Crystal structure of *N*-benzyl-*N*-1,3,4-tetramethyl-2,5-dioxo-4-imidazolidinecarboxamide, $C_{15}H_{19}N_3O_3$

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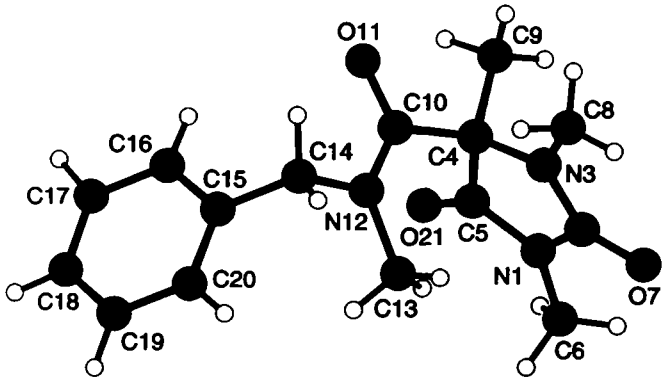

Abstract

$C_{15}H_{19}N_3O_3$, monoclinic, $P12_1/c1$ (no. 14), $a = 16.837(8)$ Å, $b = 7.368(4)$ Å, $c = 13.070(7)$ Å, $\beta = 111.005(4)°$, $V = 1513.7$ Å^3, $Z = 4$, $R_{gt}(F) = 0.050$, $wR_{ref}(F^2) = 0.120$, $T = 295$ K.

Source of material

Reaction of diethyl 2-acetylamino-2-methylmalonate with benzylurea and sodium ethoxide in ethanol at 120 °C over 4 h according to [1] and purification of the crude product by column chromatography yielded *N*-benzyl-4-methyl-2,5-dioxo-4-imidazolidinecarboxamide. Following alkylation with an excess of methyl iodide and sodium hydride in dry dimethylformamide for 4 h at 78 °C gave *N*-benzyl-*N*-1,3,4-tetramethyl-2,5-dioxo-4-imidazolidinecarboxamide as an oil. Crystals were obtained by dissolution in dimethyl sulfoxide and slow evaporation of the solvent over a period of several months.

Experimental details

All hydrogen positions were localized by Fourier difference calculations and individually refined with respect to positions and isotropic thermal displacement parameters.

Discussion

The structure contains one molecule per asymmetric unit and is held together by Van der Waals forces. All intramolecular bond lengths are normal. The shortest intermolecular distances are d(O7···H093–C9) = 2.576(1) Å and d(O11···H132–C13) = 2.760(1) Å. The main part of the molecule consists of an almost planar ring system (N1, C2, N3, C4, C5, C6, O7, C8, O21) with an r.m.s. value of the deviations from the least squares plane of 0.036 Å. A second plane, defined by the benzyl group (C14 – C20) with r.m.s. = 0.009 Å, is tilted by 66.8(1)° against the first mentioned plane. Both planes are connected by C10 and N12, which form together with (C4, C9, O11, C13, C14) an almost planar third plane with r.m.s. = 0.07 Å, tilted by 85.2(1)° against the first one and by 63.4(1)° against the second one.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.38 × 0.38 × 0.48 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	0.90 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC-6R, ω
$2\theta_{max}$:	50.1°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8997, 2681
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1356
$N(param)_{refined}$:	267
Programs:	SHELXS-97 [2], SHELXL-97 [3], DIAMOND [4]

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

Atom	Site	x	y	z	U_{iso}
H(061)	4*e*	0.825(3)	0.612(5)	0.262(4)	0.12(2)
H(062)	4*e*	0.927(3)	0.618(6)	0.332(4)	0.15(2)
H(063)	4*e*	0.858(3)	0.741(6)	0.350(4)	0.14(2)
H(081)	4*e*	0.955(3)	0.059(5)	0.550(3)	0.12(2)
H(082)	4*e*	0.866(3)	0.014(6)	0.564(3)	0.11(1)
H(083)	4*e*	0.936(3)	0.106(7)	0.652(5)	0.17(2)
H(091)	4*e*	0.827(2)	0.555(5)	0.687(3)	0.11(1)
H(092)	4*e*	0.921(2)	0.535(4)	0.675(2)	0.07(1)
H(093)	4*e*	0.889(2)	0.366(5)	0.725(3)	0.09(1)
H(131)	4*e*	0.656(2)	0.189(4)	0.301(3)	0.09(1)
H(132)	4*e*	0.734(2)	0.313(5)	0.344(3)	0.10(1)
H(133)	4*e*	0.749(3)	0.103(6)	0.355(4)	0.15(2)
H(141)	4*e*	0.646(2)	0.046(4)	0.549(3)	0.09(1)
H(142)	4*e*	0.643(2)	−0.045(4)	0.436(2)	0.08(1)
H(161)	4*e*	0.543(2)	0.284(4)	0.537(2)	0.059(9)
H(171)	4*e*	0.404(2)	0.385(4)	0.462(3)	0.09(1)
H(181)	4*e*	0.322(3)	0.290(5)	0.274(4)	0.13(2)
H(191)	4*e*	0.379(3)	0.105(6)	0.181(4)	0.15(2)
H(201)	4*e*	0.521(2)	0.001(5)	0.271(3)	0.09(1)

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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}
N(1)	4*e*	0.8645(1)	0.5100(3)	0.4175(2)	0.051(1)	0.054(2)	0.051(1)	−0.000(1)	0.019(1)	0.005(1)
N(3)	4*e*	0.8865(1)	0.2722(3)	0.5268(2)	0.049(1)	0.054(2)	0.049(1)	0.010(1)	0.019(1)	0.007(1)
N(12)	4*e*	0.7000(1)	0.1914(3)	0.4632(2)	0.051(1)	0.059(2)	0.041(1)	−0.009(1)	0.018(1)	−0.004(1)
O(7)	4*e*	0.9606(1)	0.2910(3)	0.4094(2)	0.060(1)	0.089(2)	0.077(1)	0.005(1)	0.038(1)	−0.013(1)
O(11)	4*e*	0.7318(2)	0.2728(4)	0.6382(2)	0.092(2)	0.166(3)	0.053(1)	−0.048(2)	0.044(1)	−0.026(2)
O(21)	4*e*	0.7658(1)	0.6677(3)	0.4647(2)	0.070(2)	0.062(2)	0.093(2)	0.022(1)	0.021(1)	0.000(1)
C(2)	4*e*	0.9098(2)	0.3487(4)	0.4485(2)	0.044(2)	0.062(2)	0.043(2)	−0.002(2)	0.013(1)	−0.006(2)
C(4)	4*e*	0.8277(2)	0.3860(4)	0.5571(2)	0.043(2)	0.056(2)	0.037(2)	0.003(1)	0.013(1)	−0.007(1)
C(5)	4*e*	0.8120(2)	0.5389(4)	0.4743(2)	0.046(2)	0.048(2)	0.052(2)	0.005(2)	0.010(1)	−0.005(2)
C(6)	4*e*	0.8718(3)	0.6289(7)	0.3325(4)	0.081(3)	0.097(3)	0.072(3)	−0.001(3)	0.026(2)	0.033(2)
C(8)	4*e*	0.9160(3)	0.0970(6)	0.5760(4)	0.074(3)	0 069(3)	0.099(3)	0.018(2)	0.037(3)	0.025(2)
C(9)	4*e*	0.8705(2)	0.4709(6)	0.6704(3)	0.062(2)	0.092(3)	0.047(2)	−0.013(2)	0.011(2)	−0.019(2)
C(10)	4*e*	0.7473(2)	0.2784(4)	0.5542(2)	0.051(2)	0.075(2)	0.041(2)	−0.001(2)	0.021(1)	−0.002(2)
C(13)	4*e*	0.7102(2)	0.1991(6)	0.3570(3)	0.064(2)	0.086(3)	0.041(2)	−0.019(2)	0.016(2)	−0.014(2)
C(14)	4*e*	0.6330(2)	0.0651(5)	0.4683(3)	0.060(2)	0.057(2)	0.064(2)	−0.009(2)	0.022(2)	0.005(2)
C(15)	4*e*	0.5444(2)	0.1358(4)	0.4132(2)	0.053(2)	0.044(2)	0.065(2)	−0.013(1)	0.025(2)	0.001(2)
C(16)	4*e*	0.5083(3)	0.2508(5)	0.4673(4)	0.073(3)	0.063(2)	0.092(3)	−0.020(2)	0.042(2)	−0.012(2)
C(17)	4*e*	0.4252(3)	0.3121(5)	0.4160(5)	0.083(3)	0.064(3)	0.165(5)	−0.002(2)	0.079(4)	0.005(3)
C(18)	4*e*	0.3788(3)	0.2561(7)	0.3116(6)	0.054(3)	0.076(3)	0.166(5)	−0.008(2)	0.026(3)	0.042(3)
C(19)	4*e*	0.4136(3)	0.1423(6)	0.2578(4)	0.067(3)	0.083(3)	0.111(4)	−0.014(2)	0.013(3)	0.020(3)
C(20)	4*e*	0.4956(2)	0.0813(5)	0.3079(3)	0.064(2)	0.066(2)	0.075(3)	−0.009(2)	0.020(2)	−0.002(2)

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