dimethylamino)phenyl)methanol:
1-phenylethanol:

\[
\text{OH} \quad \text{CH}_3
\]
1-(4-methoxyphenyl)ethanol
(2,6-dichlorophenyl)methanol:
6-Benzyl-2-hydroxymethyl-6H-thieno[2,3-b]pyrrole-5-carboxylic acid methyl ester
1-(6-Benzyl-5-hydroxymethyl-6H-thieno[2,3-b]pyrrol-2-yl)-ethanol
1-Thieno[2,3-b]pyridin-5-yl-ethanol
(2-methoxyphenyl) methanol:
(4-fluorophenyl)methanol:
(4-bromophenyl)methanol:
(2-bromophenyl)methanol:
$1-\rho$-tolylethanol:
1-(4-chlorophenyl)ethanol:
Phenyl \((O,1-^2H_2)\) methanol
$p$-tolyl ($O, 1^{2}\text{H}_2$) methanol
4-dimethylaminophenyl (O,1-$^2$H$_2$) methanol
4-chlorophenyl ($O,1^{2}\text{H}_2$) methanol
2,6-dichlorophenyl \((O,1^{-2}H_2)\) methanol
1-phenyl (O,1-$^2$H$_2$) ethanol