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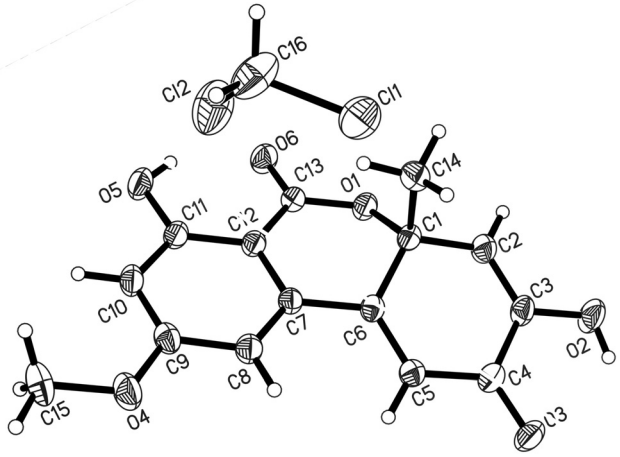
The crystal structure of 3,7-dihydroxy-9-methoxy-4*a*-methyl-4a*H*-benzo[*c*] chromene-2,6-dione — dichloromethane (1/1), $C_{16}H_{14}Cl_2O_6$



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Abstract

$C_{16}H_{14}Cl_2O_6$, monoclinic, $P2_1/c$ (no. 14), a = 8.9847(19) Å, b = 18.284(4) Å, c = 11.107(3) Å, β = 115.942(2)°, V = 1640.7(7) Å^3, Z = 4, $R_{gt}(F)$ = 0.0473, $wR_{ref}(F^2)$ = 0.1352, T = 273 K.

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The crystal 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.

Table 1: Data collection and handling.

Crystal:	Block
Size:	0.6 × 0.6 × 0.04 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.43 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, ω-scans
θ_{max}, completeness:	26.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9082, 3381, 0.019
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2691
$N(param)_{refined}$:	221
Programs:	Bruker programs [1, 2], OLEX2 [3], SHELX [4–6]

Source of materials

The title compound (without the solvent molecule) is a natural product, obtained by extraction and isolation from Fungi extracts.

Experimental details

The structure was treated with the olex2 crystallographic software package, solved with the SHELXT [6] structure solution program and refined with the SHELXL [5] refinement package. Carbon-bound hydrogen atoms were placed in calculated positions and refined with riding coordinates, with U_{iso}(H) fixed at 1.2 times of U_{eq}(C).

Comment

The title structure $C_{16}H_{14}Cl_2O_6$ contains one $C_{15}H_{12}O_6$ and one solvent molecule CH_2Cl_2. The structure of $C_{15}H_{12}O_6$ has been reported [7]. However, no three-dimensional coordinates were deposited with the Cambridge Crystallographic Data Centre in the reported literature. Furthermore, the reported structure is solvent-free, while the title structure also contains a solvent molecule CH_2Cl_2.

The title molecule is composed of three fused six-membered rings, cyclohexenone, pyranone and benzene ring. Moreover, the middle part is a pyranone ring. In the

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3475 (3)	0.64101 (11)	0.67713 (19)	0.0380 (5)
C2	0.4069 (3)	0.66159 (13)	0.8211 (2)	0.0474 (5)
H2A	0.462287	0.705694	0.851398	0.057*
C3	0.3822 (3)	0.61821 (13)	0.9068 (2)	0.0441 (5)
C4	0.3147 (3)	0.54387 (12)	0.8701 (2)	0.0426 (5)
C5	0.2807 (3)	0.51836 (12)	0.7355 (2)	0.0406 (5)
H5A	0.243896	0.470670	0.710935	0.049*
C6	0.3011 (2)	0.56187 (11)	0.64671 (19)	0.0348 (4)
C7	0.2779 (3)	0.53801 (11)	0.51289 (19)	0.0354 (4)
C8	0.1787 (3)	0.48014 (12)	0.4454 (2)	0.0421 (5)
H8	0.121254	0.454140	0.483687	0.051*
C9	0.1643 (3)	0.46036 (12)	0.3184 (2)	0.0427 (5)
C10	0.2483 (3)	0.49823 (12)	0.2598 (2)	0.0446 (5)
H10	0.237945	0.484940	0.175654	0.054*
C11	0.3484 (3)	0.55648 (12)	0.3284 (2)	0.0405 (5)
C12	0.3643 (3)	0.57801 (11)	0.45461 (19)	0.0363 (4)
C13	0.4759 (3)	0.63741 (11)	0.5261 (2)	0.0390 (5)
C14	0.2025 (3)	0.69049 (13)	0.5913 (2)	0.0531 (6)
H14A	0.236715	0.740707	0.607569	0.080*
H14B	0.112252	0.682522	0.614027	0.080*
H14C	0.167518	0.679309	0.498390	0.080*
C15	0.0447 (4)	0.37962 (17)	0.1308 (3)	0.0668 (8)
H15A	0.002696	0.419544	0.068879	0.100*
H15B	−0.031350	0.339349	0.100190	0.100*
H15C	0.150129	0.364629	0.136792	0.100*
O1	0.48593 (19)	0.65877 (8)	0.64487 (14)	0.0428 (4)
O2	0.4191 (3)	0.64085 (10)	1.03263 (16)	0.0639 (5)
H2	0.398690	0.607983	1.073400	0.096*
O3	0.2942 (2)	0.50546 (10)	0.95164 (16)	0.0579 (5)
O4	0.0637 (2)	0.40260 (10)	0.26065 (16)	0.0569 (5)
O5	0.4293 (2)	0.59183 (10)	0.26814 (16)	0.0546 (4)
H5	0.490919	0.622716	0.319255	0.082*
O6	0.5651 (2)	0.66846 (9)	0.48549 (17)	0.0525 (4)
C16	−0.2044 (4)	0.6940 (2)	0.1712 (3)	0.0839 (10)
H16A	−0.286731	0.661832	0.106888	0.101*
H16B	−0.224726	0.743024	0.134180	0.101*
Cl1	−0.22673 (9)	0.69349 (4)	0.32053 (8)	0.0703 (2)
Cl2	−0.01065 (12)	0.66619 (7)	0.19446 (9)	0.0991 (4)

structure, the benzene ring and the pyranone ring are almost coplanar, conforming to the reported [7]. The molecules are connected by intermolecular O2–H···O5 hydrogen bonds, with a distance of 2.729 Å. The reported intermolecular hydrogen bond is 2.990 Å [7]. Also there is an intramolecular hydrogen bonding force between O5–H···O6, with a distance of 2.589 Å, which is approximate to the reported 2.590 Å [7]. The geometry of title structure was characterized with the bond angles and lengths. In particular, the bond angles for C1···O1···C13, C9···O4···C15 and Cl1···C16···Cl2 are 118.34°, 117.7° and 112.52°, respectively. For another, the mean bond lengths of C–O, Cl–C and C–O are 1.402 Å (coincident with the reported) [7], 1.739 and 1.220 Å. As a consequence, bond angles and lengths of the title structure are within normal ranges.

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

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