Crystal structure of 17α-ethynylferrocenyl-17β-hydroxyestr-4-en-3-one, $C_{30}H_{34}FeO_2$

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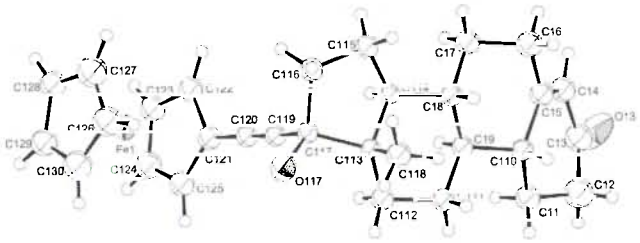
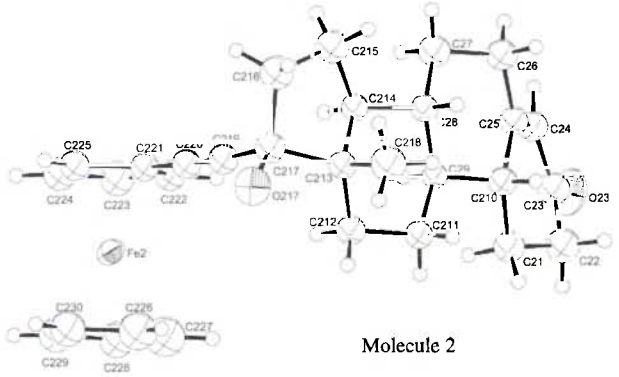


Molecule 1



Molecule 2

Abstract

$C_{30}H_{34}FeO_2$, monoclinic, $P12_11$ (No. 4), a = 8.65(1) Å, b = 9.49(1) Å, c = 30.22(1) Å, β = 93.53(9)°, V = 2476.0 Å^3, Z = 4, $R_{gt}(F)$ = 0.074, $wR(F)$ = 0.076, T = 293 K.

Source of material

17α-ethynylferrocenyl-17β-hydroxyestr-4-en-3-one was prepared from C3-protected 19-nortestosterone and ethynylferrocene. A solution of estr-4-ene-3,17-dione 3-ethylene mercaptole in tetrahydrofuran was added to an in situ preparation of lithioethynylferrocene, also in tetrahydrofuran. After work-up, the crude product was stirred with mercury(II) chloride and cadmuim carbonate to effect the removal of the C3-protecting group resulting in the isolation of the title compound, mp 461 K - 462 K.

Discussion

The 17α-ethynylferrocenyl substituted androgen was prepared as a potential marker for the androgen receptor which is strongly implicated in the progression of prostate cancer. Two independent molecules comprise the asymmetric unit and these differ in the relative disposition of the Fc groups with respect to the rest of the molecule as shown in the Figure. The molecules are linked in the crystal structure via H-bonding interactions between translationally related molecules such that d(O13···O117^i) is 2.759(7) Å and d(O23···O217^{ii}) is 2.720(7) Å; H atoms of the hydroxy groups were not located and symmetry operation i: x-1, y-1, z and ii: 1+x, 1+y, z. The crystal structure may be described as being comprised of sandwiches of steroid···Fc···steroid stacked along the c-direction.

Table 1. Data collection and handling.

Crystal:	red-orange block, size 0.4 × 0.4 × 0.52 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	6.33 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC6R, ω/2θ
$2\theta_{max}$:	55.28°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6481, 6092
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 3\ \sigma(I_{obs})$, 3391
$N(param)_{refined}$:	294
Programs:	teXsan [1], SIR92 [2], DIFABS [3]

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

Atom	Site	x	y	z	U_{iso}
C(11)	2a	-0.6808(6)	-0.2995(6)	-0.1092(2)	0.049(1)
C(12)	2a	-0.7909(8)	-0.4242(8)	-0.1021(3)	0.075(2)
C(13)	2a	-0.6980(8)	-0.5564(7)	-0.0948(3)	0.065(2)
C(14)	2a	-0.5557(7)	-0.5420(6)	-0.0660(2)	0.054(1)
C(15)	2a	-0.4943(6)	-0.4180(6)	-0.0520(2)	0.048(1)
C(16)	2a	-0.3576(7)	-0.4129(6)	-0.0215(2)	0.055(1)
C(17)	2a	-0.2255(6)	-0.3254(6)	-0.0409(2)	0.049(1)
C(18)	2a	-0.2825(6)	-0.1801(5)	-0.0543(2)	0.040(1)
C(19)	2a	-0.4210(6)	-0.1857(5)	-0.0867(2)	0.037(1)
C(21)	2a	-0.1675(7)	0.1917(7)	-0.3835(2)	0.057(1)
C(22)	2a	-0.0497(7)	0.3029(7)	-0.3910(2)	0.070(2)
C(23)	2a	0.1026(8)	0.2378(7)	-0.4013(2)	0.065(2)
C(24)	2a	0.0934(7)	0.1173(6)	-0.4308(2)	0.056(1)
C(25)	2a	-0.0393(6)	0.0534(6)	-0.4434(2)	0.048(1)
C(26)	2a	-0.0469(6)	-0.0572(6)	-0.4784(2)	0.057(1)
C(27)	2a	-0.1267(7)	-0.1915(7)	-0.4637(2)	0.062(2)
C(28)	2a	-0.2853(6)	-0.1591(6)	-0.4463(2)	0.046(1)
C(29)	2a	-0.2669(6)	-0.0484(5)	-0.4082(2)	0.039(1)
C(110)	2a	-0.5552(6)	-0.2778(5)	-0.0708(2)	0.040(1)
C(111)	2a	-0.4731(6)	-0.0403(5)	-0.1024(2)	0.044(1)
C(112)	2a	-0.3450(6)	0.0462(5)	-0.1216(2)	0.046(1)
C(113)	2a	-0.2055(5)	0.0550(5)	-0.0897(2)	0.032(1)
C(114)	2a	-0.1550(5)	-0.0966(5)	-0.0745(2)	0.033(1)
C(115)	2a	0.0021(6)	-0.0728(6)	-0.0488(2)	0.046(1)
C(116)	2a	0.0746(6)	0.0525(5)	-0.0722(2)	0.046(1)
C(117)	2a	-0.0498(6)	0.1053(5)	-0.1084(2)	0.038(1)
C(118)	2a	-0.2392(7)	0.1496(6)	-0.0496(2)	0.050(1)
C(119)	2a	-0.0232(5)	0.0389(5)	-0.1511(2)	0.036(1)

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Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
C(120)	2a	0.0082(5)	-0.0146(5)	-0.1857(2)	0.039(1)
C(121)	2a	0.0497(6)	-0.0702(6)	-0.2275(2)	0.045(1)
C(122)	2a	0.1619(6)	-0.1819(6)	-0.2332(2)	0.050(1)
C(123)	2a	0.1747(7)	-0.2005(6)	-0.2792(2)	0.056(1)
C(124)	2a	0.0750(7)	-0.1075(7)	-0.3011(2)	0.062(2)
C(125)	2a	0.0012(6)	-0.0249(6)	-0.2697(2)	0.052(1)
C(126)	2a	0.3240(8)	0.1655(8)	-0.2257(3)	0.072(2)
C(127)	2a	0.4348(8)	0.0577(7)	-0.2271(3)	0.078(2)
C(128)	2a	0.4629(6)	0.0315(6)	-0.2713(2)	0.055(1)
C(129)	2a	0.3679(7)	0.1244(7)	-0.2970(2)	0.059(2)
C(130)	2a	0.2843(8)	0.2094(8)	-0.2682(3)	0.073(2)
C(210)	2a	-0.1911(6)	0.0881(6)	-0.4228(2)	0.049(1)
C(211)	2a	-0.4205(6)	-0.0229(6)	-0.3870(2)	0.048(1)
C(212)	2a	-0.5009(6)	-0.1585(6)	-0.3726(2)	0.053(1)
C(213)	2a	-0.5178(6)	-0.2644(6)	-0.4099(2)	0.047(1)
C(214)	2a	-0.3603(6)	-0.2926(6)	-0.4285(2)	0.045(1)
C(215)	2a	-0.3903(7)	-0.4166(7)	-0.4591(2)	0.067(2)
C(216)	2a	-0.5193(7)	-0.5000(8)	-0.4379(2)	0.069(2)
C(217)	2a	-0.5578(7)	-0.4169(6)	-0.3961(2)	0.056(1)
C(218)	2a	-0.6392(8)	-0.2099(8)	-0.4450(2)	0.073(2)
C(219)	2a	-0.4631(7)	-0.4638(6)	-0.3581(2)	0.054(1)
C(220)	2a	-0.3748(6)	-0.5035(7)	-0.3289(2)	0.051(1)
C(221)	2a	-0.2574(6)	-0.5494(6)	-0.2951(2)	0.047(1)
C(222)	2a	-0.1205(6)	-0.4704(6)	-0.2828(2)	0.056(1)
C(223)	2a	-0.0386(8)	-0.5423(7)	-0.2489(3)	0.074(2)
C(224)	2a	-0.1193(8)	-0.6610(7)	-0.2386(2)	0.067(2)
C(225)	2a	-0.2596(7)	-0.6717(6)	-0.2677(2)	0.053(1)
C(226)	2a	-0.4382(9)	-0.3725(9)	-0.2188(3)	0.090(2)
C(227)	2a	-0.312(1)	-0.295(1)	-0.2103(3)	0.096(2)
C(228)	2a	-0.2202(8)	-0.3578(8)	-0.1768(3)	0.078(2)
C(229)	2a	-0.2902(7)	-0.4809(8)	-0.1640(2)	0.074(2)
C(230)	2a	-0.4304(7)	-0.4944(9)	-0.1905(2)	0.076(2)
H(11)	2a	-0.6302	-0.3157	-0.1357	0.059
H(12)	2a	-0.7409	-0.2159	-0.1121	0.059
H(13)	2a	-0.8599	-0.4350	-0.1276	0.090
H(14)	2a	-0.8486	-0.4064	-0.0770	0.090
H(15)	2a	-0.5033	-0.6258	-0.0567	0.065
H(16)	2a	-0.3846	-0.3710	0.0055	0.066
H(17)	2a	-0.3218	-0.5063	-0.0160	0.066
H(18)	2a	-0.1422	-0.3168	-0.0191	0.059
H(19)	2a	-0.1905	-0.3725	-0.0661	0.059
H(21)	2a	-0.1347	0.1397	-0.3578	0.068
H(22)	2a	-0.2635	0.2363	-0.3790	0.068
H(23)	2a	-0.0351	0.3588	-0.3650	0.084
H(24)	2a	-0.0859	0.3607	-0.4151	0.084
H(25)	2a	0.1866	0.0820	-0.4417	0.067
H(26)	2a	-0.1028	-0.0211	-0.5040	0.068
H(27)	2a	0.0556	-0.0801	-0.4856	0.068
H(28)	2a	-0.1398	-0.2538	-0.4882	0.074

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(29)	2a	-0.0635	-0.2351	-0.4408	0.074
H(110)	2a	-0.3111	-0.1320	-0.0285	0.048
H(111)	2a	-0.3866	-0.2319	-0.1121	0.045
H(112)	2a	-0.6016	-0.2284	-0.0477	0.048
H(113)	2a	-0.5548	-0.0512	-0.1246	0.053
H(114)	2a	-0.5101	0.0096	-0.0779	0.053
H(115)	2a	-0.3163	0.0032	-0.1482	0.055
H(116)	2a	-0.3824	0.1387	-0.1278	0.055
H(117)	2a	-0.1324	-0.1456	-0.1008	0.040
H(118)	2a	-0.0123	-0.0508	-0.0186	0.055
H(119)	2a	0.0657	-0.1541	-0.0502	0.055
H(120)	2a	0.1657	0.0233	-0.0857	0.055
H(121)	2a	0.0997	0.1256	-0.0515	0.055
H(122)	2a	-0.2626	0.2387	-0.0599	0.059
H(123)	2a	-0.1507	0.1482	-0.0295	0.059
H(124)	2a	-0.3248	0.1088	-0.0353	0.059
H(125)	2a	0.2167	-0.2330	-0.2102	0.060
H(126)	2a	0.2406	-0.2658	-0.2927	0.067
H(127)	2a	0.0584	-0.1000	-0.3324	0.075
H(128)	2a	-0.0700	0.0494	-0.2765	0.063
H(129)	2a	0.2832	0.2021	-0.1996	0.087
H(130)	2a	0.4826	0.0105	-0.2021	0.094
H(131)	2a	0.5326	-0.0360	-0.2820	0.066
H(132)	2a	0.3609	0.1294	-0.3284	0.070
H(133)	2a	0.2140	0.2829	-0.2767	0.087
H(210)	2a	-0.3509	-0.1214	-0.4698	0.055
H(211)	2a	-0.1985	-0.0886	-0.3859	0.047
H(212)	2a	-0.2583	0.1312	-0.4449	0.059
H(213)	2a	-0.401	0.0349	-0.3616	0.058
H(214)	2a	-0.4884	0.0249	-0.4078	0.058
H(215)	2a	-0.4411	-0.1994	-0.3485	0.064
H(216)	2a	-0.6008	-0.1353	-0.3634	0.064
H(217)	2a	-0.2938	-0.3252	-0.4045	0.054
H(218)	2a	-0.4236	-0.3858	-0.4880	0.081
H(219)	2a	-0.2997	-0.4726	-0.4606	0.081
H(220)	2a	-0.6081	-0.5064	-0.4579	0.083
H(221)	2a	-0.4842	-0.5920	-0.4301	0.083
H(222)	2a	-0.6013	-0.1305	-0.4581	0.089
H(223)	2a	-0.7324	-0.1935	-0.4314	0.089
H(224)	2a	-0.6577	-0.2836	-0.4672	0.089
H(225)	2a	-0.0908	-0.3839	-0.2957	0.068
H(226)	2a	0.0579	-0.5137	-0.2351	0.089
H(227)	2a	-0.0885	-0.7263	-0.2159	0.080
H(228)	2a	-0.3356	-0.7440	-0.2683	0.064
H(229)	2a	-0.5201	-0.3508	-0.2401	0.107
H(230)	2a	-0.2889	-0.2103	-0.2250	0.116
H(231)	2a	-0.1244	-0.3218	-0.1645	0.094
H(232)	2a	-0.2517	-0.5444	-0.1416	0.089
H(233)	2a	-0.504	-0.5686	-0.1897	0.091

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)	2a	0.23252(8)	0[a]	-0.26065(2)	0.0432(4)	0.0467(4)	0.0411(5)	-0.0058(4)	0.0076(4)	-0.0047(5)
Fe(2)	2a	-0.25132(8)	-0.4914(1)	-0.22989(3)	0.0574(5)	0.0413(4)	0.0490(5)	0.0102(5)	0.0034(5)	-0.0043(5)
O(13)	2a	-0.7422(5)	-0.6701(5)	-0.1093(2)	0.059(3)	0.054(3)	0.211(8)	-0.020(2)	-0.002(4)	-0.031(4)
O(23)	2a	0.2317(5)	0.2919(5)	-0.3892(2)	0.074(3)	0.080(3)	0.127(5)	-0.017(3)	-0.005(4)	0.019(4)
O(117)	2a	-0.0506(4)	0.2544(4)	-0.1133(1)	0.057(2)	0.032(2)	0.083(3)	-0.004(2)	0.012(3)	0.003(2)
O(217)	2a	-0.7220(4)	-0.4245(5)	-0.3874(2)	0.043(2)	0.080(3)	0.084(4)	-0.014(2)	-0.007(3)	0.001(3)

a: arbitrarily fixed for definition of the origin.

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