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Synthesis and crystal structure of 3-octyl-5,5-diphenylimidazolidine-2,4-dione, $C_{23}H_{28}N_2O_2$

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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:	Colourless block
Size:	0.27 × 0.21 × 0.17 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.08 mm ⁻¹
Diffractometer, scan mode:	Bruker Smart APEX, ω
$\theta_{\max}$, completeness:	28.8°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	37198, 5321, 0.045
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3818
$N(\text{param})_{\text{refined}}$:	325
Programs:	Bruker [1, 2], SHELX [3, 4], Diamond [5]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.33531(12)	0.68022(8)	0.33935(5)	0.0385(3)
O2	0.33600(10)	0.48148(7)	0.47518(5)	0.0309(3)
N1	0.30784(11)	0.57315(8)	0.40191(5)	0.0259(3)
N2	0.48008(11)	0.58444(8)	0.45134(5)	0.0240(3)
H2	0.539(2)	0.5660(13)	0.4760(8)	0.045(6)*
C1	0.49855(13)	0.64548(9)	0.40708(6)	0.0235(3)
C2	0.37159(15)	0.63796(10)	0.37749(6)	0.0273(3)
C3	0.37333(13)	0.53981(9)	0.44630(6)	0.0241(3)
C4	0.18987(14)	0.53876(11)	0.38085(7)	0.0304(3)
H4A	0.1394(18)	0.5228(12)	0.4129(8)	0.037(5)*
H4B	0.1471(17)	0.5856(13)	0.3640(8)	0.038(5)*
C5	0.21126(16)	0.46656(11)	0.34090(7)	0.0331(4)
H5A	0.2454(17)	0.4159(12)	0.3607(7)	0.034(5)*
H5B	0.2702(19)	0.4854(12)	0.3140(8)	0.037(5)*
C6	0.09186(18)	0.44142(12)	0.31186(8)	0.0415(4)
H6A	0.025(2)	0.4211(16)	0.3411(10)	0.067(7)*
H6B	0.059(2)	0.4950(17)	0.2967(11)	0.071(7)*
C7 ^a	0.1069(2)	0.37575(12)	0.26736(8)	0.0477(5)
H7A ^a	0.140811	0.322192	0.282764	0.057*
H7B ^a	0.165522	0.396798	0.239156	0.057*
C8 ^a	−0.0235(3)	0.35947(17)	0.24178(11)	0.0425(7)
H8A ^a	−0.080554	0.340867	0.271257	0.051*
H8B ^a	−0.055531	0.414372	0.227633	0.051*

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Abstract

$C_{23}H_{28}N_2O_2$, orthorhombic, *Pbca* (no. 61), $a = 10.713(2)$ Å, $b = 15.689(3)$ Å, $c = 24.485(5)$ Å, $V = 4115.2(14)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0581$, $wR_{\text{ref}}(F^2) = 0.1734$, $T = 120(2)$ K.

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C9 ^a	−0.0302(2)	0.29496(18)	0.19642(10)	0.0421(6)
H9A ^a	−0.118996	0.282916	0.188335	0.050*
H9B ^a	0.008621	0.241203	0.209069	0.050*
C10 ^a	0.0340(3)	0.3232(2)	0.14413(11)	0.0476(6)
H10A ^a	0.002819	0.380240	0.133587	0.057*
H10B ^a	0.124932	0.327704	0.150632	0.057*
C11 ^a	0.0105(6)	0.2613(4)	0.09809(18)	0.0645(10)
H11A ^a	0.053012	0.281315	0.065056	0.097*
H11B ^a	0.042580	0.204985	0.108171	0.097*
H11C ^a	−0.079444	0.257482	0.091139	0.097*
C7A ^b	0.1069(2)	0.37575(12)	0.26736(8)	0.0477(5)
H7C ^b	0.105359	0.319574	0.285764	0.057*
H7D ^b	0.192592	0.383068	0.252941	0.057*
C8A ^b	0.0225(6)	0.3676(3)	0.21792(19)	0.0425(7)
H8C ^b	−0.062119	0.352740	0.231067	0.051*
H8D ^b	0.016812	0.424293	0.200408	0.051*
C9A ^b	0.0581(5)	0.3043(4)	0.1749(2)	0.0421(6)
H9C ^b	0.073624	0.248257	0.192118	0.050*
H9D ^b	0.136410	0.322955	0.157012	0.050*
C10A ^b	−0.0435(5)	0.2951(4)	0.1320(2)	0.0476(6)
H10C ^b	−0.117489	0.267700	0.148721	0.057*
H10D ^b	−0.068744	0.352372	0.119108	0.057*
C11A ^b	−0.0009(13)	0.2428(10)	0.0840(4)	0.0645(10)
H11D ^b	−0.068781	0.238320	0.057389	0.097*
H11E ^b	0.071382	0.270304	0.066943	0.097*
H11F ^b	0.022657	0.185667	0.096544	0.097*
C12	0.52079(13)	0.73514(9)	0.42966(6)	0.0240(3)
C13	0.42174(15)	0.78553(10)	0.44700(7)	0.0315(3)
H13	0.3405(18)	0.7641(12)	0.4427(8)	0.035(5)*
C14	0.44298(17)	0.86504(11)	0.46929(8)	0.0390(4)
H14	0.375(2)	0.9007(15)	0.4806(10)	0.059(6)*
C15	0.56401(18)	0.89538(11)	0.47466(8)	0.0424(4)
H15	0.5789(19)	0.9536(13)	0.4916(8)	0.044(5)*
C16	0.66371(17)	0.84577(12)	0.45778(9)	0.0429(4)
H16	0.751(2)	0.8679(14)	0.4602(9)	0.053(6)*
C17	0.64206(15)	0.76573(10)	0.43551(7)	0.0336(4)
H17	0.7118(19)	0.7340(13)	0.4227(8)	0.042(5)*
C18	0.60093(15)	0.61821(10)	0.36724(6)	0.0289(3)
C19	0.6164(2)	0.66353(14)	0.31905(7)	0.0444(4)
H19	0.565(2)	0.7091(15)	0.3121(10)	0.058(7)*
C20	0.7112(2)	0.64236(17)	0.28300(9)	0.0586(6)
H20	0.720(3)	0.6718(16)	0.2539(12)	0.072(8)*
C21	0.7906(2)	0.57658(16)	0.29486(10)	0.0590(6)
H21	0.857(2)	0.5639(16)	0.2693(12)	0.076(8)*
C22	0.7757(2)	0.53048(15)	0.34221(11)	0.0603(6)
H22	0.832(2)	0.4834(17)	0.3530(11)	0.071(8)*
C23	0.68026(18)	0.55122(11)	0.37867(9)	0.0421(4)
H23	0.671(2)	0.5173(14)	0.4128(10)	0.051(6)*

^aOccupancy: 0.684(2), ^bOccupancy: 0.316(2).

Source of material

To a solution of 5,5-diphenylimidazolidine-2,4-dione (1 g), one equivalent of octylbromide in absolute dimethylformamide (DMF) was added and the resulting solution heated under reflux for 4 h in the presence of 1.3 equivalents of K₂CO₃. The reaction mixture was filtered while hot and the solvent evaporated under reduced pressure. The residue

obtained was dried and crystallized from an ethanol solution to yield colourless block-shaped crystals of the title compound [6–9].

Experimental details

The outer half of the octyl chain (C8···C11) is disordered over 2 resolved sites in a 0.684(2):0.316(2) ratio (Table 2). The two components of the disorder were refined with restraints that their geometries be comparable and the attached hydrogen atoms were included as riding contributions in idealized positions with isotropic displacement parameters tied to those of the attached atoms.

Comment

Imidazolidines, especially hydantoin, are interesting structural cores which are frequently found in natural products and pharmaceuticals [10]. Such heterocyclic scaffolds are key portions of molecules possessing a broad spectrum of biological activities including acting as anti-inflammatory, anti-diabetic, anticarcinogenic, antimicrobial, antibacterial, and anti-androgen agents [10–15]. As part of our ongoing studies of hydantoin derivatives the title compound was prepared and its crystal structure is reported here [16–21]. For directly related structures, see: [6–9].

The C12···C17 and C18···C23 benzene rings are inclined, respectively, by 76.05(5) and 63.47(6)° to the central imidazolidinedione ring. The major component of the disordered *n*-octyl chain is not quite in the “fully extended” conformation (the Figure) but, nevertheless, is directed well away from the imidazolidinedione ring. In the crystal, inversion dimers are formed by paired N2–H2···O2 hydrogen bonds and these line up along the *b*-axis direction to form layers parallel to the *ab* plane with the *n*-octyl chains extending outward to either side. Stacking of these chains with those of neighboring layers of dimers forms the final 3-D structure having alternating hydrophilic and hydrophobic regions.

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