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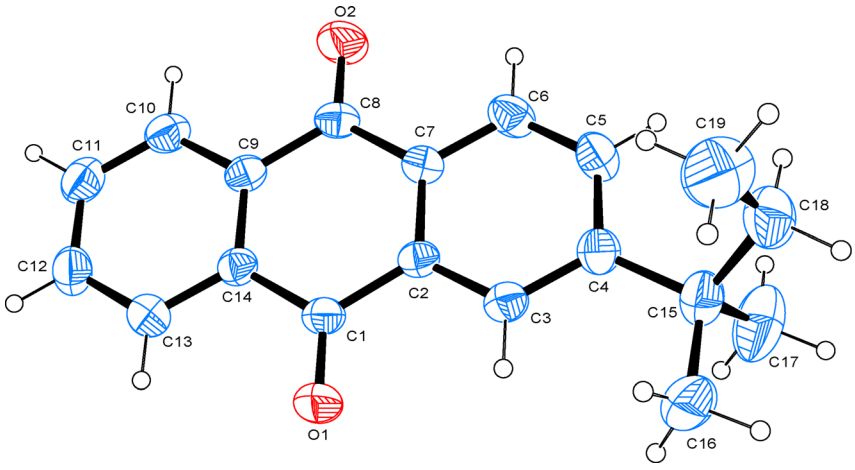
Crystal structure of 2-(*tert*-pentyl)anthracene-9,10-dione, $C_{19}H_{18}O_2$



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Abstract

$C_{19}H_{18}O_2$, orthorhombic, *Pbca* (no. 61), a = 11.017(5) Å, b = 12.140(5) Å, c = 23.346(10) Å, V = 3122(2) Å^3, Z = 8, R_{gt}(F) = 0.0498, wR_{ref}(F^2) = 0.1450, T = 296(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.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.20 × 0.18 × 0.18 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.08 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	15,220, 2884, 0.027
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 1935
$N(param)_{refined}$:	224
Programs:	Bruker [1], SHELX [2, 3], WinGX/ORTEP [4]

Source of material

All chemicals were purchased from commercial sources and used as received. The 8.9 g anthracene (0.05 mol) and 10.0 g molecular sieve (40H) were added in a 150 mL three-way flask with 80 mL mesitylene. The temperature is controlled at about 120 °C, and slowly 2.2 g *tert*-amyl alcohol (0.025 mol) mesitylene solution was added, and kept at 160 °C for 6–8 h. Take advantage of hot filtration, the white solid 2-(*tert*-pentyl)anthracene is obtained. The yield is 65%. The 4.96 g 2-(*tert*-pentyl)anthracene (0.02 mol), 3 mL acetic acid and 8 mL ethyl acetate were added to the reactor, heated to 90 °C. Finally hydrogen peroxide (50%, 12 ml) was slowly added and the reaction mixture was further reacted for 4h to obtain the 2-(*tert*-pentyl)anthracene-9,10-dione solid. The yellow crystals of the title compound recrystallized from *n*-hexane. The yield is 50%.

Experimental details

All H-atoms were placed geometrically and refined using a riding model with common isotropic displacement factors U_{iso}(H) = 1.2 or 1.5 U_{eq}(parent C-atom).

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

Atom	x	y	z	U_{iso}*/U_{eq}
O1	0.49212 (14)	0.33389 (12)	0.62706 (8)	0.0839 (5)
C1	0.49573 (18)	0.42674 (15)	0.60709 (8)	0.0533 (5)
O2	0.51095 (16)	0.74044 (13)	0.53772 (8)	0.0910 (6)
C2	0.60212 (17)	0.49941 (14)	0.61745 (8)	0.0474 (5)
C3	0.69763 (18)	0.46269 (17)	0.65176 (8)	0.0558 (5)
H3	0.692763	0.393076	0.668228	0.067*
C4	0.79963 (19)	0.52674 (18)	0.66206 (9)	0.0593 (5)
C5	0.8032 (2)	0.63060 (19)	0.63634 (10)	0.0686 (6)
H5	0.871107	0.674952	0.641710	0.082*
C6	0.7090 (2)	0.66886 (18)	0.60330 (9)	0.0660 (6)
H6	0.713799	0.738839	0.587254	0.079*
C7	0.60671 (18)	0.60467 (15)	0.59350 (8)	0.0498 (5)
C8	0.50621 (19)	0.64799 (16)	0.55836 (8)	0.0564 (5)
C9	0.39900 (18)	0.57665 (15)	0.54911 (8)	0.0505 (5)
C10	0.3014 (2)	0.61570 (18)	0.51725 (9)	0.0654 (6)
H10	0.304269	0.686332	0.501888	0.078*
C11	0.2006 (2)	0.5512 (2)	0.50817 (11)	0.0735 (7)
H11	0.135758	0.578192	0.486897	0.088*
C12	0.1963 (2)	0.4467 (2)	0.53070 (11)	0.0737 (7)
H12	0.128326	0.402884	0.524405	0.088*
C13	0.29203 (19)	0.40616 (18)	0.56263 (10)	0.0658 (6)
H13	0.288080	0.335371	0.577741	0.079*
C14	0.39414 (17)	0.47071 (15)	0.57224 (8)	0.0498 (5)
C15	0.9055 (2)	0.4881 (2)	0.69895 (11)	0.0784 (7)
C16[a]	0.8761 (3)	0.3820 (2)	0.73185 (13)	0.1049 (12)
H16A[a]	0.804345	0.392875	0.754479	0.157*
H16B[a]	0.942870	0.363574	0.756476	0.157*
H16C[a]	0.862689	0.323042	0.705161	0.157*
C17[a]	1.0134 (3)	0.4646 (3)	0.65823 (14)	0.1323 (17)
H17A[a]	0.991963	0.406436	0.632302	0.198*
H17B[a]	1.082950	0.442988	0.680305	0.198*
H17C[a]	1.032239	0.529982	0.636831	0.198*
C18[a]	0.9430 (3)	0.5779 (2)	0.74125 (12)	0.0968 (11)
H18A[a]	1.011448	0.551330	0.763408	0.116*
H18B[a]	0.970234	0.641605	0.719708	0.116*
C19[a]	0.8446 (4)	0.6143 (3)	0.78224 (13)	0.1225 (12)
H19A[a]	0.775730	0.640142	0.760892	0.184*
H19B[a]	0.874769	0.672629	0.806053	0.184*
H19C[a]	0.820665	0.553195	0.805783	0.184*
C17A[b]	1.0245 (15)	0.5540 (18)	0.6926 (12)	0.101 (11)
H17D[b]	1.038309	0.570157	0.652894	0.151*
H17E[b]	1.090932	0.511180	0.707225	0.151*
H17F[b]	1.018345	0.621532	0.713776	0.151*
C18A[b]	0.859 (2)	0.5009 (19)	0.7610 (6)	0.094 (8)
H18C[b]	0.781305	0.464006	0.763889	0.112*
H18D[b]	0.915039	0.462717	0.786205	0.112*
C16A[b]	0.932 (3)	0.3656 (9)	0.6876 (13)	0.117 (12)
H16D[b]	0.862015	0.322429	0.697251	0.176*
H16E[b]	0.999693	0.342496	0.710643	0.176*
H16F[b]	0.951312	0.355333	0.647848	0.176*
C19A[b]	0.8446 (4)	0.6143 (3)	0.78224 (13)	0.1225 (12)
H19D[b]	0.904925	0.629022	0.810881	0.184*
H19E[b]	0.765183	0.622686	0.798641	0.184*
H19F[b]	0.854021	0.665108	0.751049	0.184*

[a]Occupancy: 0.924 (4). [b]Occupancy: 0.076 (4).

Comment

Hydrogen peroxide is known as the cleanest chemical product, which is widely used in chemical industry [5–6]. The title compound is an important derivative of anthraquinone, so the synthesis and crystal structure of it is of great significance to study the reduction of the consumption rate of working fluid per unit volume and improve the productivity of H_2O_2.

Single-crystal structure analysis revealed that the title compound crystallized in the orthorhombic space group *Pbca*. The ORTEP diagram is presented in the figure. Bond lengths and angles are all in the expected ranges. The bond lengths of C1=O1 and C8=O2 in the title molecule are 1.220 and 1.223 Å, respectively. They are similar to those reported in the literature [7, 8]. There is only one molecule in the asymmetric unit. Three six-membered rings are consisting of (C9, C10, C11, C12, C13 and C14; C1, C2, C7, C8, C9 and C14; C2, C3, C4, C5, C6 and C7), and the angle between two adjacent planes is 1.0° and 1.4°, respectively. The average distance from each plane atom to the plane is 0.0013, 0.0056, and 0.0069 Å, respectively. Otherwise, the shortest center distance between aromatic cycles of 2-(*tert*-pentyl)anthracene group is 3.600 Å, shorter than 3.800 Å, indicating π–π stacking interaction between the aromatic cycles [9].

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

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