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Crystal structure of dimethyl 2-(*tert*-butylamino)-5-benzoyl-6-phenyl-4*H*-pyran-3,4-dicarboxylate, $C_{26}H_{27}NO_6$

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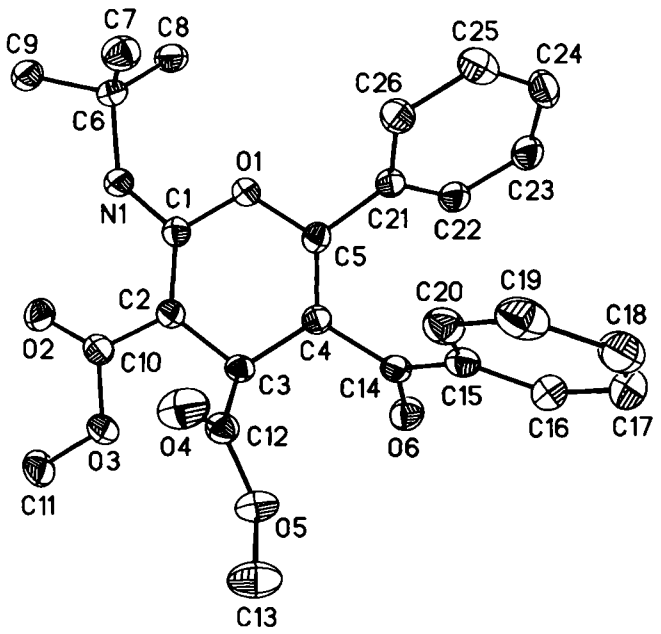

Abstract

$C_{26}H_{27}NO_6$, monoclinic, $C12/c1$ (no. 15), a = 15.0094(9) Å, b = 15.811(1) Å, c = 20.031(1) Å, β = 102.30(1)°, V = 4644.6 Å^3, Z = 8, $R_{gt}(F)$ = 0.050, $wR_{ref}(F^2)$ = 0.142, T = 120 K.

Source of material

The title compound was synthesized by the reaction of *tert*-butyl isocyanide and dimethyl acetylene dicarboxylate with 1,3-diphenylpropane-1,3-dione in the presence of triethylamine as a base in hot 1,4-xylene, then purified by column chromatography on silica gel using a mixture of ethyl acetate and hexane (30:70, *v/v*) as eluent [1]. Recrystallization from methanol gave crystals suitable for X-ray structure analysis.

Discussion

Some of 2-amino-4*H*-pyran derivatives have fairly low stability. They convert to furan and pyridine derivatives by ring transformation process in the presence of little amounts of protons or hydroxide ions [2]. Some other 2-amino-4*H*-pyrans which contain electron withdrawing groups or fused heterocyclic rings, are stable and exhibit biological activity as anticancer, antihypertensive and coronary dilating agents [3]. They have also served as convenient starting materials for synthesis of condensed heterocycles. Recently we have reported the crystal structures of some new 2-amino-4*H*-pyran derivatives [4-6]. The crystal structure determination of the title compound was performed in view of its good stability.

The molecule is stabilized by one intramolecular N–H···O type of hydrogen bonding. The details of the N1–H1N···O2 bond are d(N1–H1N) = 0.93 Å, d(H1N···O2) = 1.91 Å, d(N1···O2) = 2.693 Å, ∠N1–H1N···O2 = 141°. In addition, the molecules are interlinked by van der Waals forces.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.25 × 0.25 × 0.4 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.91 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ/ω
$2\theta_{max}$:	56°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	21404, 5582
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3711
$N(param)_{refined}$:	301
Programs:	SHELXTL [7]

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

Atom	Site	x	y	z	U_{iso}
H(1N)	8*f*	0.3594	−0.0472	0.5567	0.041
H(3A)	8*f*	0.5424	0.1563	0.5152	0.027
H(7A)	8*f*	0.1600	0.0120	0.5100	0.049
H(7B)	8*f*	0.1087	−0.0457	0.4504	0.049
H(7C)	8*f*	0.1714	0.0279	0.4350	0.049
H(8A)	8*f*	0.3055	−0.1521	0.4194	0.050
H(8B)	8*f*	0.2610	−0.0717	0.3800	0.050
H(8C)	8*f*	0.1995	−0.1472	0.3936	0.050
H(9A)	8*f*	0.2266	−0.1164	0.5723	0.048
H(9B)	8*f*	0.2845	−0.1795	0.5386	0.048
H(9C)	8*f*	0.1786	−0.1748	0.5121	0.048
H(11A)	8*f*	0.6406	0.1179	0.7196	0.064
H(11B)	8*f*	0.6250	0.0234	0.6965	0.064
H(11C)	8*f*	0.5544	0.0692	0.7316	0.064
H(13A)	8*f*	0.5696	0.4057	0.6004	0.062
H(13B)	8*f*	0.5046	0.3605	0.6408	0.062
H(13C)	8*f*	0.4647	0.4006	0.5691	0.062
H(16A)	8*f*	0.5018	0.3721	0.3209	0.043
H(17A)	8*f*	0.4122	0.4831	0.2697	0.052
H(18A)	8*f*	0.2709	0.5070	0.2945	0.054
H(19A)	8*f*	0.2175	0.4194	0.3693	0.048
H(20A)	8*f*	0.3049	0.3060	0.4193	0.037
H(22A)	8*f*	0.4257	0.1493	0.2895	0.034
H(23A)	8*f*	0.3512	0.1913	0.1814	0.040
H(24A)	8*f*	0.1974	0.2206	0.1596	0.041
H(25A)	8*f*	0.1151	0.2007	0.2436	0.040
H(26A)	8*f*	0.1882	0.1567	0.3516	0.034

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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}
O(1)	8*f*	0.32726(7)	0.06690(7)	0.43090(5)	0.0281(6)	0.0285(6)	0.0220(6)	–0.0058(5)	0.0012(5)	0.0029(5)
O(2)	8*f*	0.45723(8)	–0.01335(8)	0.62951(6)	0.0330(7)	0.0410(7)	0.0317(7)	–0.0065(5)	0.0005(5)	0.0119(6)
O(3)	8*f*	0.54524(8)	0.10362(7)	0.63548(6)	0.0281(6)	0.0393(7)	0.0231(6)	–0.0043(5)	–0.0014(5)	0.0010(5)
O(4)	8*f*	0.39074(8)	0.25076(8)	0.57415(7)	0.0334(7)	0.0452(8)	0.0564(9)	–0.0054(6)	0.0214(6)	–0.0175(6)
O(5)	8*f*	0.52948(7)	0.29237(7)	0.56377(6)	0.0263(6)	0.0266(6)	0.0431(7)	–0.0027(5)	0.0063(5)	–0.0090(5)
O(6)	8*f*	0.55335(8)	0.25639(7)	0.40844(6)	0.0244(6)	0.0385(7)	0.0386(7)	–0.0037(5)	0.0072(5)	0.0045(5)
N(1)	8*f*	0.33064(9)	–0.03116(8)	0.51292(7)	0.0259(7)	0.0262(7)	0.0259(7)	–0.0038(5)	0.0019(6)	0.0034(5)
C(1)	8*f*	0.3674(1)	0.0403(1)	0.49512(8)	0.0222(8)	0.0244(8)	0.0233(8)	0.0013(6)	0.0056(6)	0.0013(6)
C(2)	8*f*	0.4391(1)	0.0826(1)	0.53514(8)	0.0233(8)	0.0253(8)	0.0227(8)	–0.0003(6)	0.0040(6)	0.0003(6)
C(3)	8*f*	0.4766(1)	0.1627(1)	0.51150(8)	0.0186(7)	0.0262(8)	0.0224(8)	–0.0003(6)	0.0016(6)	–0.0001(6)
C(4)	8*f*	0.4317(1)	0.18257(9)	0.43837(8)	0.0210(8)	0.0246(8)	0.0232(8)	–0.0001(6)	0.0048(6)	0.0004(6)
C(5)	8*f*	0.3633(1)	0.1363(1)	0.40340(8)	0.0249(8)	0.0235(8)	0.0237(8)	–0.0007(6)	0.0079(6)	0.0006(6)
C(6)	8*f*	0.2449(1)	–0.0749(1)	0.47922(8)	0.0212(8)	0.0260(8)	0.0263(8)	–0.0038(6)	0.0040(6)	0.0001(6)
C(7)	8*f*	0.1637(1)	–0.0146(1)	0.46758(9)	0.0279(9)	0.0321(9)	0.037(1)	0.0011(7)	0.0047(7)	0.0018(8)
C(8)	8*f*	0.2535(1)	–0.1152(1)	0.41180(9)	0.0292(9)	0.040(1)	0.0318(9)	–0.0078(7)	0.0063(7)	–0.0071(8)
C(9)	8*f*	0.2325(1)	–0.1427(1)	0.53028(9)	0.0297(9)	0.0307(9)	0.0341(9)	–0.0049(7)	0.0055(7)	0.0046(7)
C(10)	8*f*	0.4783(1)	0.0515(1)	0.60244(8)	0.0239(8)	0.0331(9)	0.0248(8)	0.0011(7)	0.0053(7)	–0.0008(7)
C(11)	8*f*	0.5955(1)	0.0763(1)	0.70126(9)	0.031(1)	0.066(1)	0.027(1)	–0.0075(9)	–0.0035(8)	0.0093(9)
C(12)	8*f*	0.4592(1)	0.2385(1)	0.55442(8)	0.0231(8)	0.0272(8)	0.0251(8)	–0.0007(6)	0.0015(6)	–0.0001(6)
C(13)	8*f*	0.5160(1)	0.3714(1)	0.5962(1)	0.038(1)	0.0291(9)	0.055(1)	0.0021(8)	0.0047(9)	–0.0142(9)
C(14)	8*f*	0.4714(1)	0.2554(1)	0.40606(8)	0.0247(8)	0.0278(8)	0.0209(8)	–0.0044(6)	0.0035(6)	–0.0032(6)
C(15)	8*f*	0.4118(1)	0.3266(1)	0.37472(8)	0.0312(9)	0.0245(8)	0.0249(8)	–0.0041(6)	–0.0001(7)	–0.0038(6)
C(16)	8*f*	0.4444(1)	0.3809(1)	0.33019(9)	0.041(1)	0.0310(9)	0.034(1)	–0.0072(8)	0.0026(8)	0.0024(7)
C(17)	8*f*	0.3910(1)	0.4477(1)	0.3000(1)	0.056(1)	0.030(1)	0.039(1)	–0.0084(9)	–0.0022(9)	0.0061(8)
C(18)	8*f*	0.3063(1)	0.4619(1)	0.3147(1)	0.054(1)	0.0240(9)	0.046(1)	0.0030(8)	–0.016(1)	–0.0016(8)
C(19)	8*f*	0.2743(1)	0.4094(1)	0.3593(1)	0.039(1)	0.032(1)	0.043(1)	0.0076(8)	–0.0038(9)	–0.0115(8)
C(20)	8*f*	0.3268(1)	0.3414(1)	0.38936(9)	0.0348(9)	0.0283(9)	0.0290(9)	–0.0002(7)	0.0034(7)	–0.0054(7)
C(21)	8*f*	0.3151(1)	0.15192(9)	0.33215(8)	0.0272(8)	0.0211(7)	0.0225(8)	–0.0032(6)	0.0038(6)	–0.0012(6)
C(22)	8*f*	0.3635(1)	0.1605(1)	0.28052(8)	0.0312(9)	0.0286(8)	0.0262(9)	–0.0052(7)	0.0066(7)	–0.0022(7)
C(23)	8*f*	0.3190(1)	0.1855(1)	0.21594(9)	0.041(1)	0.0352(9)	0.0253(9)	–0.0092(8)	0.0096(8)	–0.0002(7)
C(24)	8*f*	0.2268(1)	0.2020(1)	0.20273(9)	0.047(1)	0.0275(9)	0.0230(8)	–0.0015(8)	–0.0018(8)	0.0027(7)
C(25)	8*f*	0.1776(1)	0.1909(1)	0.25317(9)	0.0323(9)	0.0318(9)	0.0330(9)	0.0029(7)	–0.0004(7)	–0.0039(7)
C(26)	8*f*	0.2214(1)	0.1652(1)	0.31787(8)	0.0304(9)	0.0289(8)	0.0248(8)	–0.0008(7)	0.0058(7)	–0.0026(7)

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