Journal of Organometallic Chemistry, 288 (1985) 189–195
Elsevier Sequoia S.A., Lausanne – Printed in The Netherlands

PREPARATION AND X-RAY CRYSTAL STRUCTURE OF 2-(DIPHENYLOXYMETHYL)AZAFERROCENE

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(Received November 27th, 1984)

Summary

Metallation of azaferrocene by n-butyllithium in ether (both in the presence and absence of TMEDA) with subsequent treatment of the reaction mixture with benzophenone gives 2-(diphenyloxymethyl)azaferrocene as the sole product. Its structure was established by an X-ray diffraction study.

Introduction

Recently we discovered [1] that azaferrocene is readily metallated by n-butyllithium in THF at −50°C. Lithium replaces either one hydrogen atom of the cyclopentadienyl ring or an α-hydrogen atom of the pyrrolyl ring. Thus a mixture of 1′- and 2-lithium azaferrocenes is formed. If the reaction time is increased, heteroannular 2,1′-dilithium azaferrocene is obtained in addition to both former compounds. The interaction of lithium azaferrocenes with electrophilic reagents (MeI, Me_3SiCl) yields a mixture of the corresponding substituted azaferrocenes, but separation of these products is difficult. Therefore we decided to look for the conditions of selective azaferrocene metallation. The results are reported below.

Results and Discussion

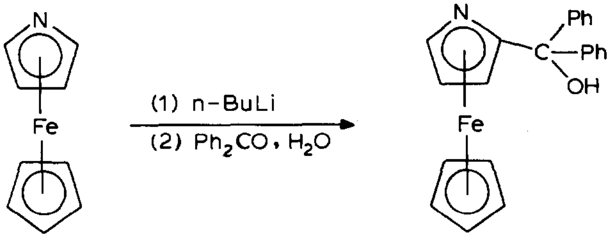
As the reactivities of the pyrrolyl and cyclopentadienyl ligands of azaferrocene towards metallation in THF are very similar [1], we considered that it was not worthwhile to try to prepare only one monosubstituted azaferrocene by varying the conditions of the above-mentioned reaction. Therefore we looked for a route of dimetallation of azaferrocene.

Ferrocene is metallated by n-butyllithium under more drastic conditions than azaferrocene [2,3]. Nevertheless, a method of ferrocene heteroannular dimetallation by n-BuLi in hexane at room temperature in the presence of tetramethylethylenediamine (TMEDA) has been well developed [4]. Taking into consideration the fact

0022-328X/85/$03.30

that azaferrocene is readily metallated, it could be expected that under the same conditions azaferrocene would be dimetallated to give heteroannular dilithium azaferrocene. However, in contrast to ferrocene, treatment of the reaction mixture with benzophenone and water yielded the original azaferrocene instead of the expected 2,1'-bis(diphenyloxymethyl)azaferrocene.

In ether the reaction proceeds quite differently. Surprisingly, metallation of azaferrocene by n-BuLi in the presence of TMEDA at −50°C in ether and subsequent interaction with benzophenone gave 2-(diphenyloxymethyl)azaferrocene (I) as the sole product. Furthermore, under the same conditions, even without TMEDA, only I was formed.



(I)

There is no reason to assume that only a change of the solvent (ether instead of THF) will result in selective metallation of the pyrrolyl ligand of azaferrocene, because in THF the reactivity of both ligands is almost the same (see above) [1]. In fact, when azaferrocene was metallated under the same conditions (n-BuLi + TMEDA in ether, −50°C), but with subsequent treatment of the reaction mixture with MeI, a mixture of 2- and 1'-methylazaferrocenes was obtained in an almost quantitative yield. Hence, although we succeeded in finding the conditions under which metallation with subsequent electrophilic attack gives only one monosubstituted azaferrocene, the selectivity of the reaction is probably caused by the nature of the electrophilic agent, i.e. benzophenone.

I is a dark orange, crystalline compound, rather stable in the solid state, unsoluble in hexane, and soluble in benzene, chlorinated solvents and ether. The IR spectrum of I (in CH_2Cl_2) exhibits the stretching band of a hydroxyl group at 3545 cm^{-1}. The ^1H NMR spectrum of I ($CDCl_3$) contains the following signals (δ, ppm): 4.17 (s, 5H, Cp), 4.36 (s, 1H, OH), 4.42 (d, $J_{3,4}$ 2.4 Hz, 1H, H_3), 4.67 (dd, $J_{4,3}$ 2.4 Hz, $J_{4,5}$ 0.6 Hz, 1H, H_4), 5.49 (s, 1H, H_5) and 6.99–7.67 (m, 10H, Ph).

The molecular structure of I was established by an X-ray diffraction study, which is the first structural investigation of an azaferrocene system.

The crystal structure of I is built up of four symmetrically independent molecules (**A**, **B**, **C** and **D**) with very similar geometries (Tables 1 and 2). Intermolecular O–H ... N hydrogen bonds (Tables 1 and 2) link the molecules pairwise (**A** with **C**, **B** with **D**) into dimers, as shown in Fig. 1.

The iron atom has a sandwich-like coordination with the planar cyclopentadienyl and pyrrolyl rings. The rings are almost parallel (interplanar angles: 1.3° in **A**, 1.2° in **B**, 3.5° in **C** and 2.9° in **D**) and equi-distant from the Fe atom (the mean Fe–ring plane distance is 1.65 Å). The geometry of the pyrrolyl ligand in I is essentially the same as those in various azacymantrene derivatives [5,6]. All the C–C and C–N bond distances (mean values 1.41(1) and 1.40(1) Å, respectively) are the same within

TABLE 1

SELECTED BOND DISTANCES (Å)

Molecule	**A**	**B**	**C**	**D**
Fe–N	2.032(5)	2.017(5)	2.035(6)	2.025(5)
Fe–C(1)	2.027(6)	2.030(7)	2.023(7)	2.009(7)
Fe–C(2)	2.067(7)	2.034(8)	2.071(7)	2.072(7)
Fe–C(3)	2.065(8)	2.061(8)	2.058(7)	2.060(7)
Fe–C(4)	2.020(7)	2.013(7)	2.011(8)	2.013(7)
Fe–C(6)	2.04(1)	2.02(1)	2.024(8)	2.01(1)
Fe–C(7)	2.03(1)	2.01(1)	2.03(1)	2.00(1)
Fe–C(8)	2.03(1)	2.01(1)	2.04(1)	2.00(1)
Fe–C(9)	2.00(1)	1.97(1)	2.06(1)	2.03(1)
Fe–C(10)	2.00(1)	2.02(1)	2.06(1)	2.04(1)
N–C(1)	1.40(1)	1.40(1)	1.40(1)	1.41(1)
C(1)–C(2)	1.41(1)	1.40(1)	1.41(1)	1.43(1)
C(2)–C(3)	1.42(1)	1.42(1)	1.41(1)	1.41(1)
C(3)–C(4)	1.42(1)	1.40(1)	1.43(1)	1.40(1)
C(4)–N	1.39(1)	1.38(1)	1.40(1)	1.41(1)
C(6)–C(7)	1.47(2)	1.35(2)	1.36(1)	1.39(2)
C(7)–C(8)	1.30(2)	1.33(2)	1.45(1)	1.36(1)
C(8)–C(9)	1.26(2)	1.30(2)	1.41(2)	1.36(2)
C(9)–C(10)	1.34(2)	1.38(2)	1.41(1)	1.36(1)
C(10)–C(6)	1.42(2)	1.36(2)	1.37(1)	1.36(1)
C(1)–C(5)	1.53(1)	1.55(1)	1.53(1)	1.54(1)
C(5)–O	1.444(8)	1.423(8)	1.419(8)	1.421(8)
C(5)–C(11)	1.53(1)	1.54(1)	1.53(1)	1.54(1)
C(5)–C(21)	1.52(1)	1.54(1)	1.54(1)	1.55(1)
O–H(0)	0.85(6)	0.74(7)	1.29(8)	0.82(9)
H(0)...N [a]	2.02(6)	2.07(7)	1.56(8)	1.98(9)
O...N [a]	2.837(7)	2.810(7)	2.798(7)	2.783(7)

[a] Hydrogen bonds between molecules **A** and **C** or **B** and **D**.

experimental accuracy, indicating an aromatic π-conjugation over the pyrrolyl ring. Similar conjugation is found in the free pyrrole molecule, in which the bond lengths (determined by microwave spectroscopy) are C–N 1.371, C=C 1.383, and C–C 1.429 Å [7].

Thus, the π-pyrrolyl ligand is coordinated by the metal atom in a similar way to the π-cyclopentadienyl ligand. The sandwich moiety of I is isostructural with ferrocene [8]; the azacymantrene system is also isostructural with cymantrene [5,6]. The lone electron pair of the nitrogen atom remains localized, viz. it participates in the n-bonding with another metal atom in azacymantrene complexes and in the hydrogen bond in I.

In each of the four independent molecules of I the CPh_2OH group and the pyrrolyl ring plane have the same relative orientation, corresponding to a staggered conformation around the C(1)–C(5) bond (Table 3). The OH group is situated on the same side from the pyrrolyl plane as the Fe atom. The relative orientation of the 5-membered rings in all four independent molecules of I is intermediate between eclipsed and staggered. However, the rotation angles φ (determined as the C(1)–pyrrolyl centroid–Cp centroid–C(7) torsion angles, Fig. 1) are somewhat different (Table 3). In molecules **B**, **C** and **D**, the C(7) atom is turned away from C(1) towards

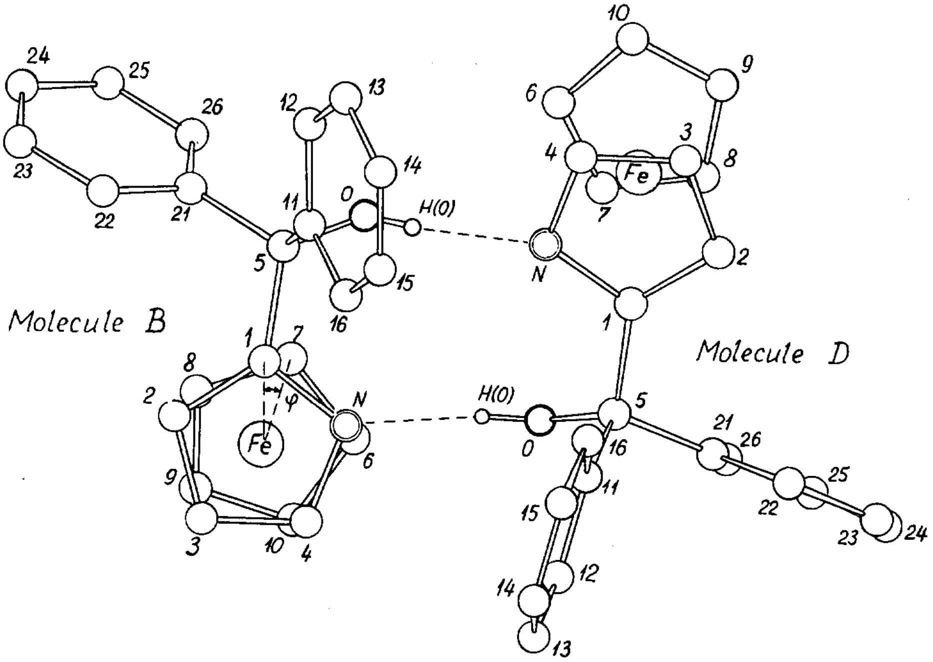


Fig. 1. One of the dimers (molecules **B** and **D**) in the structure of I. Hydrogen atoms (except hydroxyl ones) have been omitted.

TABLE 2

SELECTED BOND ANGLES (°)

Molecule	A	B	C	D
C(1)NC(4)	106.1(5)	105.0(5)	105.1(5)	104.8(5)
NC(1)C(2)	110.8(6)	110.9(6)	110.0(6)	111.0(6)
NC(1)C(5)	118.9(6)	118.9(6)	118.1(6)	117.6(6)
C(2)C(1)C(5)	130.0(6)	130.2(6)	130.4(6)	130.3(6)
C(1)C(2)C(3)	106.1(6)	106.1(7)	108.1(6)	105.5(6)
C(2)C(3)C(4)	106.8(6)	106.4(7)	105.2(6)	108.3(7)
C(3)C(4)N	110.2(6)	111.4(6)	111.5(6)	110.4(6)
C(7)C(6)C(10)	102(1)	105(1)	111(1)	107(1)
C(6)C(7)C(8)	106(1)	110(1)	106(1)	107(1)
C(7)C(8)C(9)	113(1)	109(1)	107(1)	109(1)
C(8)C(9)C(10)	111(1)	107(1)	108(1)	107(1)
C(9)C(10)C(6)	108(1)	108(1)	108(1)	109.5(9)
C(1)C(5)O	109.7(5)	109.9(5)	110.2(5)	110.6(5)
C(1)C(5)C(11)	112.7(5)	108.8(5)	108.6(5)	109.5(5)
C(1)C(5)C(21)	109.3(5)	109.6(5)	109.5(5)	109.1(5)
OC(5)C(11)	108.3(5)	108.9(5)	110.4(5)	110.6(5)
OC(5)C(21)	107.3(5)	107.7(5)	107.4(5)	107.6(5)
C(11)C(5)C(21)	109.5(5)	111.9(5)	110.8(5)	109.5(5)
C(5)C(11)C(12)	119.8(6)	117.1(6)	117.7(6)	120.1(6)
C(5)C(11)C(16)	120.5(6)	125.0(6)	123.4(6)	123.6(6)
C(12)C(11)C(16)	119.4(6)	117.3(6)	118.9(7)	116.2(6)
C(5)C(21)C(22)	120.7(6)	120.1(6)	121.2(6)	120.3(6)
C(5)C(21)C(26)	121.5(6)	119.8(6)	120.9(6)	121.2(6)
C(22)C(21)C(26)	117.8(6)	120.0(7)	117.9(6)	118.6(7)
C(5)OH(0)	98(4)	112(6)	103(3)	120(6)
OH(0) ... N [a]	160(6)	173(8)	159(8)	170(8)

[a] See footnote to Table 1.

TABLE 3

TORSION ANGLES (°) [a]

Molecule	A	B	C	D
NC(1)C(5)O	−42.9(6)	41.8(6)	−46.7(6)	45.9(6)
NC(1)C(5)C(11)	77.8(8)	−77.4(8)	74.4(8)	−76.2(8)
NC(1)C(5)C(21)	−160.3(9)	160.0(9)	−164.6(9)	164.0(9)
C(1)C(5)C(11)C(12)	165(1)	−168(1)	−167(1)	173(1)
C(1)C(5)C(21)C(22)	−69.0(9)	70.5(9)	−89.1(9)	79.1(9)
φ	17(1)	13(1)	−21(1)	27(1)

[a] The same conformation of the *S* (molecules **A** and **C**) and *R* (molecules **B** and **D**) enantiomers corresponds to torsion angles of opposite signs.

N, while in molecule **A** it is in the opposite direction, viz. towards C(2).

Due to the unequivalence of the two positions in the pyrrolyl ring (i.e. N and C(1), carrying the substituent), molecule I is chiral. Both molecules of each dimer have the same absolute configuration, viz. S for the reference **A** and **C** molecules, R for **B** and **D**, according to the nomenclature recommended in ref. 9.

Experimental

Metallation and isolation of I were performed in an argon atmosphere. The solvents were carefully dried and distilled in an argon stream.

Azaferrocene was prepared by the method reported previously [10].

0.1 g (0.53 mmol) of azaferrocene in 15 ml of ether was added dropwise and under stirring to a solution of 2.5 ml (1.50 mmol) of n-BuLi in 2.5 ml of ether at −50°C. The reaction mixture was stirred at −50°C for 1 h and at −20°C for 2 h, then it was allowed to warm to room temperature. 30 ml of water was added, the organic layer was removed, and the water layer was extracted several times with ether. The ether extracts were dried over $MgSO_4$ and the solvent was removed in vacuo. The residue (a dark-red, viscous oil) was dissolved in benzene and chromatographed on a silica gel column. I was eluted with a petroleum ether/ether mixture (1/1), and 0.02 g of the original azaferrocene was eluted with ether. I was recrystallized from a hexane/ether mixture (1/1), yielding 0.08 g of dark-orange crystals, m.p. 121–123°C. The yield was 40% with respect to the total and 50% with respect to the reacted amount of azaferrocene. Found: C, 71.34; H, 5.26; N, 3.64; Fe, 15.46. $C_{22}H_{19}NOFe$ calcd.: C, 71.56; H, 5.19; N, 3.79; Fe, 15.13%.

Metallation was performed similarly in the presence of TMEDA. From 0.1 g of azaferrocene, 0.08 g of I was obtained (40% with respect to the total and 70% with respect to the reacted amount of azaferrocene).

The IR spectra were recorded on a UR-20 spectrophotometer, ^1H NMR spectra on a Bruker WP-200-SY spectrometer (200.13 MHz).

The X-ray diffraction experiment was performed with a four-circle Hilger–Watts autodiffractometer (using graphite-monochromated Mo-K_α radiation) at room temperature. The calculations were carried out with an Eclipse S/200 computer using INEXTL programs [11]. Crystal data: $C_{22}H_{19}FeNO$, monoclinic, a 11.294(1), b 33.961(2), c 18.314(2) Å, β 91.371(8)°, V 7023(1) Å^3, $Z = 16$, d_{calcd} 1.40 g cm^{-3}, space group $P2_1/n$. The intensities of 5210 independent reflections with $I \geqslant 2\sigma$

TABLE 4

ATOMIC COORDINATES (×10^4; for Fe, ×10^5; for H, ×10^3) AND THERMAL FACTORS ($B_{\text{iso}}^{\text{eq}} = 1/3\sum_{ij} B_{ij}a_i^* a_j^*(a_i a_j)$, for H B_{iso}, Å^2)

Atom	x	y	z	B	x	y	z	B
Molecule **A**					*Molecule* **C**			
Fe	42234(9)	44782(3)	11270(6)	3.29(3)	77110(10)	33172(3)	28708(6)	3.79(3)
O	5203(4)	4088(1)	2805(2)	3.3(1)	7118(4)	3875(1)	1235(2)	3.5(1)
N	5861(5)	4556(2)	1588(3)	3.1(2)	7658(5)	3914(2)	2771(3)	3.2(2)
C(1)	4992(5)	4679(2)	2067(3)	2.7(2)	8436(6)	3750(2)	2268(4)	3.0(2)
C(2)	4302(6)	4984(2)	1754(4)	3.5(2)	9360(6)	3547(2)	2642(4)	3.4(2)
C(3)	4774(7)	5056(2)	1053(4)	3.9(2)	9153(6)	3569(2)	3396(4)	4.2(3)
C(4)	5723(6)	4788(2)	967(4)	3.7(2)	8110(7)	3802(2)	3456(4)	4.2(3)
C(5)	4989(6)	4507(2)	2836(3)	2.7(2)	8324(6)	3881(2)	1473(4)	2.8(2)
C(6)	3920(10)	3889(3)	1010(3)	11.2(5)	6019(7)	3128(3)	2943(6)	6.6(3)
C(7)	2924(10)	4087(3)	1354(4)	8.5(4)	6435(9)	3032(3)	2273(5)	7.6(4)
C(8)	2514(8)	4339(3)	875(5)	7.1(4)	7449(10)	2778(3)	2402(6)	7.9(4)
C(9)	3100(10)	4350(3)	300(5)	8.1(4)	7583(9)	2734(3)	3162(6)	7.7(4)
C(10)	3973(9)	4085(3)	327(5)	9.8(4)	6676(9)	2953(3)	3491(5)	7.6(4)
C(11)	5933(5)	4692(2)	3344(4)	2.6(2)	8830(6)	4296(2)	1410(4)	3.0(2)
C(12)	5879(6)	4641(2)	4055(4)	4.3(3)	8596(6)	4500(2)	776(4)	4.0(2)
C(13)	6768(7)	4773(3)	4528(4)	5.0(3)	9045(7)	4869(3)	673(5)	5.3(3)
C(14)	7725(7)	4957(3)	4251(4)	4.9(3)	9752(7)	5046(2)	1213(5)	5.5(3)
C(15)	7814(7)	5010(3)	3513(4)	4.8(3)	9988(7)	4846(2)	1852(4)	5.2(3)
C(16)	6915(6)	4881(2)	3056(4)	3.7(3)	9525(6)	4473(2)	1954(4)	4.1(2)
C(21)	3773(6)	4565(2)	3157(3)	2.9(2)	9002(6)	3591(2)	987(3)	3.1(2)
C(22)	3372(6)	4943(2)	3340(4)	4.1(3)	10186(6)	3646(2)	845(4)	4.4(3)
C(23)	2252(7)	5002(3)	3597(4)	5.6(3)	10774(7)	3391(3)	386(4)	4.8(3)
C(24)	1493(7)	4693(3)	3701(4)	6.2(3)	10194(7)	3080(2)	74(4)	4.3(3)
C(25)	1856(7)	4317(3)	3534(4)	5.8(3)	9037(7)	3016(2)	219(4)	4.6(3)
C(26)	3001(6)	4246(2)	3262(4)	4.1(3)	8435(6)	3272(2)	668(4)	3.9(2)
H(0)	596(5)	410(2)	283(3)	3(2)	675(7)	422(2)	142(4)	8(2)
Molecule **B**					*Molecule* **D**			
Fe	−7420(9)	13555(3)	8093(6)	3.66(3)	26762(9)	24957(3)	26107(6)	3.39(3)
O	228(4)	1751(1)	2479(2)	3.3(1)	2126(4)	1959(1)	956(2)	3.5(1)
N	882(5)	1275(2)	1267(3)	3.3(2)	2675(5)	1903(2)	2492(3)	3.2(2)
C(1)	17(6)	1154(2)	1754(4)	2.9(2)	3449(6)	2083(2)	1998(4)	2.8(2)
C(2)	−713(7)	862(2)	1440(4)	4.0(2)	4360(6)	2297(2)	2377(4)	3.9(2)
C(3)	−227(8)	774(2)	750(4)	5.1(3)	4134(6)	2247(2)	3127(4)	4.2(2)
C(4)	728(6)	1032(2)	665(4)	4.0(2)	3119(6)	2011(2)	3189(4)	3.8(2)
C(5)	9(6)	1339(2)	2525(4)	2.9(2)	3334(6)	1963(2)	1191(4)	3.1(2)
C(6)	−781(9)	1910(3)	417(7)	10.4(5)	1009(8)	2676(3)	2780(7)	8.1(4)
C(7)	−1547(9)	1877(3)	966(5)	8.5(4)	1340(9)	2761(3)	2068(5)	7.8(4)
C(8)	−2356(8)	1603(4)	803(7)	10.2(5)	2292(9)	3008(3)	2120(5)	7.2(3)
C(9)	−2137(11)	1452(4)	168(7)	11.7(5)	2557(8)	3079(3)	2838(6)	8.0(4)
C(10)	−1193(11)	1657(4)	−107(5)	10.8(5)	1752(9)	2880(3)	3236(5)	7.5(4)
C(11)	1000(6)	1147(2)	2999(4)	2.9(2)	3896(6)	1556(2)	1086(4)	3.1(2)
C(12)	987(6)	1220(2)	3756(4)	4.0(2)	3781(7)	1364(2)	413(4)	4.4(3)
C(13)	1926(7)	1104(3)	4201(4)	5.0(3)	4278(8)	1004(2)	289(4)	5.2(3)
C(14)	2885(7)	910(3)	3900(5)	5.4(3)	4894(8)	808(2)	827(4)	5.1(3)
C(15)	2871(6)	846(2)	3160(4)	3.7(2)	5022(7)	985(2)	1495(5)	5.3(3)
C(16)	1978(6)	957(2)	2732(4)	3.9(2)	4536(7)	1355(2)	1637(4)	4.4(3)
C(21)	−1220(6)	1283(2)	2858(3)	3.1(2)	3986(6)	2271(2)	720(4)	3.0(2)
C(22)	−1586(6)	912(2)	3071(4)	3.9(2)	5188(7)	2260(2)	670(4)	4.8(3)
C(23)	−2701(7)	864(3)	3351(4)	5.8(3)	5775(7)	2534(3)	251(5)	6.1(3)
C(24)	−3431(7)	1178(3)	3435(5)	7.0(4)	5119(9)	2816(3)	−122(5)	6.6(3)

TABLE 4 (continued)

Atom	*x*	*y*	*z*	*B*	*x*	*y*	*z*	*B*
Molecule **B**					*Molecule* **D**			
C(25)	−3059(8)	1543(3)	3220(5)	6.3(3)	3940(8)	2837(3)	−50(5)	6.5(3)
C(26)	−1991(6)	1607(2)	2914(4)	4.7(3)	3370(7)	2565(2)	353(4)	4.6(3)
H(0)	87(6)	179(2)	245(4)	5(2)	170(7)	178(3)	107(4)	8(3)

Tables of the anisotropic thermal factors and coordinates of other hydrogen atoms can be obtained from the authors on request.

were measured by the $\theta/2\theta$ scan mode ($\theta \leqslant 30°$). The structure was solved by the direct method and refined by block-diagonal least squares. All non-hydrogen atoms were refined with anisotropic thermal factors, and hydroxyl hydrogens (located from the difference synthesis) with isotropic ones; other hydrogens were included as fixed contributions in calculated positions. The weighting scheme $w = \sigma_F^{-2}$ was used. The refinement converged to $R = 0.050$ and $R_w = 0.042$. The positional and thermal atomic parameters are listed in Table 4.

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