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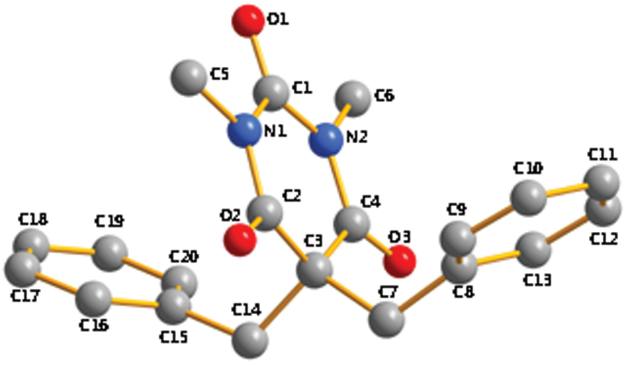
Crystal structure of 1,3-dimethyl-5,5-dibenzylbarbituric acid, $C_{20}H_{20}N_2O_3$



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Abstract

$C_{20}H_{20}N_2O_3$, orthorhombic, *Pbca* (no. 61), $a = 9.1300(18)$ Å, $b = 16.170(3)$ Å, $c = 24.000(5)$ Å, $V = 3543.2(12)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.1274$, $wR_{ref}(F^2) = 0.1434$, $T = 293$ K.

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A part of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was synthesized through two steps. Firstly, 1,3-dimethyl barbituric acid was easily synthesized according to the literature [3, 4]. Secondly, 1,3-methyl barbituric acid (1.56 g, 0.01 mol) was dissolved in approximately 70 mL of acetone, and we added benzyl chloride (1.86 g, 0.01 mol), anhydrous potassium carbonate as dehydrogenation agent and potassium iodide as catalyst. The mixture was heated

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Table 1: Data collection and handling.

Crystal:	Block, colourless
Size:	0.32 × 0.30 × 0.20 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
$2\theta_{max}$, completeness:	26.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	18209, 3512, 0.029
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2556
$N(param)_{refined}$:	226
Programs:	Bruker programs [1], SHELX [2]

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

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.7276(2)	−0.10645(12)	0.31703(8)	0.0533(5)
C2	0.60565(19)	−0.06394(10)	0.40278(7)	0.0446(4)
C3	0.69893(17)	0.01292(10)	0.40530(6)	0.0397(4)
C4	0.79089(17)	0.02945(10)	0.35407(7)	0.0421(4)
C5	0.5486(3)	−0.19739(14)	0.36065(11)	0.0867(8)
H5A	0.5755	−0.2296	0.3286	0.130*
H5B	0.4453	−0.1864	0.3597	0.130*
H5C	0.5722	−0.2275	0.3939	0.130*
C6	0.9049(3)	−0.02081(16)	0.26821(9)	0.0808(7)
H6A	0.9006	−0.0680	0.2440	0.121*
H6B	1.0023	−0.0153	0.2828	0.121*
H6C	0.8797	0.0281	0.2476	0.121*
C7	0.60354(19)	0.08915(10)	0.41894(7)	0.0461(4)
H7A	0.5519	0.0782	0.4535	0.055*
H7B	0.6684	0.1356	0.4256	0.055*
C8	0.49232(18)	0.11501(10)	0.37582(7)	0.0432(4)
C9	0.3487(2)	0.08880(12)	0.37873(8)	0.0561(5)
H9	0.3218	0.0501	0.4055	0.067*
C10	0.2444(2)	0.11927(15)	0.34250(10)	0.0716(6)
H10	0.1479	0.1013	0.3452	0.086*
C11	0.2826(3)	0.17595(15)	0.30245(9)	0.0712(6)
H11	0.2123	0.1967	0.2781	0.085*
C12	0.4245(2)	0.20149(14)	0.29865(9)	0.0707(6)
H12	0.4513	0.2393	0.2713	0.085*
C13	0.5283(2)	0.17171(12)	0.33499(8)	0.0574(5)
H13	0.6245	0.1901	0.3320	0.069*
C14	0.8065(2)	0.00189(11)	0.45543(7)	0.0486(4)
H14A	0.8643	0.0519	0.4590	0.058*
H14B	0.7491	−0.0038	0.4892	0.058*
C15	0.9092(2)	−0.07028(10)	0.45161(7)	0.0476(4)
C16	0.8816(3)	−0.14255(13)	0.47997(9)	0.0683(6)
H16	0.7971	−0.1474	0.5014	0.082*

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C17	0.9788(4)	−0.20807(14)	0.47689(12)	0.0923(9)
H17	0.9596	−0.2566	0.4963	0.111*
C18	1.1030(3)	−0.20166(17)	0.44535(14)	0.0967(10)
H18	1.1679	−0.2459	0.4433	0.116*
C19	1.1315(3)	−0.13068(18)	0.41706(12)	0.0871(8)
H19	1.2156	−0.1262	0.3954	0.105*
C20	1.0357(2)	−0.06570(14)	0.42059(10)	0.0640(5)
H20	1.0568	−0.0171	0.4015	0.077*
N1	0.62952(16)	−0.11876(9)	0.36013(6)	0.0510(4)
N2	0.80071(16)	−0.03192(9)	0.31453(6)	0.0490(4)
O1	0.7474(2)	−0.15887(10)	0.28190(7)	0.0880(6)
O2	0.51534(16)	−0.07913(9)	0.43836(6)	0.0674(4)
O3	0.85894(15)	0.09301(8)	0.34929(6)	0.0591(4)

with stirring to 353 K for 3 h. The product was produced in 50% yield by recrystallization from anhydrous ethanol.

Experimental details

The hydrogen atoms were placed on calculated positions using a riding model (AFIX 33, 43 and 23 options of the SHELX program [2]).

Comment

It is well known that for the treatment of nervous system, barbiturates and its derivatives are widely used in medicine. They may have anti-cancer, anesthesia, sedation, sleep and other medical effects [5–10] because the characteristic ring of barbituric acid and the base of DNA have certain similarity [11], which also gives a special significance of barbituric acid compounds. In the cyclic system of 1,3-dimethyl barbituric acid, the hydrogen atoms on the 5-position carbon are affected by the electron-withdrawing action of two adjacent carbonyl groups, and are easily lost to form a cyclic conjugate stable carbon anion, which acts as the nucleophilic reagent and the halogenated hydrocarbon carries out alkylation reaction.

All bond lengths and angles are within normal ranges. The typical structural characteristic of the title compound is the C3 atom which acts as a bridge between the 1,3-dimethyl barbituric acid ring and the two benzyl moieties. The dihedral angle between the benzyl rings and the 1,3-dimethyl barbituric acid are 50° and 27°, influenced by steric hindrance. The O2 atom and a H atom from a benzyl ring form a weak hydrogen bonding interaction.

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