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Corrigendum

Corrigendum to "A new method for the reduction of nitronyl nitroxides" [Tetrahedron Lett. 44 (2003) 6397]

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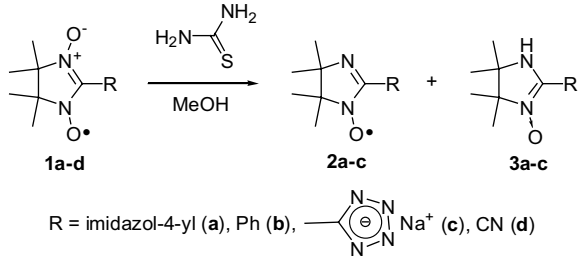
The authors regret that 'hexamethylenetetramine (HTMA)' was cited as the reducing agent in this manuscript. Thiourea was the actual reducing agent used. The error appeared due to the wrong label on the chemical box. Thus 'hexamethylenetetramine' and 'HTMA' should read 'thiourea' throughout (Scheme 1).



Scheme 1. The reduction of nitronyl nitroxides to imino nitroxides and 2-imidazoline *N*-oxides.

A revised experimental section is appended.

Experimental

2-(1*H*-Tetrazol-5-yl)-4,4,5,5-tetramethyl-4,5-dihydro-1*H*-imidazol-1-oxyl sodium salt (2c). (Typical procedure)

A solution of **1c** (1.12 g, 3.96 mmol) and thiourea (137 mg, 1.8 mmol) in MeOH (10 ml) was stirred for 10 h at room temperature. Then, PbO_2 (2.0 g, 8.4 mmol) was added and the reaction mixture was stirred for 1 h. The precipitate was filtered off and washed with EtOH. The solvent was distilled from the filtrate in vacuo, and the residue was quenched with acetone (5 ml) and filtered off. The product was crystallized from a mixture of MeOH with AcOEt. The initial colorless crystalline precipitate was removed by filtration and the filtrate was kept in a refrigerator for a few days. The resulting orange crystals were filtered off. Yield 520 mg (57%); temp of dec 285 °C. Anal. Found: C, 41.4; H, 5.2. Calcd for $C_8H_{12}N_6NaO$: C, 41.6; H, 5.2. $\mu_{eff}/\beta = 1.73$ (295 K), $a_{N1} = 9.39$ G, $a_{N2} = 4.55$ G.

2-(Imidazol-4-yl)-4,4,5,5-tetramethyl-4,5-dihydro-1*H*-imidazol-1-oxyl (2a)

Yield 57%; mp 174–175 °C; $\mu_{eff}/\beta = 1.74$ (295 K), $a_{N1} = 9.35$ G, $a_{N2} = 4.30$ G. Anal. Found: C, 57.7; H, 7.5. Calcd for $C_{10}H_{15}N_4O$: C, 57.9; H, 7.3.

4,4,5,5-Tetramethyl-4,5-dihydro-1*H*,3′*H*-[2,4′]biimidazolyl-3-oxide (3a)

The reaction time was 3 days. Yield 55%; mp 216–220 °C. Anal. Found: C, 57.5; H, 8.0; N, 26.9. Calcd for $C_{10}H_{16}N_4O$: C, 57.6; H, 7.7; N, 26.9.

Crystal data

For **2a**: $C_{10}H_{15}N_4O$, $M_w = 255.30$, triclinic, space group *P*-1, $a = 13.972(3)$, $b = 14.044(3)$, $c = 14.484(3)$ Å, $\alpha = 111.036(5)°$, $\beta = 95.644(5)°$, $\gamma = 113.217(5)°$, $V = 2340.7(9)$ Å^3, $Z = 8$, $D_c = 1.176$ g cm^{-3}, $\mu = 0.080$ mm^{-1}, reflections collected/unique = 9993/6626 ($R_{int} = 0.0851$), 752 parameters, Goof = 0.763,

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$R_1 = 0.0642$, $wR_2 = 0.1039$ ($I > 2\sigma(I)$), $R_1 = 0.2088$ $wR_2 = 0.1447$ for all data. CCDC reference number 209719. For **2c**: $C_8H_{12}N_6NaO$, $M_w = 231.23$, monoclinic, space group $P2_1/c$, $a = 10.354(2)$, $b = 8.962(1)$, $c = 12.915(2)$ Å, $\beta = 113.630(1)°$, $V = 1097.9(3)$ Å^3, $Z = 4$, $D_c = 1.399$ g cm^{-3}, $\mu = 0.133$ mm^{-1}, reflections collected/unique = 3059/1564 ($R_{int} = 0.1199$), 194 parameters, Goof = 0.870, $R_1 = 0.0619$, $wR_2 = 0.1114$ ($I > 2\sigma(I)$), $R_1 = 0.1155$, $wR_2 = 0.1287$ for all data. CCDC reference number 209720. For **3a**: $C_{10}H_{16}N_4O$, $M_w = 208.27$, orthorhombic, space group $P2_12_12_1$, $a = 7.5254(9)$, $b = 12.096(2)$, $c = 12.488(2)$ Å, $V = 1136.7(2)$ Å^3, $Z = 4$, $D_c = 1.217$ g cm^{-3}, $\mu = 0.083$ mm^{-1}, reflections collected/unique = 4924/1638 ($R_{int} = 0.1301$), 201 parameters, Goof = 1.009, $R_1 = 0.0391$, $wR_2 = 0.0989$ ($I > 2\sigma(I)$), $R_1 = 0.0404$, $wR_2 = 0.1002$ for all data. CCDC reference number 209721. The reflections data were collected on a Smart Apex Bruker AXS, Mo-Kα radiation ($\lambda = 0.71073$ Å), all structures were solved using standard direct method techniques, and refined using full-matrix least-squares based on F^2. Hydrogen atoms were localized in $\Delta\rho$ synthesis and refined isotropically with anisotropic non-hydrogen atoms.