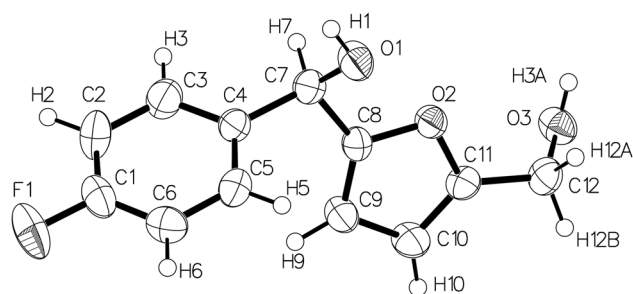


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The crystal structure of (4-fluorophenyl)(5-(hydroxymethyl)furan-2-yl)methanol, $C_{12}H_{11}FO_3$



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Abstract

$C_{12}H_{11}FO_3$, monoclinic, $P2_1/c$ (no. 14), $a = 12.0648(9)$ Å, $b = 8.3896(5)$ Å, $c = 10.5727(9)$ Å, $\beta = 104.204(8)^\circ$, $V = 1037.44(14)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0614$, $wR_{ref}(F^2) = 0.1516$, $T = 293$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of materials

An amount of 5 mmol 5-hydroxymethylfurfural (HMF) was dissolved in anhydrous tetrahydrofuran (THF). The fluoro-Grignard reagent (4-fluorophenyl magnesium bromide, 12.5 mL, 1 mol/L) was added to the solution slowly at ice-

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	$0.25 \times 0.18 \times 0.09$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.11 mm ⁻¹
Diffractometer, scan mode:	Rigaku EOS, φ and ω -scans
θ_{max} , completeness:	29.3° , >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	4794, 2386, 0.029
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 1831
$N(param)_{refined}$:	147
Programs:	CrysAlis ^{PRO} [1], OLEX2 [2], SHELX [3, 4]

water bath conditions. The mixture was then stirred for 30 min at room temperature. Subsequently, 0.5 mL saturated ammonium chloride solution was added, and stirred for 30 min. The solution was later extracted three times with ethyl acetate, and the organic phases were combined. The product was further purified by column chromatography (1:1 petroleum ether/ethyl acetate). Upon slow evaporation at room temperature, crystals were acquired after one week.

Experimental details

All H atoms were refined as riding atoms in their geometrically idealized positions, with $d(C-H) = 0.93$ Å and $U_{iso}(H) = 1.2 U_{eq}(C)$.

Comment

HMF (Hydroxymethylfurfural) is considered as an important building block platform, because it can be obtained from direct transformation of fructose, glucose, sucrose, cellulose, and inulin [5, 6]. The functional groups of HMF, including C=O, C-O, and furan ring, allows this compound some flexibility in its catalytic conversions of multifunctional substrates and in the future of value-added chemicals and fuel component [5, 7]. For example, the synthesis of 2,5-diformylfuran (DFF) from selective oxidation of HMF is commercially important, because DFF is a versatile precursor

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1	0.5807 (2)	1.1485 (3)	0.4555 (3)	0.0465 (6)
C2	0.5541 (2)	1.1186 (3)	0.3251 (3)	0.0553 (7)
H2	0.488307	1.161184	0.270524	0.066*
C3	0.6273 (2)	1.0232 (3)	0.2753 (3)	0.0477 (6)
H3	0.610352	1.001393	0.186385	0.057*
C4	0.7247 (2)	0.9603 (3)	0.3562 (2)	0.0365 (5)
C5	0.7490 (2)	0.9956 (3)	0.4879 (2)	0.0409 (6)
H5	0.815466	0.955703	0.543011	0.049*
C6	0.6763 (2)	1.0892 (3)	0.5394 (3)	0.0454 (6)
H6	0.692091	1.111043	0.628249	0.054*
C7	0.8018 (2)	0.8522 (3)	0.3006 (2)	0.0403 (6)
H7	0.772644	0.846663	0.205654	0.048*
C8	0.80144 (18)	0.6883 (3)	0.3564 (2)	0.0340 (5)
C9	0.7376 (2)	0.6190 (3)	0.4284 (2)	0.0393 (5)
H9	0.678465	0.665697	0.457347	0.047*
C10	0.7784 (2)	0.4594 (3)	0.4518 (3)	0.0427 (6)
H10	0.751049	0.382337	0.499653	0.051*
C11	0.86342 (19)	0.4413 (2)	0.3917 (2)	0.0344 (5)
C12	0.9415 (2)	0.3077 (3)	0.3858 (2)	0.0404 (6)
H12A	1.019196	0.347937	0.404234	0.048*
H12B	0.937413	0.231329	0.453436	0.048*
F1	0.50954 (15)	1.2440 (2)	0.5034 (2)	0.0749 (6)
O1	0.91720 (15)	0.90533 (19)	0.3301 (2)	0.0508 (5)
H1	0.920018	0.994687	0.299638	0.076*
O2	0.87890 (13)	0.58147 (17)	0.33035 (15)	0.0364 (4)
O3	0.91688 (15)	0.22764 (19)	0.26324 (17)	0.0437 (4)
H3A	0.955981	0.265661	0.217323	0.065*

for the synthesis of fungicides, pharmaceuticals, and functional polymers [8–12]. Additionally, the oxidized derivative 2,5-furandicarboxylic acid (FDCA) was identified as one of the most valuable biomass-derived platform chemicals by U. S. Department of Energy [13, 14]. Therefore, a novel HMF analog, i.e., (4-fluorophenyl)(5-(hydroxymethyl)furan-2-yl)methanol, was synthesized and crystallized.

As displayed in figure, there is one molecule in the asymmetric unit of the title compound. The C–O bond lengths of 1.422(3) Å (C7–O1) and 1.425(3) Å (C12–O3) suggest the localized single bonds. The dihedral angle of the 4-fluorobenzene ring and furan ring is quite large (70.25°). The hydroxymethyl group donates its H atom to the O atom of the methanol group in the strong intermolecular hydrogen bond (O1–H1⋯O3, 1.9905(17) Å), and then contributes to the stable three-dimensional structures, which is like similar structures [15, 16]. Bond lengths and angles are as expected [15–17].

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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