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The crystal structure of dimethyl ((3,5-di-*tert*-butyl-4-hydroxyphenyl)(phenyl)methyl) phosphonate, C₂₃H₃₃O₄P

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Abstract

C₂₃H₃₃O₄P, monoclinic, *P*2₁/*c* (no. 14), *a* = 10.4629(3) Å, *b* = 11.4909(5) Å, *c* = 19.3341(9) Å, β = 98.201(4)°, *V* = 2300.73(16) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0550, *wR*_{ref}(*F*²) = 0.1109, *T* = 298 K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colourless plate
Size:	0.20 × 0.20 × 0.05 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.14 mm ^{−1}
Diffractometer, scan mode:	SuperNova, ω
θ _{max} , completeness:	27.5°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	20317, 5187, 0.035
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 3987
<i>N</i> (<i>param</i>) _{refined} :	262
Programs:	CrysAlis ^{PRO} [1], Olex2 [2], SHELX [3, 4]

Source of material

To a stirring solution of 4-benzylidene-2,6-di-*tert*-butylcyclohexa-2,5-dienol (237 mg, 0.8 mmol) in anhydrous DMSO (4 mL) were added dimethyl phosphonate (106 mg, 0.96 mmol) and 1,3-dimesityl-1*H*-imidazol-3-ium-2-carboxylate (28 mg, 0.08 mmol). After completion of the reaction monitored by TLC, the reaction mixture was quenched by 60 mL water and extracted with dichloromethane (25 × 3 mL). The organic phase was dried over sodium sulfate, filtered, and concentrated. The residue was then purified by flash chromatography (petroleum ether/ethylacetate) on silica gel to afford the title compound as white solid in yield 78%.

Experimental details

All hydrogen atoms were placed geometrically on calculated positions using a riding model with the help of the SHELX program.

Comment

Organophosphorus compounds have generated considerable interest in the area of organometallic chemistry and organic synthesis for their widely applications as ligands [5], catalysts [6] and Wittig and Horner-Wadsworth - Emmons (HWE) reagents [7]. Among them, diarylmethyl phosphonate compounds and their derivatives have been widely used in drug discovery [8] and materials chemistry. For example,

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}
P1	0.32594(5)	0.30152(5)	0.21898(3)	0.03518(14)
O1	0.18530(13)	0.28946(14)	0.21144(8)	0.0479(4)
O2	0.37597(14)	0.42625(14)	0.24274(9)	0.0513(4)
O3	0.40435(15)	0.22056(15)	0.27532(8)	0.0519(4)
O4	0.93552(12)	0.23334(15)	0.14154(8)	0.0451(4)
H4	0.9774	0.2705	0.1732	0.068*
C1	0.2988(3)	0.5297(2)	0.22976(16)	0.0672(8)
H1A	0.2541	0.5280	0.1828	0.101*
H1B	0.3537	0.5970	0.2358	0.101*
H1C	0.2372	0.5332	0.2620	0.101*
C2	0.3773(4)	0.2197(4)	0.34635(15)	0.0958(12)
H2A	0.2880	0.2012	0.3468	0.144*
H2B	0.3957	0.2950	0.3669	0.144*
H2C	0.4304	0.1623	0.3727	0.144*
C3	0.39252(17)	0.26411(17)	0.14027(10)	0.0305(4)
H3	0.3619	0.1852	0.1279	0.037*
C4	0.32780(18)	0.34304(18)	0.08152(11)	0.0358(5)
C5	0.2088(2)	0.3148(3)	0.04501(14)	0.0584(7)
H5	0.1686	0.2460	0.0552	0.070*
C6	0.1489(3)	0.3874(3)	−0.00650(17)	0.0813(10)
H6	0.0688	0.3670	−0.0307	0.098*
C7	0.2061(3)	0.4889(3)	−0.02229(16)	0.0751(9)
H7	0.1650	0.5379	−0.0568	0.090*
C8	0.3245(3)	0.5177(3)	0.01322(16)	0.0687(8)
H8	0.3644	0.5866	0.0027	0.082*
C9	0.3852(2)	0.4448(2)	0.06470(14)	0.0526(6)
H9	0.4660	0.4649	0.0882	0.063*
C10	0.53969(17)	0.25939(17)	0.14481(10)	0.0285(4)
C11	0.59132(17)	0.19057(17)	0.09672(10)	0.0298(4)
H11	0.5353	0.1485	0.0643	0.036*
C12	0.72341(17)	0.18197(17)	0.09501(10)	0.0280(4)
C13	0.77718(18)	0.1045(2)	0.04080(10)	0.0366(5)
C14	0.6691(2)	0.0424(2)	−0.00725(13)	0.0568(7)
H14A	0.6210	−0.0059	0.0203	0.085*
H14B	0.7061	−0.0049	−0.0402	0.085*
H14C	0.6126	0.0991	−0.0319	0.085*
C15	0.8523(2)	0.1777(3)	−0.00610(13)	0.0587(7)
H15A	0.7964	0.2360	−0.0296	0.088*
H15B	0.8836	0.1283	−0.0400	0.088*
H15C	0.9240	0.2147	0.0220	0.088*
C16	0.8637(2)	0.0086(2)	0.07769(14)	0.0605(7)
H16A	0.9378	0.0434	0.1050	0.091*
H16B	0.8915	−0.0423	0.0434	0.091*
H16C	0.8160	−0.0351	0.1077	0.091*
C17	0.80619(16)	0.24589(17)	0.14518(10)	0.0285(4)
C18	0.75806(17)	0.31597(16)	0.19541(10)	0.0271(4)
C19	0.84663(18)	0.38253(17)	0.25277(10)	0.0316(4)
C20	0.7678(2)	0.4511(2)	0.30057(12)	0.0479(6)
H20A	0.7120	0.5051	0.2730	0.072*
H20B	0.8255	0.4928	0.3349	0.072*
H20C	0.7168	0.3980	0.3235	0.072*
C21	0.9307(2)	0.2955(2)	0.29965(11)	0.0405(5)
H21A	0.8761	0.2420	0.3199	0.061*
H21B	0.9838	0.3370	0.3361	0.061*
H21C	0.9845	0.2533	0.2721	0.061*

Table 2 (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C22	0.9297(2)	0.4726(2)	0.22069(12)	0.0441(5)
H22A	0.9836	0.4335	0.1918	0.066*
H22B	0.9829	0.5135	0.2574	0.066*
H22C	0.8746	0.5268	0.1929	0.066*
C23	0.62414(17)	0.32108(17)	0.19280(10)	0.0296(4)
H23	0.5902	0.3682	0.2248	0.035*

dimethyl (bis(4-fluorophenyl)(isopropoxy) methyl) phosphonate is known as an important leukocyte elastase inhibitor [9]. Ethyl 4-((4-oxo-1-((1-(*p*-tolyl)butyl)carbamoyl)azetidin-2-yl)oxy) benzylphosphinate is a potassium channel modulator [10]. In addition, diarylmethyl phosphonates also have been utilized to the synthesis of flame retardants [11] and chemiluminescence materials [12]. Our group has maintained a keen interest in the preparation of the sulfone [13, 14] and diarylmethyl derivatives [15]. Herein, we report the crystal structure of dimethyl ((3,5-di-*tert*-butyl-4-hydroxyphenyl)(phenyl)methyl)phosphonate.

The geometry and labeling for the crystal structure of the title complex is depicted in the figure. The title structure crystallizes in monoclinic space group *P*2₁/*c* with one molecule in the asymmetric unit. There is at least an intermolecular O4—H4···O1 hydrogen bond interaction (*d*(O1'···H4) = 2.206 Å, *d*(O4—H4) = 0.82 Å and *d*(O4···O1') = 2.840 Å; ' = 1 + *x*, *y*, *z*) in the title structure. Hydrogen bonds connect individual molecules to a chain along the [100] direction.

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