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Crystallographic report

Crystal structure of 1-bromo-1′-[(2*S*)-*N*-(1-hydroxy-3-methylbutane-2-yl)]-ferroceneamide

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For the unsymmetrical title compound, 1-bromo-1′-[(2*S*)-*N*-(1-hydroxy-3-methylbutane-2-yl)]-ferroceneamide, two independent molecules were found in the asymmetric unit. Copyright © 2003 John Wiley & Sons, Ltd.

KEYWORDS: unsymmetrical ferrocene derivatives; crystal structure

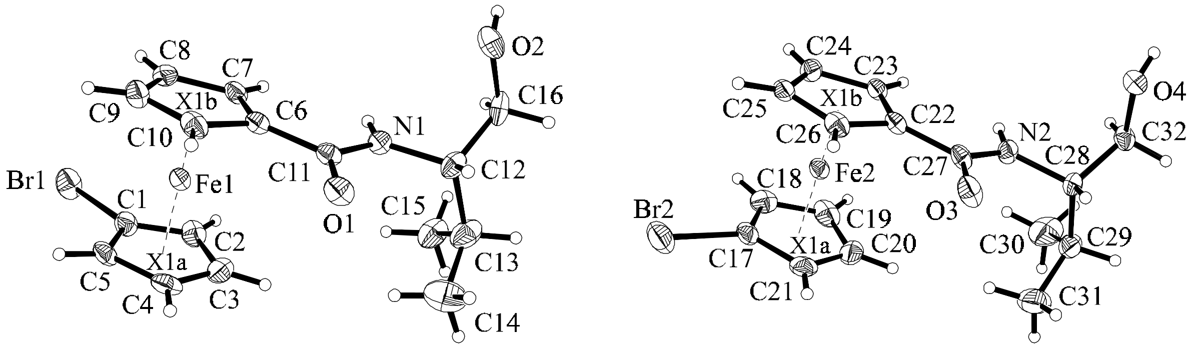

Figure 1. An ORTEP view of two independent molecules in **I**. Selected bond lengths and torsional angles: C6–C11 1.490(5), C22–C27 1.486(5), C11–O1 1.244(5), C27–O3 1.249(5), C11–N1 1.338(5), C27–N2 1.332(5), N1–C12 1.462(5), N2–C28 1.468(5), C12–C16 1.524(6), C28–C32 1.510(5), C16–O2 1.418(5), C32–O4 1.425(5), C12–C13 1.552(6), C28–C29 1.550(6) Å; N1–C12–C16–O2 57.5(4), N2–C28–C32–O4 64.9°.

COMMENT

For more than 20 years chiral ferrocenes have been used in metal-catalyzed enantioselective synthesis, which plays an important role in preparative organic chemistry.[1] The title compound, 1-bromo-1′-[(2*S*)-*N*-(1-hydroxy-3-methylbutane-2-yl)]-ferroceneamide (**I**), is a synthetic precursor for unsymmetrically substituted ferrocenyl-derivatives, a very promising ligand system for many types of asymmetric synthesis.[2] Two independent molecules found in the asymmetric unit (see Fig. 1) show similar geometric parameters. Even the torsional angles in the 1-hydroxy-3-methylbutane-2-yl fragments compare favorably and differ by not more than by 7.4°. The main difference consists of a different arrangement of the Cp rings to each other in the two independent molecules. Although the Cp rings tend to adopt the same anticlinal conformation in both molecules, the Cp· · ·Fe1· · ·Cp torsional

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twist angle at −152° in the first molecule differs substantially from this torsion angle at 134° in the second one. The Cp rings are almost parallel to each other in both molecules, with the dihedral angle being 1.4° in the first and 2.1° in the second molecule. The atoms C12 and C28 possess the same *S* absolute configuration.

EXPERIMENTAL

Single crystals of the title compound were obtained by slow crystallization of the crude product from a 1:1 mixture of diethyl ether and ethyl acetate. X-ray diffraction data were collected on a Nonius Kappa CCD diffractometer at 120 K. $C_{16}H_{20}BrFeNO_2$, $M = 394.09$, orthorhombic, $P2_12_12_1$, $a = 6.208(1)$, $b = 15.467(3)$, $c = 32.965(7)$ Å, $V = 3165.3(10)$ Å^3, $Z = 8$, 7710 unique data ($\theta_{max} = 28.3°$), $R_1 = 0.040$, $wR_2 = 0.094$, Flack parameter 0.024(9). Programs used: SHELXS-97, SHELXL-97 and ORTEP. CCDC deposition number 208 218.

Acknowledgements

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