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5,5-Dibenzylbarbituric acid monohydrate

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.054
wR factor = 0.143
Data-to-parameter ratio = 12.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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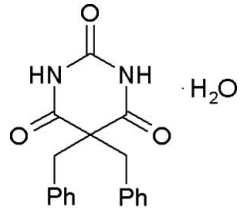
In the crystal structure of the title compound, $C_{18}H_{16}N_2O_3\cdot H_2O$, N—H···O and O—H···O hydrogen bonds assemble the molecules in a two-dimensional network.

Comment

Barbiturates are among the oldest known hypnotics. Barbital (5,5-diethylbarbituric acid) is the most common and most widely studied barbituric acid derivative, with several polymorphs studied in the solid state (Craven *et al.*, 1969; Craven *et al.*, 1971). As a part of our interest in barbiturates, we have investigated the crystal structure of 5,5-dibenzylbarbituric acid monohydrate, (I) (Fig. 1). The least-squares plane of the central heterocyclic ring makes an angle of 54.09 (11)° with the least-squares planes of the terminal phenyl ring C6–C11 and an angle of 62.71 (11)° with the other ring (C13–C18).



(I)

The molecules are linked *via* N–H···O hydrogen bonds, forming one-dimensional infinite chains along the *a* axis. These chains are linked together by the water molecules which act both as donors and acceptors of H atoms, assembling the molecules in an infinite two-dimensional network (Table 1 and Fig. 2).

Experimental

Absolute ethanol (30 ml) was added dropwise to clean sodium pieces (2.3 g, 100 mmol) placed in a 100 ml three-necked round-bottom flask equipped with a dropping funnel and water condenser. The resulting sodium ethoxide solution was allowed to cool with stirring. Diethyl malonate (7.1 ml, 50 mmol) was added dropwise to the sodium ethoxide solution over a period of 30 min. The mixture was heated on an oil bath at 363 K for 15 min and then allowed to cool. Dry benzyl chloride (11.3 ml, 100 mmol) was added dropwise to the reaction mixture over a period of 30 min. The reaction mixture was heated at 373 K in an oil bath for 3 h to complete the reaction. The ethanol was removed by distillation. The residue in the flask was diluted with water and extracted with three 25 ml portions of diethyl ether. The combined ether extracts were washed with water and dried with anhydrous magnesium sulfate. The diethyl ether was removed and the residue was distilled to yield benzyl diethyl malonate.

Absolute ethanol (20 ml) was added dropwise to clean sodium pieces (0.6 g, 26.2 mmol) placed in a 100 ml round-bottom flask fitted with a water condenser. When all the sodium had reacted, benzyl diethyl malonate (4 g, 11.8 mmol) and dry urea (0.8 g, 13.1 mmol) were added to the reaction mixture. The flask was heated in an oil bath at 383 K and the ethanol was slowly distilled off. The flask was kept at 383 K for 4 h and then allowed to cool. The solid in the flask was dissolved in water (15 ml). The solution was poured into a beaker and acidified with dilute sulfuric acid. The resulting white precipitate of (I) was filtered, dried and purified by recrystallization from ethanol to give the pure compound in 60% yield. A solution of (I) (100 mg) in ethanol (5 ml) was filtered and the filtrate was allowed to crystallize by slow evaporation over a period of two days. Block-like crystals were obtained from the solution and used for single-crystal X-ray diffraction studies.

Crystal data

$C_{18}H_{16}N_2O_3\cdot H_2O$
$M_r = 326.34$
Monoclinic, $P2_1/n$
$a = 6.6713\ (8)$ Å
$b = 15.3876\ (18)$ Å
$c = 16.0725\ (19)$ Å
$\beta = 101.162\ (2)^\circ$
$V = 1618.7\ (3)$ Å^3
$Z = 4$
$D_x = 1.339$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 100\ (2)$ K
Block, colourless
0.42 × 0.28 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
8317 measured reflections
2821 independent reflections
2374 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 25.0^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.143$
$S = 1.10$
2821 reflections
233 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.067P)^2 + 0.8695P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O1^{i}	0.98 (4)	2.18 (4)	3.132 (2)	163 (3)
O4—H3···O2^{ii}	0.85 (3)	1.94 (3)	2.779 (2)	167 (3)
N2—H2···O3^{iii}	0.88 (3)	1.85 (3)	2.726 (3)	173 (2)
N1—H1···O4^{iv}	0.89 (3)	1.90 (3)	2.779 (2)	166 (2)

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (iii) $x+1, y, z$; (iv) $-x, -y+1, -z+1$.

Atoms H1 and H2 on N1 and N2, respectively, and H3 and H4 on water atom O4 were located in difference Fourier maps and refined isotropically. All other H atoms were found in a difference map but then placed in calculated positions with C—H = 0.93 Å and U_{iso}(H) = $1.2U_{eq}$(C) for aromatic H atoms, and for methylene H atoms, C—-H = 0.97 Å and U_{iso}(H) = $1.2U_{eq}$(C).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

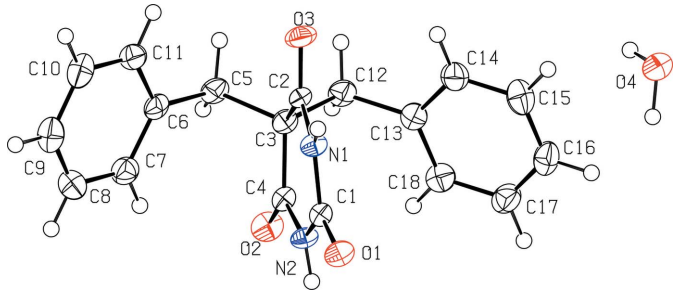


Figure 1
The molecular structure of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radius.

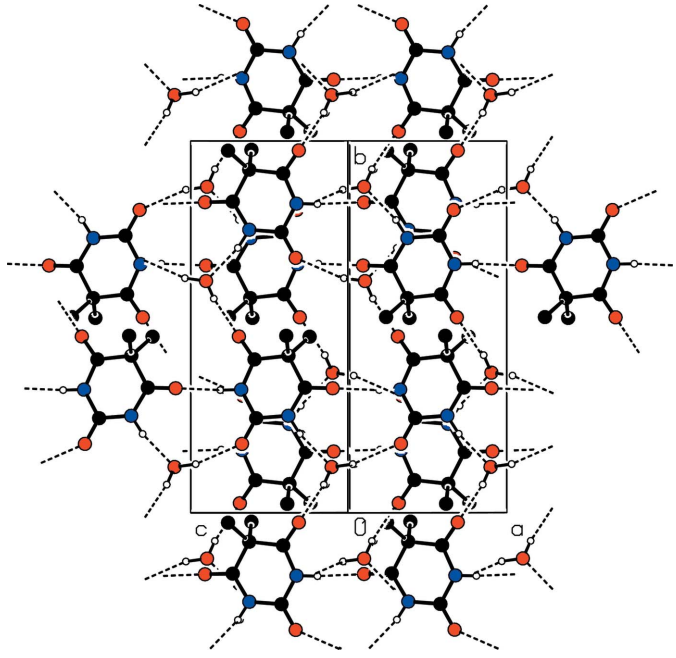


Figure 2
A packing diagram of the two-dimensional sheets. Dashed lines indicate hydrogen bonds. The phenyl rings and H atoms not involved in hydrogen bonding have been omitted for clarity.

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