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The crystal structure of bis(2-acetyl-5-methoxyphenyl)carbonate 1.5 hydrate, $C_{19}H_{18}O_7$

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.15 × 0.08 × 0.05 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.11 mm ⁻¹
Diffractometer, scan mode:	BRUKER APEX-II, φ and ω
$\theta_{\max}$, completeness:	27.6°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	23,174, 8508, 0.032
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 6621
$N(\text{param})_{\text{refined}}$:	531
Programs:	OLEX2 [1, 2], SHELX [3–5]

<https://doi.org/10.1515/ncrs-2021-0336>

Received August 19, 2021; accepted September 16, 2021;

published online September 29, 2021

Abstract

$C_{19}H_{18}O_7$, orthorhombic, $P2_12_12$ (no. 18), $a = 12.467(3)$ Å, $b = 39.168(8)$ Å, $c = 7.6005(16)$ Å, $V = 3711.4(14)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0440$, $wR_{\text{ref}}(F^2) = 0.1054$, $T = 296.15$ K.

CCDC no.: 2102799

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

Source of material

The synthetic process are as follows: firstly, paeonol (0.5 g; 3.01 mmol) and anhydrous pyridine (0.50 g; 6 mmol) were dissolved in 10 ml methylene chloride at -10 °C under nitrogen protection. Secondly, triphosgene (0.9 g; 3.1 mmol) was dissolved in 5 mL dichloromethane, then added dropwise at room temperature. The reaction proceeded for 3–4 h, monitored by Thin-Layer Chromatography (TLC). Furthermore, the pyridine salt was filtered off and 50 mL

ethyl acetate was added to extract, and the extracted organic phase was treated by distilled water washing, anhydrous sodium sulfate drying and vacuum distillation. Finally, column chromatography with petroleum ether and ethyl acetate in a molar ratio of 6:1 was adopted to obtain the white final compound, which crystallized for several days at room temperature, giving colorless block crystals.

Experimental details

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

Comment

2-Hydroxy-4-methoxyacetophenone also known as paeonol or peonol, is obtained from the dried root bark of Xu Changqing or peony. Additionally, it has various bioactivities, such as antineoplastic, whitening, antiallergic, anti-inflammatory, analgesic and anti-virus effect [6]. At the same time, the presence of phenolic hydroxyl groups causes paeonol to be irritating, and thus limiting its further development for clinical applications. Consequently, bis(2-acetyl-5-methoxyphenyl) carbonate was synthesized by coupling the formyl group as well as phenolic hydroxyl of paeonol, which can improve the lipid solubility and bioavailability [7–9].

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	0.4413 (3)	0.11012 (10)	0.3131 (5)	0.0752 (11)
H1A	0.377158	0.119257	0.261562	0.113*
H1B	0.439493	0.113464	0.438188	0.113*
H1C	0.445989	0.086150	0.287797	0.113*
C2	0.5367 (2)	0.16163 (7)	0.2520 (4)	0.0410 (6)
C3	0.4571 (2)	0.18203 (8)	0.3220 (4)	0.0443 (7)
H3	0.395642	0.172300	0.369939	0.053*
C4	0.4698 (2)	0.21678 (8)	0.3200 (4)	0.0410 (7)
H4	0.415344	0.230342	0.365995	0.049*
C5	0.56072 (19)	0.23265 (7)	0.2522 (3)	0.0333 (6)
C6	0.64112 (18)	0.21097 (6)	0.1876 (3)	0.0309 (5)
C7	0.6299 (2)	0.17635 (7)	0.1855 (3)	0.0362 (6)
H7	0.684111	0.162656	0.139834	0.043*
C8	0.5671 (2)	0.27034 (7)	0.2462 (4)	0.0402 (6)
C9	0.4684 (3)	0.29051 (9)	0.2864 (5)	0.0648 (9)
H9A	0.483573	0.314412	0.273660	0.097*
H9B	0.446018	0.285944	0.404903	0.097*
H9C	0.412293	0.284131	0.206420	0.097*
C10	0.80461 (18)	0.24022 (6)	0.2102 (3)	0.0289 (5)
C11	0.95285 (19)	0.27629 (7)	0.1782 (3)	0.0329 (6)
C12	0.9351 (2)	0.31069 (7)	0.1772 (3)	0.0391 (6)
H12	0.870592	0.319525	0.135958	0.047*
C13	1.0155 (2)	0.33234 (8)	0.2392 (4)	0.0439 (7)
C14	1.1101 (2)	0.31883 (8)	0.3008 (4)	0.0482 (7)
H14	1.163304	0.333226	0.343623	0.058*
C15	1.04812 (19)	0.26136 (7)	0.2361 (3)	0.0369 (6)
C16	1.1261 (2)	0.28445 (8)	0.2993 (4)	0.0434 (7)
H16	1.190721	0.275859	0.341341	0.052*
C17	1.0707 (2)	0.22445 (8)	0.2305 (4)	0.0433 (7)
C18	1.1820 (3)	0.21249 (10)	0.2779 (6)	0.0784 (12)
H18A	1.185214	0.188051	0.270046	0.118*
H18B	1.198638	0.219480	0.395842	0.118*
H18C	1.232966	0.222333	0.197963	0.118*
C19	0.9160 (3)	0.38305 (9)	0.1725 (5)	0.0757 (11)
H19A	0.905346	0.375094	0.054311	0.114*
H19B	0.926724	0.407314	0.171548	0.114*
H19C	0.853930	0.377686	0.242064	0.114*
C20	0.8559 (3)	0.46750 (8)	0.4639 (5)	0.0578 (8)
H20A	0.909441	0.473577	0.549094	0.087*
H20B	0.876068	0.476309	0.350730	0.087*
H20C	0.850075	0.443089	0.457580	0.087*
C21	0.7508 (2)	0.48225 (7)	0.5168 (4)	0.0408 (6)
C22	0.73827 (19)	0.51975 (7)	0.5198 (3)	0.0328 (6)
C23	0.64808 (19)	0.53629 (7)	0.5845 (3)	0.0314 (5)
C24	0.6393 (2)	0.57112 (7)	0.5926 (3)	0.0354 (6)
H24	0.577867	0.581246	0.638374	0.042*
C25	0.7232 (2)	0.59103 (7)	0.5315 (3)	0.0370 (6)
C26	0.8133 (2)	0.57577 (7)	0.4616 (4)	0.0402 (6)
H26	0.869000	0.589107	0.417868	0.048*
C27	0.8204 (2)	0.54089 (7)	0.4569 (4)	0.0388 (6)
H27	0.881866	0.530895	0.410326	0.047*
C28	0.6338 (3)	0.64290 (8)	0.6061 (5)	0.0578 (8)
H28A	0.570054	0.636580	0.543097	0.087*
H28B	0.644366	0.667113	0.597202	0.087*
H28C	0.626312	0.636633	0.727561	0.087*

Table 2: (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C29	0.500000	0.500000	0.5544 (5)	0.0331 (8)
C30	0.3373 (2)	0.47801 (9)	0.0707 (4)	0.0464 (7)
H30	0.393896	0.491529	0.109440	0.056*
C31	0.3415 (3)	0.55210 (10)	0.0211 (7)	0.0910 (14)
H31A	0.327824	0.575805	−0.001944	0.137*
H31B	0.398235	0.544226	−0.054105	0.137*
H31C	0.362293	0.549285	0.141822	0.137*
C32	0.2415 (3)	0.53171 (8)	−0.0143 (4)	0.0489 (7)
C33	0.2465 (2)	0.49404 (7)	0.0009 (3)	0.0375 (6)
C34	0.16419 (18)	0.47225 (7)	−0.0532 (3)	0.0330 (6)
C35	0.1720 (2)	0.43761 (7)	−0.0424 (4)	0.0389 (6)
H35	0.115912	0.423881	−0.081246	0.047*
C36	0.2642 (2)	0.42295 (7)	0.0269 (4)	0.0431 (7)
C37	0.3464 (2)	0.44332 (9)	0.0845 (4)	0.0484 (7)
H37	0.407845	0.433631	0.132521	0.058*
C38	0.3552 (3)	0.37151 (11)	0.1028 (5)	0.0833 (13)
H38A	0.418811	0.379545	0.045107	0.125*
H38B	0.348511	0.347313	0.085809	0.125*
H38C	0.359685	0.376380	0.226411	0.125*
C39	0.000000	0.500000	−0.0304 (5)	0.0336 (8)
O1	0.53243 (17)	0.12717 (5)	0.2414 (3)	0.0588 (6)
O2	0.64969 (17)	0.28509 (5)	0.2060 (3)	0.0593 (6)
O3	0.73369 (13)	0.22393 (4)	0.1066 (2)	0.0339 (4)
O4	0.80873 (14)	0.23938 (5)	0.3655 (2)	0.0406 (4)
O5	0.87108 (13)	0.25650 (4)	0.1008 (2)	0.0351 (4)
O6	1.00302 (17)	0.20357 (5)	0.1858 (3)	0.0585 (6)
O7	1.0074 (2)	0.36685 (6)	0.2458 (3)	0.0649 (6)
O8	0.6523 (4)	0.35828 (9)	0.2314 (7)	0.1150 (12)
O9	0.6594 (4)	0.39111 (9)	0.5608 (6)	0.1249 (14)
O10	0.5910 (3)	0.36449 (9)	0.8789 (5)	0.1094 (11)
O11	0.67800 (19)	0.46331 (5)	0.5623 (4)	0.0656 (7)
O12	0.56314 (13)	0.51836 (5)	0.6613 (2)	0.0367 (4)
O13	0.500000	0.500000	0.3996 (3)	0.0518 (8)
O14	0.72361 (16)	0.62566 (5)	0.5325 (3)	0.0507 (5)
O15	0.1596 (2)	0.54623 (6)	−0.0570 (4)	0.0734 (7)
O16	0.07183 (13)	0.48503 (5)	−0.1354 (2)	0.0379 (4)
O17	0.000000	0.500000	0.1256 (3)	0.0465 (7)
O18	0.26413 (19)	0.38819 (6)	0.0307 (3)	0.0668 (6)
H8A	0.658 (7)	0.3645 (19)	0.123 (5)	0.19 (4)*
H8B	0.655 (4)	0.3366 (7)	0.238 (7)	0.13 (2)*
H9D	0.638 (5)	0.3882 (17)	0.452 (4)	0.16 (3)*
H9E	0.667 (4)	0.4124 (7)	0.562 (7)	0.119 (18)*
H10A	0.630 (6)	0.374 (2)	0.793 (8)	0.22 (4)*
H10B	0.568 (19)	0.345 (3)	0.86 (2)	1.0 (12)*

The structure of diphenylcarbonate have been reported, in which a diphenylcarbonate structure, a 2-nitrophenyl phenoxyformate structure, a bis(*o*-nitrophenyl) carbonate structure and a bis(2-methoxyphenyl) carbonate structure were described [10–13].

The asymmetric unit contains one complete organic molecule, two halves of that molecule, located on twofold axis and three water molecules. The title compound bis(2-acetyl-

5-methoxyphenyl) carbonate is shown in the figure, with two paeonol fragments connected by carbonyl groups. Based on the diphenylcarbonate structure [10], there is an acetyl and a methoxy at each phenyl group. The phenyl rings are bent towards the double bonded oxygen atom on the central carbon atom. Moreover, the mean bond angles for $O=C-O$, $O-C-O$, $C-C=O$ and $C-O-C$ are 127.15° , 105.66° , 120.78° and 117.94° respectively, closely resembling to the reported ones [12]. The ester bond lengths of $C8-O2$, $C17-O6$ and $C10-O4$ are 1.220, 1.223 and 1.182 Å (consistent with [11]). The average ether bond length is 1.381 Å, moreover, the $C-O$ ether bond lengths are similar to the reported ones in [11, 13].

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: Natural Science Foundation for Colleges and Universities of Jiangsu Province (No. 18KJB350005), Science & Technology Funds of Kangda College of Nanjing Medical University (Nos. KD2019KYJJZD002 & KD2020 KYJJZD002), Science & Technology Funds of Nanjing Medical University (No. NMUB2019272).

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

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