Catalytic Reduction of $o/p$-Azidonitrobenzenes through *tert* Butoxide Ion Mediated Electron Transfer

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Contents:

S2: General experimental procedures
S3 – S8: Characterisation of compounds listed in Table 2
S9: References
S9 - S19: Copies of $^1$H and $^{13}$C NMR spectra of compounds listed in Table 2
Experimental

The azides were synthesized according to the literature procedure from the corresponding aniline. All reagents were purchased from Sigma Aldrich Inc., Alfa Aesar or Fischer Scientific (including thiazolium 3, CAS: 4568-71-2) and used without further purification. \(^1\)H and \(^{13}\)C-NMR spectra were recorded on a Bruker AV (III) 400, Bruker AV 400, Bruker DPX 400 (400MHz (\(^1\)H) and 100 MHz (\(^{13}\)C)), spectrometers. Chemical shifts are expressed in parts per million (ppm) and the spectra calibrated to residual solvent signals of DMSO (2.54 ppm (\(^1\)H) and 40.5 ppm (\(^{13}\)C)). Coupling constants are given in hertz (Hz) and the following notations indicate the multiplicity of the signals: s (singlet), br s (broad singlet), d (doublet), dd (double doublet), t (triplet), q (quartet), m (multiplet). High Resolution Mass Spectra were recorded on a VG micron Autospec or Bruker microTOF. Fourier Transform Infrared Spectroscopy (FT-IR) spectra were obtained using a Perkin Elmer 1600 series or Bruker Tensor 27 spectrometer. Melting points were recorded using a STUART SMP3 apparatus and are uncorrected. Thin layer chromatography was carried out on Merck pre-coated silica gel plates (60F-254) and visualised using ultra violet light or KMnO\(_4\) solution. THF was freshly distilled from sodium-benzophenone. Where necessary, reactions requiring anhydrous conditions were performed in dry solvents in flame dried or oven-dried apparatus under argon atmosphere.

**General procedure for the reduction of azidonitrobenzenes (Table 2)**

3

tert

mL) under argon at room temperature. The resulting suspension was stirred for 5 minutes, and then sodium tert-butoxide (23.4 mg, 0.244 mmol) was added in one portion, and the mixture stirred (see table 2). Water (2 mL) was added and the products extracted with EtOAc (2 x 2 mL), the combined organic phase was dried over anhydrous magnesium sulphate, filtered and concentrated in vacuo resulting residue through a short pad of silica (eluting with 50% light petroleum: ethyl acetate) unless otherwise stated.
4-Nitroaniline (2)

Yellow solid (15.0 mg, 88%), \( R_f \) (70:30 petrol-EtOAc) 0.3; mp 145-148 °C (lit., \(^2\) 146-147 °C); IR (FTIR, CHCl\(_3\)) \( \nu_{\text{max}} \) cm\(^{-1}\): 3509 (NH\(_2\)), 3417 (NH\(_2\)), 1624, 1600 1505 (NO\(_2\)), 1335, 1311; \(^1\)H NMR (400 MHz, DMSO-\(d_6\)) 7.96-7.92 (m, 2H), 6.70 (br s, 2H, NH\(_2\)), 6.62-6.58 (m, 2H); \(^{13}\)C NMR (100 MHz, DMSO-\(d_6\)) 155.7 (C), 135.6 (C), 126.4 (2 x CH), 112.4 (2 x CH); HRMS ESI: calcd for C\(_6\)H\(_4\)N\(_2\)NaO\(_2\) [M+Na]\(^+\), 161.0321; found, 161.0322.

(10)

\( J = 8.7, 1.3 \) Hz, 1H), 7.82-7.78 (m, 3H), 7.43 (dd, \( J = 8.7, 1.3 \) Hz, 1H), 7.05-7.01 (m, 1H); \(^{13}\)C NMR (100 MHz, DMSO-\(d_6\)) 146.2 (C), 135.7 (CH), 130.3 (C), 125.3 (CH), 119.1 (CH), 115.4 (CH); HRMS ESI: calcd for C\(_6\)H\(_5\)N\(_2\)O\(_2\) [M+H]\(^+\), 139.0502; found, 139.0490; calcd for C\(_6\)H\(_5\)N\(_2\)NaO\(_2\) [M+Na]\(^+\), 161.0321; found, 161.0328.
Pale yellow solid (21.0 mg, 84%), f (70:30 petrol-EtOAc) 0.5; mp 90-93 °C (lit.,4 92-93 °C); IR (FTIR, CHCl$_3$) $\nu_{\text{max}}$ cm$^{-1}$: 3535 (NH$_2$), 3434 (NH$_2$), 1635, 1592 (NO$_2$), 1520 (NO$_2$), 1119; $^1$H NMR (400 MHz, DMSO-$d_6$) 8.59 (d, $J = 2.7$ Hz, 1H), 8.55 (dd, $J = 9.3$, 2.7 Hz, 1H), 7.55 (br s, 2H), 7.32 (d, $J = 9.3$ Hz, 1H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) 151.8 (C), 135.0 (C), 128.7 (CH), 123.7 ($J = 5.9$ Hz, $J$

$\text{F}_3\text{N}_2\text{NaO}_2[\text{M+Na}]^+$, 229.0195; found, 229.0195.

Yellow solid (18.0 mg, 88%), f (70:30 petrol-EtOAc) 0.6; mp 125-128 °C (lit.,5 127-129 °C); IR (FTIR, CHCl$_3$) $\nu_{\text{max}}$ cm$^{-1}$: 3515 (NH$_2$), 3418 (NH$_2$), 1627, 1598 (NO$_2$), 1526 (NO$_2$), 1343; $^1$H NMR (400 MHz, DMSO-$d_6$) 8.42 (d, $J = 9.1$ Hz, 1H), 7.46 (d, $J = 2.5$ Hz, 1H), 7.40 (br s, 2H), 7.22 (dd, $J = 9.1$, 2.5 Hz, 1H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) 154.4 (C), 133.7 (C), 129.7 (CH), 124.1 ($J = 32.2$ Hz, CF$_3$), 123.9 (C), 114.4 (CH), 111.7 ($J = 6.6$ Hz, CH); HRMS ESI: calcd for C$_7$H$_9$F$_3$N$_2$NaO$_2[\text{M+Na}]^+$, 229.0195; found, 229.0205.
4-Methoxy-2-nitroaniline (16)
\[ \text{N}_2\text{O}_3 [\text{M+H}]^+, 169.0608; \text{found, } 166.0611; \text{calcd for } \text{C}_7\text{H}_8\text{N}_2\text{NaO}_3 [\text{M+Na}]^+, 191.0427; \text{found, } 191.0437. \]

(17)

2-Amino-5-nitrophenyl)(phenyl)methanone (18)

\[ \text{NH}_2 \text{O} \text{Me} \text{NO}_2 \]

\[ \text{NH}_2 \text{O} \text{NO}_2 \]
\[
^{10} \text{N}_{2} \text{O}_3 [M]^{+}, 242.0691; \text{found, 242.0686.}
\]

\[
Z E 2,3\text{-dihydrothiazol-5-yl) ethanol (19)}
\]

\[
\begin{aligned}
\text{O}_2 \text{N} & \quad \text{N} \quad \text{N} \quad \text{N} \quad \text{S} \\
\end{aligned}
\]

\[
\text{HO} \quad \text{O}_2 \text{N} \quad \text{N} \quad \text{N} \quad \text{N} \quad \text{S} \\
\end{aligned}
\]

\[
\begin{aligned}
\text{1} & \quad \text{N} \quad \text{N} \quad \text{N} \quad \text{S} \\
\end{aligned}
\]

\[
\begin{aligned}
\text{3} & \quad \text{N} \quad \text{N} \quad \text{N} \quad \text{S} \\
\end{aligned}
\]

\[
\text{°C. NaH (30.5 mg, 60% w/w mineral oil, 0.761 mmol) was added to the mixture in one portion. The reaction mixture was allowed to warm to room temperature, at which point a bright red colour appeared. The reaction was stirred at room temperature until complete (TLC, 5 h). The reaction mixture was then poured onto saturated ammonium chloride solution (5 mL) and the products extracted with ethyl acetate (3 x 5 mL). The combined organic extracts were dried over anhydrous magnesium sulfate, filtered and the solvents removed. The resulting residue was finally subjected to flash column chromatography (eluting with ethyl acetate) to deliver the product as a bright red solid (98.0 mg, 81%), f (EtOAc) 0.2; IR (FTIR, CHCl\textsubscript{3}) \nu_{\text{max}} \text{ cm}^{-1}: 3108 (OH), 1520 (NO\textsubscript{2}), 1428 (N=N), 1327, 1134, 1106; ^{1}H \text{ NMR (400 MHz, DMSO-} \text{d}_6) \quad 8.28 (d, J = 8.9 \text{ Hz, 2H}), 7.64 (d, J = 8.9
\]
Hz, 2H), 7.41-7.37 (m, 2H), 7.33-7.30 (m, 1H), 7.26-7.24 (m, 2H), 5.42 (s, 2H), 4.93 (t, \( = 5.7 \) Hz, 1H), 3.59 (app q, \( = 5.7 \) Hz, 2H), 2.76 (t, \( = 5.7 \) Hz, 2H), 2.16 (s, 3H); \(^{13}\)C NMR (100 MHz, DMSO-\( \_d_6 \)) 176.8 (C), 155.6 (C), 145.4 (C), 135.8 (C), 133.1 (C), 128.9 (CH), 127.7 (CH), 126.5 (CH), 125.0 (CH), 121.7 (CH), 116.5 (C), 60.5 (CH\(_2\)), 48.6 (CH\(_2\)), 29.7 (CH\(_3\)), 11.2 (CH\(_3\)); HRMS ESI: calcd for C\(_{19}\)H\(_{20}\)N\(_5\)O\(_3\)S [M+H]\(^+\), 398.1281; found, 398.1291; calcd for C\(_{19}\)H\(_{19}\)N\(_5\)NaO\(_3\)S [M+Na]\(^+\), 420.1101; found, 420.1122.
References:


   *Tetrahedron* 68

   *J. Org. Chem.* 75

   *J. Org. Chem.* 26

   *J. Org. Chem.* 1968, 33

   *J. Org. Chem.* 63

   *J. Am. Chem. Soc.* 68

   *J. Indian Chem. Soc.* 73

   *Chem. Commun.* 47
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**FW:** 138.1240

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**Nucleus:** 1H

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**Proton ESP**

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**Nucleus:** 13C

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**Spectrum Type:** STANDARD  
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**Carbon ESP**

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Formula: C₇H₅F₃N₂O₂

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Date Stamp
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Date
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Date Stamp
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NMR Spectra:

Proton (¹H) spectrum:
- Chemical Shift (ppm): 8.10, 8.09, 7.96, 7.95, 7.94, 7.93, 6.91, 6.85, 6.83
- Origin: av400
- Number of Transients: 16
- Original Points Count: 16384
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- Pulse Sequence: zg30
- Spectrum Offset (Hz): 2197.4238
- Temperature (degree C): 25.16

Carbon (¹³C) spectrum:
- Chemical Shift (ppm): 151.36, 135.88, 125.64, 124.66, 115.48, 113.54
- Origin: av400
- Number of Transients: 16
- Original Points Count: 16384
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**Chemical Shift (ppm)**

- 7.75
- 7.73
- 7.57
- 6.67
- 6.65
- 6.41
- 3.87

**Formula**: \( C_7H_8N_2O_3 \)  
**FW**: 168.1500

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**Chemical Shift (ppm)**

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- 144.50
- 135.53
- 119.67
- 110.82
- 105.57
- 55.69

**Formula**: \( C_7H_8N_2O_3 \)  
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### Carbon Spectrum

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**12/10/2012 10:28:58**

**UserID:** j_bur  **SampleID:** jb1267 dry  **SupervisorID:** moses  **Lab Phone No.:** 13540  **Slot Number:** 38

**File Name:** R:\MOSES GROUP WORK\JAMES BURNLEY\AZIDE REDUCTION PAPER\NMR SI\4-METHOXY-2-NITROANILINE\PROTON.ESP

**Acquisition Time (sec):** 3.4210  
**Comment:**

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**11 Oct 2012 12:52:32**

**UserID:** j_bur  **SampleID:** jb1267 dry  **SupervisorID:** moses  **Lab Phone No.:** 13540  **Slot Number:** 38

**File Name:** R:\MOSES GROUP WORK\JAMES BURNLEY\AZIDE REDUCTION PAPER\NMR SI\4-METHOXY-2-NITROANILINE\CARBON.ESP

**Acquisition Time (sec):** 0.6832  
**Comment:**