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Synthesis and Crystal Structure of 2-Amino-1, 1-diferrocenylethanol

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Abstract: 2-Amino-1,1-diferrocenylethanol **2** was prepared by reduction of trimethylsilyl cyanohydrin ether of diferrocenyl ketone. The crystal structure of **2** was further defined by X-ray diffraction.

Keywords: 2-Aminoalcohol, reduction, trimethylsilyl cyanohydrin ether of diferrocenyl ketone.

2-Aminoalcohols are useful intermediates in organic chemistry, for example, they serve for the synthesis of various heterocycles[1], chelate complexes[2], and for the ring expansion of cycloalkanones[3]. 2-Aminoalcohols containing ferrocene as bidentate ligand may be converted into multinuclear compounds. Several methods for the preparation of 2-aminoalcohols have been developed including reduction of the trimethylsilyl cyanohydrins[4] or β-nitromethyl alcohols[5] and treatment of the epoxides with ammonia[1]. Herein, we wish to report the synthesis of 2-amino-1,1-diferrocenylethanol by reduction of trimethylsilyl cyanohydrin ether of diferrocenyl ketone[6] **1** (**Scheme 1**), and the crystal structure of compound **2** was further defined by X-ray diffraction.

Experimental

A solution of compound **1** (2 mmol) in anhydrous ethylether (20 mL) was added to a suspension of lithium aluminium hydride (16 mmol) in anhydrous ethylether (45 mL) at a rate maintaining gentle reflux. Stirring was continued under reflux for 1.5 h after the addition had been completed. Destruction of the excess of lithium aluminium hydride was completed by cautious addition of water, cooling in an ice water bath. The granular precipitate was filtrated off, washed with ethylether, and the filtrate was dried over anhydrous potassium carbonate. Removal of part solvent in *vacuo*, to give the yellow crystal suitable for X-ray analysis, yield 58.3%, mp 154-155℃. IR (KBr, cm^{-1}) v 3500-3250 (br OH), 3383(m), 1590(s), NH, 3095(m, ferrocenyl, CH). ^1H NMR ($CDCl_3$, 500MHz, δ ppm) 4.22, 4.19, 4.17, 4.13(s, together 18H, 2×ferrocenyl-*H*), 3.20(s, 2H, CH_2NH_2), 1.51(br, 3H, O*H*, NH_2). Calcd. for $C_{22}H_{23}Fe_2NO$: C, 61.58; H, 5.40; N, 3.26. Found: C, 61.32; H, 5.54; N, 3.07%.

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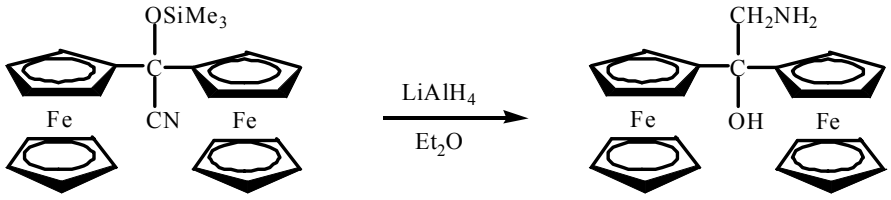
Scheme 1



1 **2**

X-ray crystallography[7]

Data of compound **2** were collected on a Bruker Smart CCD area detector with a Mo-Kα radiation (λ = 0.71073Å) at 293 K in the ϕ–ω scan mode. Total 10635 reflections were collected in the range of $2.36 \leq \theta \leq 28.29$, of which 4074 were unique. The final cycle of full-matrix least squares refinements was base on 2022 observed reflections ($I > 2\sigma(I)$) and 276 variable parameters. The structures were determined by direct methods (SHELXS-97) and successive Fourier synthesis and refined by using the SHELXL-97 program. The nonhydrogen atoms were refined anisotropically, whereas the hydrogen atoms were located from difference Fourier maps or were placed in calculated positions and refined isotropically. The molecule structure is shown in **Figure 1**. Stereo drawing of the unit cell is shown in **Figure 2**.

The crystal is orthorhombic, space group $Pna2_1$, with a = 12.1450(9) Å, b = 12.2301(9) Å, c = 12.1686(9) Å, α = β = γ = 90°, V = 1807.5(2) $Å^3$, Z = 4, Mr = 429.11, Dc = 1.577Mg/m^3, μ = 1.615mm^{-1}, F(000) = 888, R_1 = 0.0452, wR_2 = 0.0544. The stereo drawing of the unit cell (**Figure 2**) shows that compound **2** is a polymeric chain structure with intermolecular hydrogen bonding interactions between the OH proton and the N of NH_2.

Figure 1 The molecule structure of **2**

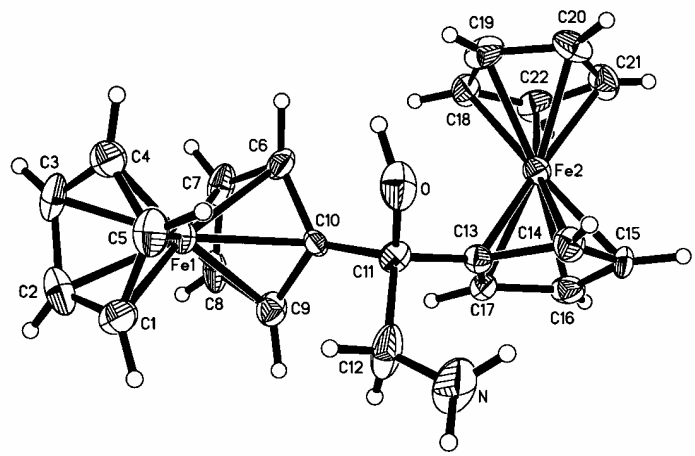
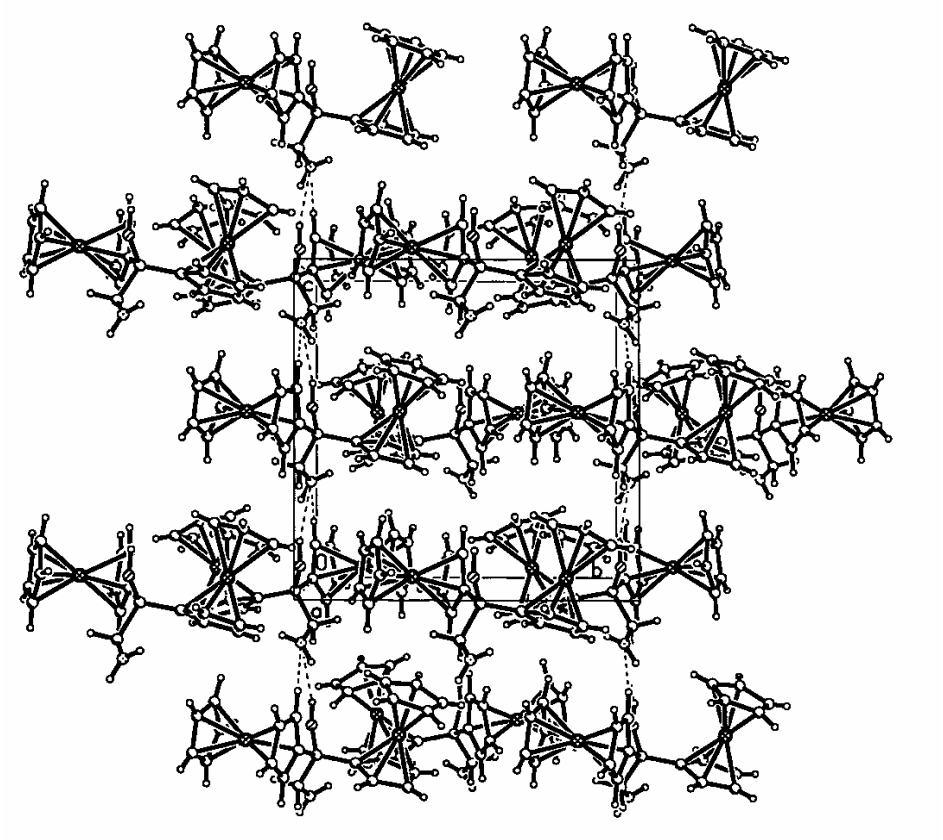


Figure 2 Stereo drawing of the unit cell of **2**



Acknowledgments

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