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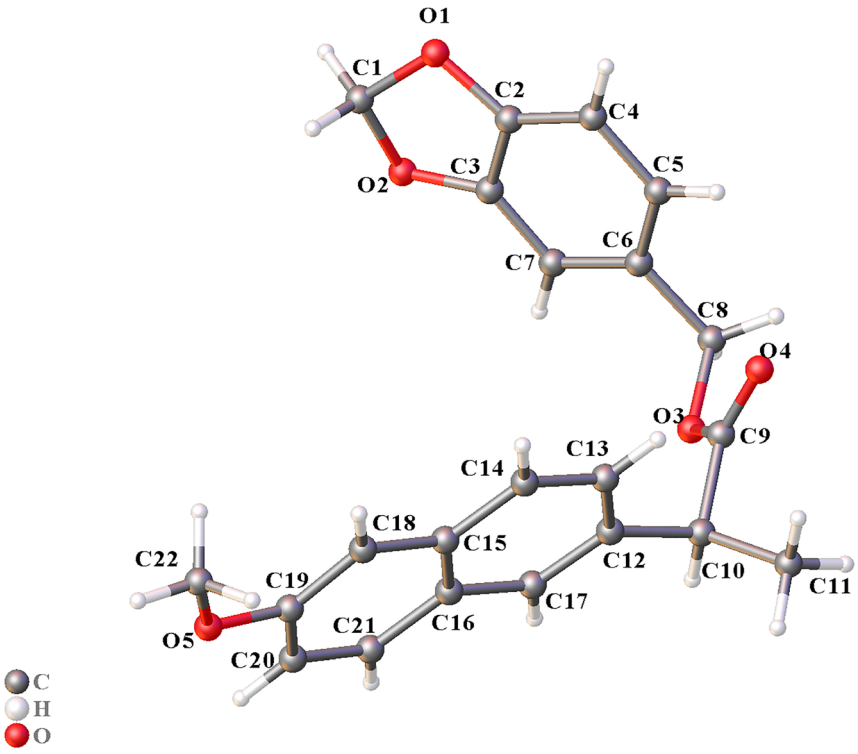
Crystal structure of benzo[*d*][1,3]dioxol-5-ylmethyl 2-(6-methoxynaphthalen-2-yl)propanoate, $C_{22}H_{20}O_5$



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Abstract

$C_{22}H_{20}O_5$, orthorhombic, $P2_12_12_1$ (no. 19), a = 5.8585(3) Å, b = 16.2727(10) Å, c = 18.4886(11) Å, V = 1762.59(18) Å^3, Z = 4, $R_{gt}(F)$ = 0.0276, $wR_{ref}(F^2)$ = 0.0700, T = 100(2) 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.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.12 × 0.11 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.5°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	86,122, 4046, 0.029
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3925
$N(param)_{refined}$:	246
Programs:	Olex2,[1] SHELX[2,3]

1 Source of material

The yellow solid of naproxen acylchloride was synthesized according to the literature method.[4,5] Benzo[*d*][1,3]dioxol-5-ylmethanol (0.02 mol, 3.04 g) and 4-(dimethylamino)-pyridin (DMAP, 0.003 mol, 0.36 g) were dissolved in dry

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1022 (3)	0.30974 (10)	0.82329 (9)	0.0225 (3)
H1A	0.155351	0.266488	0.789620	0.027*
H1B	0.001805	0.284051	0.859961	0.027*
C2	0.3260 (3)	0.42017 (9)	0.81873 (8)	0.0183 (3)
C3	0.1382 (3)	0.43434 (9)	0.77500 (8)	0.0168 (3)
C4	0.5063 (3)	0.47382 (9)	0.82271 (8)	0.0195 (3)
H4	0.633164	0.463916	0.853517	0.023*
C5	0.4935 (3)	0.54409 (9)	0.77887 (8)	0.0181 (3)
H5	0.615479	0.582556	0.779522	0.022*
C6	0.3057 (3)	0.55867 (9)	0.73440 (8)	0.0164 (3)
C7	0.1211 (3)	0.50304 (9)	0.73213 (8)	0.0172 (3)
H7	−0.008611	0.512607	0.702448	0.021*
C8	0.2917 (3)	0.63607 (9)	0.69014 (8)	0.0191 (3)
H8A	0.133615	0.657450	0.692088	0.023*
H8B	0.393305	0.678032	0.711742	0.023*
C9	0.5814 (2)	0.62538 (8)	0.59991 (8)	0.0161 (3)
C10	0.6281 (3)	0.61686 (9)	0.51931 (8)	0.0170 (3)
H10	0.486015	0.631761	0.492342	0.020*
C11	0.8175 (3)	0.67755 (9)	0.49794 (9)	0.0233 (3)
H11A	0.770538	0.733534	0.510589	0.035*
H11B	0.844514	0.674117	0.445720	0.035*
H11C	0.958162	0.663675	0.523893	0.035*
C12	0.6916 (3)	0.52861 (9)	0.50034 (8)	0.0155 (3)
C13	0.8991 (3)	0.49304 (9)	0.52492 (8)	0.0173 (3)
H13	0.995576	0.523325	0.556461	0.021*
C14	0.9619 (3)	0.41519 (9)	0.50359 (8)	0.0166 (3)
H14	1.100429	0.392302	0.521165	0.020*
C15	0.8235 (2)	0.36852 (9)	0.45580 (7)	0.0149 (3)
C16	0.6134 (3)	0.40322 (9)	0.43250 (7)	0.0152 (3)
C17	0.5509 (3)	0.48330 (9)	0.45603 (8)	0.0163 (3)
H17	0.409246	0.505950	0.440891	0.020*
C18	0.8906 (3)	0.28925 (9)	0.43014 (8)	0.0162 (3)
H18	1.029198	0.265069	0.446253	0.019*
C19	0.7529 (3)	0.24836 (9)	0.38198 (8)	0.0170 (3)
C20	0.5413 (3)	0.28229 (9)	0.35973 (8)	0.0185 (3)
H20	0.446703	0.252615	0.327170	0.022*
C21	0.4722 (3)	0.35740 (9)	0.38475 (8)	0.0177 (3)
H21	0.328884	0.379064	0.370121	0.021*
C22	1.0098 (3)	0.13469 (9)	0.36899 (9)	0.0221 (3)
H22A	1.021641	0.082385	0.342995	0.033*
H22B	1.015520	0.124482	0.421198	0.033*
H22C	1.136931	0.170480	0.355067	0.033*
O1	0.2938 (2)	0.34857 (7)	0.85779 (6)	0.0238 (3)
O2	−0.01995 (19)	0.37217 (7)	0.78445 (6)	0.0214 (2)
O3	0.35646 (18)	0.62359 (7)	0.61434 (6)	0.0183 (2)
O4	0.72732 (19)	0.63360 (7)	0.64556 (6)	0.0217 (2)
O5	0.7984 (2)	0.17372 (7)	0.35105 (6)	0.0215 (2)

tetrahydrofuran (25 mL) and triethylamine (0.03 mol, 4 mL). The solution of naproxen acylchloride in dry tetrahydrofuran was dropwise added at 0 °C. The reaction mixture was stirred for 3 h at room temperature. The reaction mixture was filtrated and the filtrate was concentrated under vacuum. The residue was dissolved in dichloromethane, successively washed with 5 % NaOH solution and water to pH = 7, and finally dried with anhydrous Na_2SO_4. The solution was filtrated, and concentrated under vacuum to obtain the crude product. The crude product was purified by recrystallization in ethyl acetate. The single crystals were obtained from tetrahydrofuran.

2 Experimental details

The C-bound H atoms were geometrically placed and refined as riding with U_{iso}(H) = 1.2–1.5 U_{eq}(C) using the appropriate *SHELXL* commands. The N-bound H atoms were refined freely.

3 Comment

As a nonselective nonsteroidal anti-inflammatory drug, naproxen often brings gastrointestinal side effects. A view on the structure of the title molecule is shown in the figure. One carbon-oxygen double bond exists in the compound, the C–O double bond distance (C9–O4) is 1.2086(19) Å. The dihedral angle of naphthalene ring and benzo[*d*][1,3]dioxole moiety is 53.4°. All the bond lengths and angles are in the expected ranges.[6,7]

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