Crystal structure of (3*R*,4*S*)-4-(4-(benzyloxy)phenyl)-1-(4-fluorophenyl)-3-(3-(4-fluorophenyl)-3-oxopropyl)azetidin-2-one, $C_{31}H_{25}F_2NO_3$

Zhao Yang and Zhi-Xiang Wang*

School of Pharmacy, China Pharmaceutical University, Tongjiaxiang No.24 Nanjing, Nanjing 210009, P. R. China

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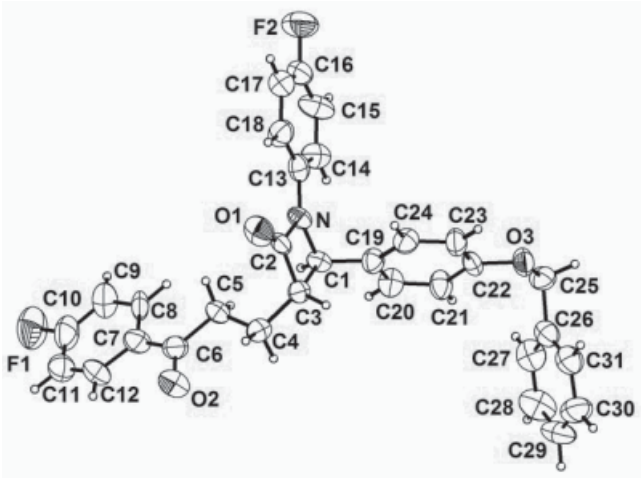

Abstract

$C_{31}H_{25}F_2NO_3$, orthorhombic, $P2_12_12_1$ (no. 19), a = 5.840(1) Å, b = 16.645(3) Å, c = 25.716(5) Å, V = 2499.8 Å^3, Z = 4, $R_{gt}(F)$ = 0.0672, $wR_{ref}(F^2)$ = 0.1072, T = 293 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.10×0.10×0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.95 cm^{-1}
Diffractometer, scan mode:	Nonius CAD4, $\omega/2\theta$
$2\theta_{max}$:	50.74°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5195, 2655
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1275
$N(param)_{refined}$:	334
Programs:	SHELX [5]

Source of material

The title compound was obtained from nucleophilic substitution reaction of 3-((2*S*,3*R*)-2-(4-(benzyloxy)phenyl)-1-(4-fluorophenyl)-4-oxoazetidin-3-yl)propanoyl chloride and (4-fluorophenyl)magnesium bromide as described in literature and recrystallized from ethanol solution at room temperature to give crystals suitable for single-crystal X-ray diffraction.

Experimental details

H atoms were positioned geometrically with C–H = 0.93, 0.97 and 0.98 Å for aromatic, methylene and methyl, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2 (or 1.5 for methyl groups) times U_{eq}(C).

Discussion

(3*R*,4*S*)-4-(4-(benzyloxy)phenyl)-1-(4-fluorophenyl)-3-(3-(4-fluorophenyl)-3-oxopropyl)azetidin-2-one is an important intermediate of the synthetic protocol of Ezetimibe [1]. Until now, there are a few examples of Ezetimibe derivatives which have been synthesized and characterized [2-4].

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

Atom	Site	x	y	z	U_{iso}
H(1A)	4*a*	0.9167	1.0599	–0.0561	0.054
H(3A)	4*a*	1.3650	1.0759	–0.0128	0.052
H(4A)	4*a*	1.1370	1.1932	–0.0017	0.062
H(4B)	4*a*	1.3463	1.2151	–0.0371	0.062
H(5A)	4*a*	0.8976	1.1889	–0.0720	0.072
H(5B)	4*a*	1.1039	1.1989	–0.1105	0.072
H(8A)	4*a*	0.6820	1.2435	–0.1305	0.072
H(9A)	4*a*	0.3772	1.3044	–0.1741	0.097
H(11A)	4*a*	0.6145	1.5186	–0.1318	0.089
H(12A)	4*a*	0.9213	1.4579	–0.0898	0.088
H(14A)	4*a*	0.8139	0.9344	–0.1103	0.075
H(15A)	4*a*	0.7088	0.8443	–0.1754	0.091
H(17A)	4*a*	1.2873	0.8904	–0.2522	0.097
H(18A)	4*a*	1.3924	0.9816	–0.1861	0.075
H(20A)	4*a*	0.7436	0.9988	0.0151	0.072
H(21A)	4*a*	0.6977	0.8960	0.0749	0.074
H(23A)	4*a*	1.2911	0.8009	0.0274	0.069
H(24A)	4*a*	1.3296	0.9049	–0.0317	0.065
H(25A)	4*a*	0.7583	0.7030	0.1265	0.070
H(25B)	4*a*	0.6392	0.7701	0.0935	0.070
H(27A)	4*a*	0.4246	0.8474	0.1498	0.090
H(29A)	4*a*	0.6640	0.9283	0.2848	0.091
H(28A)	4*a*	0.3718	0.9169	0.2263	0.100
H(30A)	4*a*	0.9971	0.8551	0.2717	0.097
H(31A)	4*a*	1.0491	0.7863	0.1941	0.086

* Correspondence author (e-mail: yzcpu@163.com)

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}
N	4*a*	1.1840(8)	1.0152(3)	–0.1009(2)	0.043(3)	0.034(2)	0.059(3)	–0.008(3)	–0.003(3)	–0.007(2)
F(1)	4*a*	0.2972(8)	1.4521(3)	–0.1809(2)	0.108(4)	0.119(4)	0.125(4)	0.039(3)	–0.016(4)	0.038(3)
C(1)	4*a*	1.064(1)	1.0329(3)	–0.0507(2)	0.043(3)	0.039(3)	0.052(4)	–0.001(3)	–0.003(3)	–0.010(3)
O(2)	4*a*	1.1416(8)	1.3464(3)	–0.0483(2)	0.069(3)	0.060(3)	0.089(4)	0.000(3)	–0.001(3)	–0.013(3)
F(2)	4*a*	0.9290(9)	0.8094(2)	–0.2571(2)	0.148(5)	0.090(3)	0.089(3)	0.009(3)	–0.033(4)	–0.034(3)
C(2)	4*a*	1.358(1)	1.0691(3)	–0.0941(2)	0.056(4)	0.026(3)	0.053(4)	–0.002(3)	–0.004(4)	–0.002(3)
O(1)	4*a*	1.5233(8)	1.0876(2)	–0.1205(2)	0.063(3)	0.063(3)	0.093(4)	–0.022(3)	0.011(3)	0.000(3)
O(3)	4*a*	0.9749(8)	0.7710(2)	0.0879(2)	0.078(3)	0.046(2)	0.061(3)	0.008(3)	0.018(3)	0.015(2)
C(3)	4*a*	1.265(1)	1.0940(3)	–0.0410(2)	0.039(3)	0.046(3)	0.045(4)	0.009(3)	–0.004(3)	0.000(3)
C(4)	4*a*	1.206(1)	1.1842(3)	–0.0356(2)	0.053(4)	0.057(4)	0.045(4)	0.002(4)	0.000(3)	–0.003(3)
C(5)	4*a*	1.046(1)	1.2144(3)	–0.0766(2)	0.087(6)	0.036(4)	0.057(4)	–0.014(4)	–0.006(4)	–0.005(3)
C(6)	4*a*	1.014(1)	1.3046(4)	–0.0756(2)	0.062(5)	0.055(4)	0.051(4)	0.010(4)	–0.008(4)	–0.007(3)
C(7)	4*a*	0.830(1)	1.3431(4)	–0.1048(3)	0.072(5)	0.050(4)	0.064(4)	–0.014(4)	–0.004(4)	–0.009(4)
C(8)	4*a*	0.669(1)	1.2992(4)	–0.1304(2)	0.081(5)	0.044(4)	0.056(4)	0.001(4)	–0.008(4)	0.024(3)
C(9)	4*a*	0.486(1)	1.3348(4)	–0.1567(3)	0.078(6)	0.075(5)	0.089(6)	–0.003(5)	–0.010(5)	0.025(5)
C(10)	4*a*	0.476(2)	1.4169(5)	–0.1554(3)	0.083(7)	0.082(6)	0.078(6)	0.031(6)	0.000(5)	0.025(5)
C(11)	4*a*	0.629(2)	1.4630(4)	–0.1315(3)	0.094(6)	0.062(5)	0.067(5)	0.017(5)	0.019(5)	0.001(4)
C(12)	4*a*	0.811(2)	1.4264(4)	–0.1061(3)	0.095(6)	0.045(4)	0.081(5)	0.019(5)	0.005(5)	–0.010(4)
C(13)	4*a*	1.119(1)	0.9629(4)	–0.1419(3)	0.045(4)	0.047(4)	0.062(4)	0.005(3)	–0.010(3)	0.013(3)
C(14)	4*a*	0.911(1)	0.9248(4)	–0.1384(3)	0.051(4)	0.067(5)	0.068(5)	0.012(4)	–0.009(4)	–0.012(4)
C(15)	4*a*	0.847(1)	0.8719(4)	–0.1775(3)	0.057(5)	0.068(5)	0.102(6)	0.000(4)	–0.010(5)	–0.037(5)
C(16)	4*a*	0.986(2)	0.8607(4)	–0.2181(3)	0.116(8)	0.045(4)	0.065(5)	0.001(5)	–0.025(6)	–0.006(4)
C(17)	4*a*	1.193(2)	0.8994(4)	–0.2236(3)	0.121(7)	0.057(5)	0.064(5)	0.007(6)	–0.011(6)	–0.001(4)
C(18)	4*a*	1.256(1)	0.9530(4)	–0.1837(2)	0.067(5)	0.066(5)	0.054(4)	–0.015(4)	–0.005(4)	0.005(4)
C(19)	4*a*	1.044(1)	0.9628(4)	–0.0146(2)	0.050(4)	0.051(4)	0.050(4)	0.008(4)	–0.001(4)	0.006(3)
C(20)	4*a*	0.854(1)	0.9589(4)	0.0174(2)	0.048(4)	0.067(4)	0.064(4)	0.007(4)	0.007(4)	0.010(4)
C(21)	4*a*	0.824(1)	0.8970(4)	0.0530(2)	0.061(4)	0.057(4)	0.066(4)	0.020(4)	0.017(4)	0.016(4)
C(22)	4*a*	0.986(1)	0.8376(3)	0.0550(2)	0.056(4)	0.029(3)	0.058(4)	–0.003(3)	–0.001(4)	0.005(3)
C(23)	4*a*	1.179(1)	0.8402(3)	0.0247(2)	0.066(5)	0.047(4)	0.059(4)	0.003(4)	–0.007(4)	0.014(3)
C(24)	4*a*	1.201(1)	0.9034(4)	–0.0104(2)	0.050(4)	0.062(4)	0.051(4)	0.008(4)	0.005(4)	0.000(3)
C(25)	4*a*	0.768(1)	0.7590(4)	0.1164(2)	0.067(5)	0.058(4)	0.051(4)	–0.007(4)	0.016(4)	–0.004(3)
C(26)	4*a*	0.743(1)	0.8107(4)	0.1652(3)	0.054(4)	0.048(4)	0.065(5)	–0.003(4)	0.016(4)	0.002(4)
C(27)	4*a*	0.542(1)	0.8494(4)	0.1743(3)	0.061(5)	0.076(5)	0.088(6)	–0.004(5)	0.005(5)	0.005(5)
C(29)	4*a*	0.682(1)	0.8969(4)	0.2551(3)	0.080(6)	0.062(5)	0.086(6)	–0.020(5)	0.033(5)	–0.032(4)
C(28)	4*a*	0.511(1)	0.8917(4)	0.2198(3)	0.041(4)	0.086(6)	0.123(7)	0.012(5)	0.008(5)	–0.023(6)
C(30)	4*a*	0.882(1)	0.8551(4)	0.2466(3)	0.095(7)	0.085(6)	0.063(5)	0.003(6)	–0.017(5)	–0.017(4)
C(31)	4*a*	0.912(1)	0.8129(4)	0.2005(3)	0.075(5)	0.062(5)	0.078(6)	0.014(5)	–0.002(5)	–0.001(4)

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