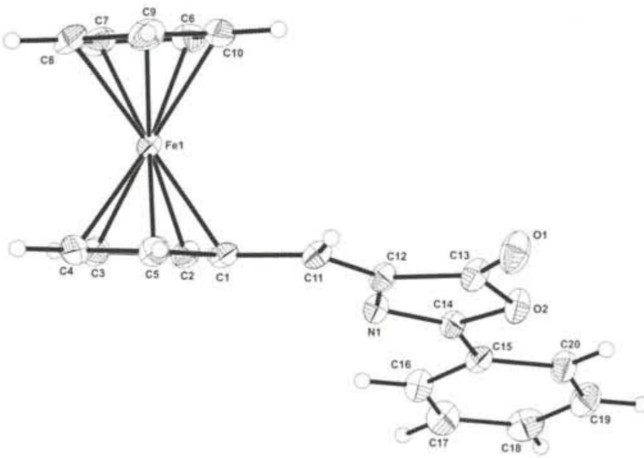
Crystal structure of 4-ferrocenylmethylene-2-phenyl-4*H*-oxazol-5-one, $C_{20}H_{15}FeNO_2$

W. Ponikwar, W. Bauer and W. Beck*

Ludwig-Maximilians-Universität München, Department Chemie, Butenandt-Str. 5-13 (Haus D), D-81377 München, Germany

Received June 8, 2000, CCDC-No. 1267/472



Abstract

$C_{20}H_{15}FeNO_2$, triclinic, $P\bar{1}$ (No. 2), $a = 7.252(3)$ Å, $b = 10.391(2)$ Å, $c = 20.662(2)$ Å, $\alpha = 82.526(6)°$, $\beta = 89.90(2)°$, $\gamma = 89.71(3)°$, $V = 1543.8$ Å^3, $Z = 4$, $R_{gt}(F) = 0.033$, $wR_{ref}(F^2) = 0.096$, $T = 293$ K.

Source of material

The compound was synthesised from hippuric acid and ferrocenylaldehyde as reported [1] and crystals were obtained from a dichloromethane/n-hexane solution.

Discussion

The title compound is a useful starting material for the synthesis of racemic [1] and optically active [2] ferrocenylalanine. In continuation of our studies on organometallic complexes of amino acids [3] we report on its crystal structure. The unit cell contains two independent molecules. In the molecule, the phenyl ring and the oxazolone ring are almost coplanar (dihedral angle (C2-C1-C11-C12) 18.43(1)°)) which allows conjugation [supported by the observed C11—C12 (134.8(4) pm) and C1—C11 (143.8(4) pm) bond lengths] and may explain the deep violet color of the compound. The C11—C12 distance (135 pm) is typical for a C=C double bond. The other bond lengths and angles are similar to those which were found for various oxazolone complexes [4-7]. In the crystal the molecules form staples where the molecules are head to tail arranged. The two cyclopentadienyl rings of the ferrocene unit show the eclipsed conformation.

Table 1. Data collection and handling.

Crystal:	dark violet plate, size 0.20 × 0.20 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.88 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5833, 4722
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3953
$N(param)_{refined}$:	442
Programs:	SHELXTL-suite [8, 9]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2A)	2*i*	0.1846	−0.2653	0.3082	0.045
H(3A)	2*i*	0.0607	−0.1707	0.1990	0.055
H(4A)	2*i*	−0.1082	0.0329	0.2123	0.058
H(5A)	2*i*	−0.0895	0.0703	0.3290	0.052
H(6A)	2*i*	0.5691	−0.0847	0.2812	0.061
H(7A)	2*i*	0.4402	−0.0174	0.1700	0.066
H(8A)	2*i*	0.2591	0.1872	0.1736	0.072
H(9A)	2*i*	0.2756	0.2439	0.2877	0.066
H(10A)	2*i*	0.4712	0.0772	0.3533	0.061
H(1)	2*i*	0.088(4)	−0.041(3)	0.434(1)	0.031(7)
H(16A)	2*i*	0.1878	−0.5901	0.4033	0.055
H(17A)	2*i*	0.2367	−0.8104	0.4154	0.066
H(18A)	2*i*	0.3451	−0.9173	0.5130	0.068
H(19A)	2*i*	0.4033	−0.8028	0.5986	0.067
H(20A)	2*i*	0.3530	−0.5818	0.5879	0.057
H(22A)	2*i*	0.5884	0.5695	0.8289	0.052
H(23A)	2*i*	0.6081	0.5323	0.7121	0.059
H(24A)	2*i*	0.4400	0.3294	0.6991	0.057
H(25A)	2*i*	0.3148	0.2356	0.8083	0.046
H(26A)	2*i*	0.0278	0.5778	0.8530	0.060
H(27A)	2*i*	0.2247	0.7431	0.7875	0.067
H(28A)	2*i*	0.2425	0.6865	0.6734	0.070
H(29A)	2*i*	0.0612	0.4828	0.6698	0.067
H(30A)	2*i*	−0.0697	0.4157	0.7811	0.060
H(2)	2*i*	0.415(4)	0.456(3)	0.934(1)	0.032(7)
H(36A)	2*i*	0.3118	−0.0900	0.9033	0.055
H(37A)	2*i*	0.2646	−0.3103	0.9152	0.066
H(38A)	2*i*	0.1545	−0.4168	1.0130	0.068
H(39A)	2*i*	0.0955	−0.3018	1.0988	0.066
H(40A)	2*i*	0.1484	−0.0816	1.0882	0.056

* Correspondence author (e-mail: wbe@cup.uni-muenchen.de)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Fe(1)	2*i*	0.21697(5)	–0.00598(4)	0.27302(2)	0.0359(2)	0.0297(3)	0.0293(2)	–0.0018(2)	0.0027(1)	0.0007(2)
Fe(2)	2*i*	0.28300(5)	0.49403(4)	0.77300(2)	0.0361(2)	0.0299(3)	0.0289(2)	0.0015(2)	–0.0016(1)	0.0006(2)
O(1)	2*i*	0.2105(4)	–0.1528(2)	0.5586(1)	0.100(2)	0.045(2)	0.036(1)	0.003(1)	0.002(1)	–0.008(1)
O(2)	2*i*	0.2506(3)	–0.3613(2)	0.54151(9)	0.066(1)	0.038(2)	0.029(1)	0.004(1)	–0.0025(8)	–0.0019(8)
O(3)	2*i*	0.2900(4)	0.3469(2)	1.0586(1)	0.101(2)	0.044(2)	0.037(1)	–0.001(1)	0.000(1)	–0.010(1)
O(4)	2*i*	0.2495(3)	0.1383(2)	1.04156(9)	0.066(1)	0.037(2)	0.030(1)	–0.004(1)	0.0025(8)	–0.0015(8)
N(1)	2*i*	0.1834(3)	–0.3510(2)	0.4342(1)	0.045(1)	0.035(2)	0.032(1)	0.002(1)	0.0010(9)	–0.002(1)
N(2)	2*i*	0.3165(3)	0.1496(2)	0.9342(1)	0.043(1)	0.035(2)	0.031(1)	–0.001(1)	0.0001(9)	–0.001(1)
C(1)	2*i*	0.0617(3)	–0.1059(3)	0.3461(1)	0.035(1)	0.031(2)	0.037(1)	–0.001(1)	0.006(1)	0.002(1)
C(2)	2*i*	0.1157(4)	–0.1893(3)	0.2995(1)	0.045(2)	0.029(2)	0.038(1)	–0.003(1)	0.003(1)	–0.002(1)
C(3)	2*i*	0.0460(4)	–0.1357(3)	0.2379(1)	0.057(2)	0.044(2)	0.037(2)	–0.015(2)	–0.002(1)	–0.006(1)
C(4)	2*i*	–0.0487(4)	–0.0208(3)	0.2454(2)	0.040(2)	0.054(2)	0.047(2)	–0.003(1)	–0.010(1)	0.007(1)
C(5)	2*i*	–0.0393(4)	0.0002(3)	0.3113(1)	0.039(2)	0.043(2)	0.047(2)	0.006(1)	0.004(1)	0.002(1)
C(6)	2*i*	0.4963(4)	–0.0117(4)	0.2689(2)	0.037(2)	0.058(2)	0.057(2)	–0.004(1)	0.008(1)	–0.006(2)
C(7)	2*i*	0.4238(4)	0.0258(4)	0.2062(2)	0.055(2)	0.067(3)	0.044(2)	–0.016(2)	0.020(1)	–0.009(2)
C(8)	2*i*	0.3219(5)	0.1407(4)	0.2084(2)	0.072(2)	0.054(3)	0.048(2)	–0.022(2)	0.002(2)	0.016(2)
C(9)	2*i*	0.3317(5)	0.1728(3)	0.2726(2)	0.070(2)	0.033(2)	0.062(2)	–0.011(2)	0.007(2)	–0.005(2)
C(10)	2*i*	0.4409(4)	0.0789(3)	0.3095(2)	0.050(2)	0.056(2)	0.046(2)	–0.017(2)	–0.001(1)	–0.006(2)
C(11)	2*i*	0.1053(4)	–0.1182(3)	0.4145(1)	0.043(2)	0.030(2)	0.035(1)	–0.000(1)	0.009(1)	–0.003(1)
C(12)	2*i*	0.1625(4)	–0.2268(3)	0.4521(1)	0.044(2)	0.034(2)	0.032(1)	0.002(1)	0.006(1)	–0.004(1)
C(13)	2*i*	0.2061(4)	–0.2320(3)	0.5215(1)	0.060(2)	0.037(2)	0.033(2)	0.001(1)	0.005(1)	–0.002(1)
C(14)	2*i*	0.2314(4)	–0.4239(3)	0.4864(1)	0.039(1)	0.041(2)	0.031(1)	–0.001(1)	0.002(1)	–0.004(1)
C(15)	2*i*	0.2655(4)	–0.5626(3)	0.4945(1)	0.040(2)	0.036(2)	0.036(1)	0.000(1)	0.005(1)	0.002(1)
C(16)	2*i*	0.2309(4)	–0.6326(3)	0.4428(1)	0.053(2)	0.044(3)	0.040(2)	0.005(1)	0.000(1)	–0.004(1)
C(17)	2*i*	0.2604(5)	–0.7640(4)	0.4501(2)	0.066(2)	0.044(3)	0.057(2)	0.002(2)	0.005(2)	–0.015(2)
C(18)	2*i*	0.3250(5)	–0.8281(4)	0.5084(2)	0.064(2)	0.034(2)	0.071(2)	0.005(2)	0.007(2)	–0.001(2)
C(19)	2*i*	0.3595(5)	–0.7597(4)	0.5593(2)	0.067(2)	0.042(3)	0.053(2)	0.005(2)	–0.002(2)	0.010(2)
C(20)	2*i*	0.3298(4)	–0.6274(3)	0.5530(1)	0.059(2)	0.044(3)	0.037(2)	0.001(2)	–0.004(1)	0.002(1)
C(21)	2*i*	0.4382(4)	0.3937(3)	0.8459(1)	0.036(1)	0.030(2)	0.036(1)	0.002(1)	–0.005(1)	0.003(1)
C(22)	2*i*	0.5387(4)	0.4996(3)	0.8111(1)	0.035(1)	0.045(2)	0.048(2)	–0.008(1)	–0.004(1)	0.003(1)
C(23)	2*i*	0.5487(4)	0.4789(3)	0.7453(2)	0.045(2)	0.054(2)	0.046(2)	0.001(2)	0.011(1)	0.009(2)
C(24)	2*i*	0.4542(4)	0.3644(3)	0.7379(1)	0.061(2)	0.045(2)	0.036(2)	0.013(2)	0.004(1)	–0.003(1)
C(25)	2*i*	0.3839(4)	0.3110(3)	0.7996(1)	0.047(2)	0.029(2)	0.038(1)	0.005(1)	–0.003(1)	–0.002(1)
C(26)	2*i*	0.0586(4)	0.5791(3)	0.8093(2)	0.049(2)	0.053(2)	0.049(2)	0.015(2)	0.002(1)	–0.007(2)
C(27)	2*i*	0.1687(5)	0.6726(3)	0.7723(2)	0.068(2)	0.033(2)	0.066(2)	0.012(2)	–0.007(2)	–0.005(2)
C(28)	2*i*	0.1794(5)	0.6405(4)	0.7081(2)	0.072(2)	0.051(3)	0.047(2)	0.018(2)	–0.004(2)	0.016(2)
C(29)	2*i*	0.0771(5)	0.5259(4)	0.7061(2)	0.058(2)	0.069(3)	0.041(2)	0.019(2)	–0.018(1)	–0.008(2)
C(30)	2*i*	0.0031(4)	0.4882(3)	0.7688(2)	0.037(2)	0.053(2)	0.059(2)	0.007(1)	–0.006(1)	–0.007(2)
C(31)	2*i*	0.3950(4)	0.3816(3)	0.9144(1)	0.043(2)	0.030(2)	0.036(1)	0.001(1)	–0.009(1)	–0.004(1)
C(32)	2*i*	0.3377(4)	0.2736(3)	0.9519(1)	0.044(2)	0.034(2)	0.032(1)	–0.001(1)	–0.005(1)	–0.002(1)
C(33)	2*i*	0.2938(4)	0.2682(3)	1.0216(1)	0.057(2)	0.037(2)	0.035(2)	0.002(1)	–0.004(1)	–0.004(1)
C(34)	2*i*	0.2692(4)	0.0764(3)	0.9863(1)	0.043(2)	0.040(2)	0.029(1)	–0.001(1)	–0.001(1)	–0.004(1)
C(35)	2*i*	0.2350(4)	–0.0625(3)	0.9946(1)	0.041(2)	0.036(2)	0.036(1)	–0.002(1)	–0.002(1)	0.000(1)
C(36)	2*i*	0.2691(4)	–0.1324(3)	0.9428(1)	0.056(2)	0.041(3)	0.040(2)	–0.004(1)	0.002(1)	–0.002(1)
C(37)	2*i*	0.2402(5)	–0.2638(4)	0.9499(2)	0.067(2)	0.044(3)	0.057(2)	–0.002(2)	–0.004(2)	–0.012(2)
C(38)	2*i*	0.1748(5)	–0.3277(3)	1.0084(2)	0.064(2)	0.031(2)	0.074(2)	–0.002(2)	–0.009(2)	0.000(2)
C(39)	2*i*	0.1400(5)	–0.2591(4)	1.0596(2)	0.067(2)	0.043(3)	0.051(2)	–0.005(2)	0.006(2)	0.009(2)
C(40)	2*i*	0.1708(4)	–0.1273(3)	1.0531(1)	0.059(2)	0.040(3)	0.040(2)	–0.000(1)	0.004(1)	0.001(1)

Acknowledgments. We thank the Fonds der Chemischen Industrie and Deutsche Forschungsgemeinschaft for generous support.

References

1. Schlögl, K.: Über Ferrocen-Aminosäuren und verwandte Verbindungen. Monatsh. Chem. **88** (1957) 601
2. Brunner, H.; König, W.; Nuber, B.: Enantioselective catalysis. 83. Synthesis of optically active ferrocenylalanine. Tetrahedron: Asymmetry **4** (1993) 699-707.
3. Severin, K.; Bergs, R.; Beck, W.: Bioorganometallic Chemistry-Transition Metal Complexes with α-Amino Acids and Peptides. Angew. Chem. Int. Ed. Engl. **37** (1998) 1634-1654.
4. Bauer, W.; Polborn, K.; Beck, W.: Metal Complexes of biologically important Ligands, CXIV Ferrocenyl-oxazolones as N and C donors in Pd(II), Pt(II) and Ir(II) complexes and ferrocenyl-dipeptides. J. Organomet. Chem. **579** (1999) 269.
5. Prem, M.; Bauer, W.; Polborn, K.; Beck, W.: Metallkomplexe von biologisch wichtigen Liganden, CVI Metallkomplexe von Donor-substituierten Oxazolin-5-onen sowie von Bis(oxazolin-5-onen). Z. Naturforsch. **53b** (1998) 965.
6. Bauer, W.; Prem, M.; Polborn, K.; Sünkel, K.; Steglich, W.; Beck, W.: Metal Complexes of biologically important Ligands CII, Organometallic Complexes of Iridium, Palladium, Chromium and Iron from 2-Phenyl-5(4H)-oxazolones - Organometallic Labeled Dipeptides. Eur. J. Inorg. Chem. (1998) 485.
7. Schreiner, B.; Prem, M.; Bauer, W.; Polborn, K.; Beck, W.: Metallkomplexe mit biologisch wichtigen Liganden, XCVI Palladium(II), Platin(II)- und Rhodium(I)- Komplexe mit (Phenyl)-C-H...Metall-Wechselwirkung von Diphenylmethylenglycinestern und von 2-Phenyloxazolinonen. Z. Naturforsch. **52b** (1997) 1199.
8. Bruker.X S. Structure Solution Program, Version 5.10. Bruker AXS, Inc., Madison, Wisconsin, USA.
9. Bruker. SHELXTL. Structure Determination Program, Version 5.10. Bruker AXS, Inc., Madison, Wisconsin, USA 1997.