Crystal structure of 1-[2,4,6-tris(benzoyloxy)phenyl]ethanone, $C_{29}H_{20}O_7$

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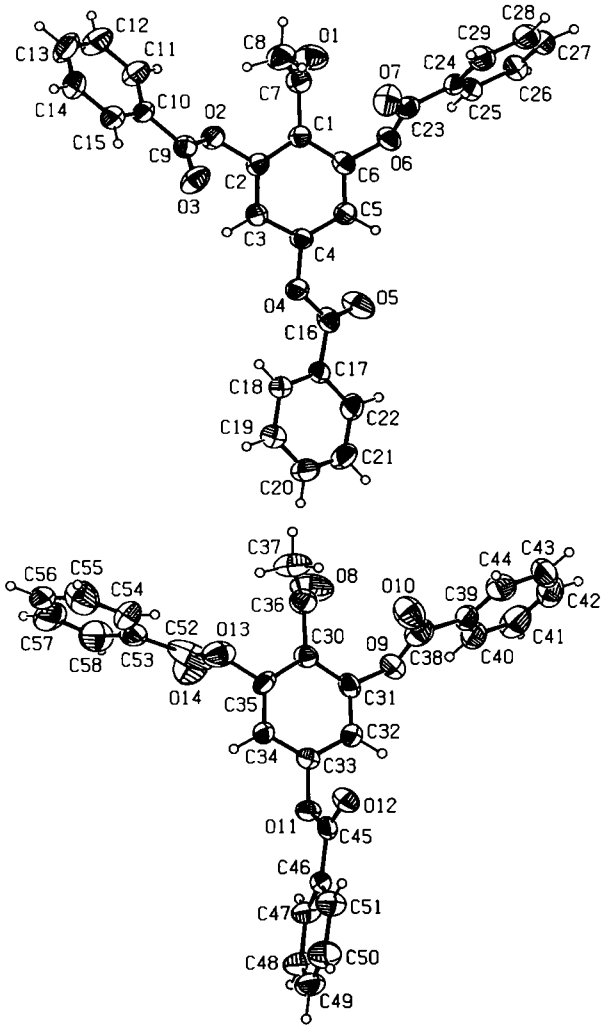

Abstract

$C_{29}H_{20}O_7$, monoclinic, $P12_1/c1$ (no. 14), a = 9.583(4) Å, b = 25.30(1) Å, c = 19.975(9) Å, β = 96.732(8)°, V = 4809.0 Å^3, Z = 8, $R_{gt}(F)$ = 0.071, $wR_{ref}(F^2)$ = 0.189, T = 298 K.

Source of material

A solution of 2,4,6-trihydroxyacetophenone (1.0 g, 5.95 mmol) in acetone (50 ml, in a flask) was stirred in a water bath and then mixed with potassium carbonate (2.5 g, 18.12 mmol) and benzoyl chloride (3.0 g, 21.35 mmol). After being stirred for 0.5 h, the reaction mixture was filtered and the filtrate was distilled. The leftover was recrystallized from ethanol to give white crystals of 1-[2,4,6-tris(benzoyloxy)phenyl]ethanone (yield 2.3 g, 80.5 %).

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Experimental details

The small N_{gt}/N_{param} ratio and the large R values are due to the small scattering power of the constituents of the crystal structure.

Discussion

The title compound was obtained during a study of the synthesis of 5,7-dihydroxyflavone. Two independent molecules form the asymmetric unit. They differ in the conformations of the benzoyloxy groups. In molecule C1 to C29, the angles between phenyl group (C1 to C6) and benzoyloxy groups (O2, C9 to C15), (O4, C16 to C22) and (O6, C23 to C29) are 55.3°, 29.8° and 65.9°, respectively. In molecule C30 to C58, three benzoyloxy groups are almost perpendicular with phenyl group (C30 to C35), whereas the angles between phenyl group (C30 to C35) and benzoyloxy groups (O9, C38 to C44), (O11, C45 to C51) and (O13, C52 to C58) are 71.9°, 87.2° and 92.9°, respectively. Both molecules are connected by hydrogen bridges C32–H32···O3 (d(C32···O3) = 3.464(9) Å, ∠C32–H32···O3 = 146.1°) and C3–H3···O12 (d(C3···O12) = 3.323(6) Å, ∠C3–H3···O12 = 150.1°). Moreover, three intramolecular hydrogen bridges, C8–H8B···O7, C8–H8C···O2 and C37–H37C···O13, are formed: (d(C8···O7) = 3.083(9) Å, d(C8···O2) = 3.038(3) Å, d(C37···O13) = 3.057(9) Å and ∠C8–H8B···O7 = 117.2°, ∠C8–H8C···O2 = 118.2°, ∠C37–H37C···O13 = 121.9°). Via three other intermolecular hydrogen bridges, C34–H34···O5, C44–H44···O14 and C50–H50···O8 (d(C34···O5) = 3.206(4) Å, d(C44···O14) = 3.184(5) Å, d(C50···O8) = 3.434(4) Å, ∠C34–H34···O5 = 160.6°, ∠C44–H44···O14 = 145.9°, and ∠C50–H50···O8 = 166.1°), the title compound forms layers parallel to a,c plane. Furthermore, two C–H···π bonds are observed in the asymmetric unit. The first one exists between H18 and phenyl ring C30 to C35 (d(H18···centroid ring) = 3.089 Å) and the second one exists between H37B and phenyl ring C17 to C22 (d(H37B···centroid ring) = 3.348 Å). The other two C–H···π bond are H42 with phenyl ring C46^{i} to C59^{i} (d(H42···centroid ring) = 3.348 Å) and H47 with phenyl ring C1^{ii} to C6^{ii} (d(H47···centroid ring) = 3.117 Å) (symmetry codes i: $-1+x,\frac{1}{2}-y,\frac{1}{2}-z$; ii: $1+x,y,z$). The hydrogen bridges and C–H···π bonds in the title structure result in the formation of 3D network. 1-[2,4,6-tris(benzoyloxy)phenyl]propanone [1] crystallizes isotypically to the title compound.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.15 × 0.18 × 0.38 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.95 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.06°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	25093, 8490
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2882
$N(param)_{refined}$:	649
Program:	SHELXTL [2]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(3)	4e	0.1828	0.1930	0.3228	0.077
H(5)	4e	−0.1120	0.2708	0.4098	0.074
H(8A)	4e	0.0491	0.0467	0.5441	0.144
H(8B)	4e	0.1117	0.1036	0.5568	0.144
H(8C)	4e	0.1604	0.0682	0.4993	0.144
H(11)	4e	0.1927	0.0091	0.3797	0.107
H(12)	4e	0.2984	−0.0724	0.3808	0.143
H(13)	4e	0.5381	−0.0797	0.4021	0.140
H(14)	4e	0.6729	−0.0055	0.4250	0.119
H(15)	4e	0.5667	0.0761	0.4201	0.094
H(18)	4e	0.2589	0.3322	0.2794	0.085
H(19)	4e	0.3680	0.4015	0.2325	0.101
H(20)	4e	0.2442	0.4765	0.2003	0.119
H(21)	4e	0.0137	0.4829	0.2144	0.125
H(22)	4e	−0.1023	0.4129	0.2601	0.108
H(25)	4e	−0.4372	0.1941	0.4918	0.096
H(26)	4e	−0.6421	0.1992	0.5438	0.115
H(27)	4e	−0.6262	0.1993	0.6595	0.131
H(28)	4e	−0.4182	0.1877	0.7219	0.133
H(29)	4e	−0.2113	0.1845	0.6718	0.109

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(32)	4e	0.3669	0.2479	0.4277	0.077
H(34)	4e	0.6507	0.3312	0.3406	0.079
H(37A)	4e	0.2953	0.4957	0.4545	0.174
H(37B)	4e	0.2335	0.4536	0.4016	0.174
H(37C)	4e	0.3819	0.4782	0.3964	0.174
H(40)	4e	0.2252	0.2911	0.6092	0.115
H(41)	4e	0.0950	0.2715	0.6944	0.147
H(42)	4e	−0.1350	0.2930	0.6860	0.168
H(43)	4e	−0.2434	0.3320	0.5889	0.159
H(44)	4e	−0.1081	0.3525	0.5014	0.125
H(47)	4e	0.7113	0.1596	0.3476	0.093
H(48)	4e	0.8155	0.0872	0.3039	0.124
H(49)	4e	0.7316	0.0558	0.1996	0.111
H(50)	4e	0.5431	0.0956	0.1394	0.120
H(51)	4e	0.4324	0.1651	0.1856	0.101
H(54)	4e	0.6162	0.4976	0.2995	0.143
H(55)	4e	0.7259	0.5666	0.2549	0.167
H(56)	4e	0.9408	0.5944	0.3024	0.185
H(57)	4e	1.0418	0.5526	0.4001	0.196
H(58)	4e	0.9314	0.4797	0.4404	0.151

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	4e	−0.1449(4)	0.0796(1)	0.4651(2)	0.081(3)	0.098(3)	0.128(3)	−0.024(2)	0.008(3)	0.021(2)
O(2)	4e	0.1665(4)	0.1034(1)	0.3847(2)	0.056(2)	0.059(2)	0.099(2)	−0.002(2)	0.011(2)	−0.001(2)
O(3)	4e	0.3723(4)	0.1439(1)	0.4114(2)	0.067(3)	0.060(2)	0.158(4)	−0.001(2)	−0.006(2)	−0.008(2)
O(4)	4e	0.0810(3)	0.2818(1)	0.3254(2)	0.058(2)	0.064(2)	0.089(2)	0.012(2)	0.017(2)	0.020(2)
O(5)	4e	−0.1297(4)	0.3196(2)	0.3055(2)	0.058(3)	0.157(4)	0.131(3)	0.025(3)	0.020(2)	0.074(3)
O(6)	4e	−0.1951(3)	0.1973(1)	0.4786(2)	0.067(2)	0.088(2)	0.059(2)	0.008(2)	0.014(2)	0.015(2)
O(7)	4e	−0.0597(4)	0.1765(2)	0.5747(2)	0.101(3)	0.117(3)	0.074(3)	0.026(3)	−0.014(2)	−0.001(2)
O(8)	4e	0.3970(5)	0.4283(2)	0.5301(2)	0.197(5)	0.137(4)	0.076(3)	0.057(3)	0.016(3)	−0.024(3)
O(9)	4e	0.2804(5)	0.3180(1)	0.4991(2)	0.083(3)	0.086(3)	0.149(4)	0.012(2)	0.053(3)	0.008(2)
O(10)	4e	0.1106(4)	0.3643(2)	0.4430(2)	0.109(4)	0.146(4)	0.102(3)	0.014(3)	0.021(3)	0.014(3)
O(11)	4e	0.5654(3)	0.2363(1)	0.3522(2)	0.062(2)	0.057(2)	0.078(2)	0.011(2)	0.002(2)	−0.008(2)
O(12)	4e	0.3824(4)	0.2340(1)	0.2728(2)	0.063(2)	0.088(3)	0.081(2)	0.017(2)	0.010(2)	0.004(2)
O(13)	4e	0.5808(5)	0.4243(2)	0.3719(3)	0.087(3)	0.090(3)	0.162(5)	0.014(3)	−0.024(3)	−0.026(3)
O(14)	4e	0.7408(6)	0.4209(2)	0.4555(3)	0.169(5)	0.145(4)	0.188(5)	−0.025(4)	−0.016(4)	0.021(4)
C(1)	4e	−0.0047(5)	0.1493(2)	0.4359(2)	0.047(3)	0.058(3)	0.071(3)	0.003(3)	0.003(3)	0.008(3)
C(2)	4e	0.0943(5)	0.1508(2)	0.3922(2)	0.044(3)	0.057(3)	0.082(4)	0.004(3)	0.005(3)	0.003(3)
C(3)	4e	0.1169(5)	0.1940(2)	0.3534(2)	0.053(3)	0.064(3)	0.076(3)	0.003(3)	0.015(3)	0.005(3)
C(4)	4e	0.0395(5)	0.2389(2)	0.3610(2)	0.052(3)	0.060(3)	0.068(3)	−0.001(3)	0.009(3)	0.012(3)
C(5)	4e	−0.0601(5)	0.2403(2)	0.4045(2)	0.057(3)	0.064(3)	0.064(3)	0.006(3)	0.006(3)	0.007(3)
C(6)	4e	−0.0811(5)	0.1952(2)	0.4403(2)	0.046(3)	0.077(4)	0.055(3)	−0.001(3)	0.003(3)	0.006(3)
C(7)	4e	−0.0325(6)	0.0997(2)	0.4746(3)	0.062(4)	0.075(4)	0.084(4)	−0.005(3)	0.007(3)	0.003(3)
C(8)	4e	0.0823(6)	0.0776(2)	0.5230(3)	0.096(4)	0.086(4)	0.105(4)	0.012(3)	0.007(4)	0.022(3)
C(9)	4e	0.3079(6)	0.1036(2)	0.3993(2)	0.063(4)	0.062(4)	0.070(3)	−0.003(3)	0.010(3)	−0.001(3)
C(10)	4e	0.3700(6)	0.0509(2)	0.3991(2)	0.063(4)	0.048(3)	0.077(3)	0.008(3)	0.010(3)	0.005(3)
C(11)	4e	0.2899(6)	0.0064(2)	0.3880(3)	0.093(5)	0.060(4)	0.114(5)	0.004(4)	0.007(4)	0.000(3)
C(12)	4e	0.3530(9)	−0.0422(2)	0.3890(3)	0.143(7)	0.060(5)	0.153(6)	0.001(5)	0.012(6)	−0.005(4)
C(13)	4e	0.4958(9)	−0.0466(3)	0.4020(3)	0.149(8)	0.067(5)	0.136(6)	0.041(5)	0.021(6)	−0.004(4)
C(14)	4e	0.5760(7)	−0.0027(3)	0.4148(3)	0.099(5)	0.087(5)	0.115(5)	0.036(5)	0.030(4)	0.014(4)
C(15)	4e	0.5119(6)	0.0459(2)	0.4124(2)	0.075(4)	0.068(4)	0.093(4)	0.008(3)	0.018(3)	0.010(3)
C(16)	4e	−0.0070(6)	0.3213(2)	0.3026(2)	0.055(4)	0.091(4)	0.062(3)	0.015(4)	0.009(3)	0.010(3)
C(17)	4e	0.0681(6)	0.3653(2)	0.2736(2)	0.071(4)	0.065(4)	0.055(3)	0.012(3)	0.017(3)	0.011(3)
C(18)	4e	0.2080(6)	0.3624(2)	0.2658(2)	0.076(4)	0.064(4)	0.076(3)	0.008(3)	0.022(3)	0.013(3)
C(19)	4e	0.2733(6)	0.4039(2)	0.2382(2)	0.092(4)	0.079(4)	0.086(4)	0.002(4)	0.025(3)	0.013(3)
C(20)	4e	0.1996(8)	0.4484(2)	0.2191(3)	0.136(6)	0.070(4)	0.094(4)	0.000(4)	0.028(4)	0.013(3)
C(21)	4e	0.0626(8)	0.4521(2)	0.2273(3)	0.143(7)	0.070(4)	0.102(5)	0.042(4)	0.025(5)	0.022(4)
C(22)	4e	−0.0075(6)	0.4104(2)	0.2547(3)	0.092(4)	0.089(4)	0.091(4)	0.041(4)	0.026(3)	0.021(3)
C(23)	4e	−0.1719(7)	0.1868(2)	0.5461(3)	0.096(5)	0.054(3)	0.062(4)	0.003(3)	0.009(4)	−0.002(3)
C(24)	4e	−0.3063(7)	0.1889(2)	0.5766(3)	0.098(5)	0.054(3)	0.055(4)	0.015(3)	0.021(3)	0.008(3)
C(25)	4e	−0.4324(7)	0.1933(2)	0.5386(3)	0.098(5)	0.076(4)	0.072(4)	0.013(3)	0.032(4)	0.015(3)
C(26)	4e	−0.5550(6)	0.1965(2)	0.5696(3)	0.104(5)	0.091(4)	0.100(5)	0.016(3)	0.045(4)	0.015(4)
C(27)	4e	−0.5453(9)	0.1958(2)	0.6383(4)	0.167(8)	0.080(4)	0.095(6)	0.022(5)	0.073(5)	0.016(4)
C(28)	4e	−0.422(1)	0.1900(2)	0.6753(3)	0.198(9)	0.081(5)	0.060(4)	0.014(5)	0.047(5)	0.008(3)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(29)	4e	−0.2978(7)	0.1873(2)	0.6456(3)	0.136(6)	0.079(4)	0.061(4)	0.000(4)	0.022(4)	0.000(3)
C(30)	4e	0.4268(5)	0.3743(2)	0.4381(2)	0.078(4)	0.064(4)	0.054(3)	0.008(3)	0.023(3)	0.003(3)
C(31)	4e	0.3707(5)	0.3252(2)	0.4497(2)	0.066(3)	0.077(4)	0.063(3)	0.000(3)	0.029(3)	0.005(3)
C(32)	4e	0.4098(5)	0.2800(2)	0.4201(2)	0.068(4)	0.056(3)	0.070(3)	−0.004(3)	0.017(3)	0.003(3)
C(33)	4e	0.5144(5)	0.2828(2)	0.3787(2)	0.057(3)	0.060(4)	0.061(3)	0.006(3)	0.006(3)	−0.005(3)
C(34)	4e	0.5771(5)	0.3295(2)	0.3670(2)	0.071(4)	0.056(3)	0.074(3)	−0.009(3)	0.029(3)	−0.008(3)
C(35)	4e	0.5284(5)	0.3735(2)	0.3952(3)	0.086(4)	0.042(3)	0.084(4)	−0.014(3)	0.019(3)	0.001(3)
C(36)	4e	0.3830(6)	0.4245(2)	0.4696(3)	0.096(4)	0.080(4)	0.077(4)	0.001(4)	0.021(4)	−0.012(4)
C(37)	4e	0.3178(7)	0.4666(2)	0.4269(3)	0.168(6)	0.078(4)	0.105(5)	0.039(4)	0.023(4)	−0.003(4)
C(38)	4e	0.1588(8)	0.3373(3)	0.4918(3)	0.120(6)	0.077(5)	0.086(5)	−0.011(4)	0.013(5)	−0.009(4)
C(39)	4e	0.0704(8)	0.3237(2)	0.5479(3)	0.104(6)	0.071(4)	0.078(4)	−0.021(4)	0.046(4)	−0.018(3)
C(40)	4e	0.1302(7)	0.2997(2)	0.6049(4)	0.114(5)	0.083(4)	0.099(5)	−0.022(4)	0.045(5)	−0.019(4)
C(41)	4e	0.053(1)	0.2881(3)	0.6557(3)	0.188(8)	0.098(5)	0.091(5)	−0.034(6)	0.052(6)	−0.012(4)
C(42)	4e	−0.083(1)	0.3005(3)	0.6505(5)	0.17(1)	0.125(7)	0.142(8)	−0.059(7)	0.097(9)	−0.055(6)
C(43)	4e	−0.1480(8)	0.3241(3)	0.5930(5)	0.094(6)	0.128(7)	0.188(8)	−0.029(5)	0.065(7)	−0.073(6)
C(44)	4e	−0.0671(9)	0.3361(2)	0.5406(3)	0.105(6)	0.095(5)	0.114(5)	−0.005(4)	0.018(5)	−0.031(4)
C(45)	4e	0.4909(6)	0.2152(2)	0.2967(3)	0.052(3)	0.062(4)	0.067(4)	−0.002(3)	0.017(3)	0.009(3)
C(46)	4e	0.5625(5)	0.1698(2)	0.2708(3)	0.051(3)	0.053(3)	0.066(3)	0.000(3)	0.008(3)	−0.001(3)
C(47)	4e	0.6757(6)	0.1464(2)	0.3056(3)	0.087(4)	0.061(3)	0.082(4)	0.013(3)	−0.001(3)	−0.014(3)
C(48)	4e	0.7388(6)	0.1033(2)	0.2793(3)	0.107(5)	0.079(4)	0.115(5)	0.026(4)	−0.020(4)	−0.024(4)
C(49)	4e	0.6887(7)	0.0846(2)	0.2176(3)	0.113(5)	0.067(4)	0.100(5)	0.007(4)	0.020(4)	−0.020(4)
C(50)	4e	0.5765(7)	0.1079(3)	0.1821(3)	0.123(6)	0.101(5)	0.073(4)	0.007(4)	0.002(4)	−0.026(4)
C(51)	4e	0.5117(5)	0.1500(2)	0.2094(3)	0.080(4)	0.086(4)	0.083(4)	0.013(3)	−0.008(3)	−0.014(3)
C(52)	4e	0.681(1)	0.4347(4)	0.4026(3)	0.141(8)	0.21(1)	0.055(5)	0.123(9)	−0.011(5)	0.018(5)
C(53)	4e	0.765(1)	0.4838(2)	0.3715(5)	0.109(7)	0.062(4)	0.126(6)	0.026(5)	0.053(5)	0.034(5)
C(54)	4e	0.7044(7)	0.5084(3)	0.3191(5)	0.109(6)	0.079(5)	0.172(8)	−0.028(5)	0.023(7)	−0.025(5)
C(55)	4e	0.769(1)	0.5495(4)	0.2930(4)	0.148(9)	0.16(1)	0.116(6)	0.018(7)	0.038(6)	0.029(6)
C(56)	4e	0.896(1)	0.5664(3)	0.3210(7)	0.16(1)	0.058(5)	0.27(1)	0.008(6)	0.15(1)	0.015(7)
C(57)	4e	0.9564(9)	0.5409(4)	0.3783(7)	0.103(7)	0.097(7)	0.29(1)	−0.026(6)	0.000(8)	−0.079(8)
C(58)	4e	0.890(1)	0.4981(4)	0.4029(4)	0.112(7)	0.127(8)	0.133(6)	0.007(6)	−0.007(6)	−0.018(5)

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