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Crystal structure of methyl 2-(6,11-dioxo-2,3,6,11-tetrahydro-1*H*-benzo[*f*]pyrrolo[2,1-*a*]isoindole-5-carbonyl)benzoate, C₂₄H₁₇NO₅

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:	Orange block
Size:	0.26 × 0.22 × 0.17 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.10 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.0°, 98%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	4729, 3276, 0.025
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1968
$N(\text{param})_{\text{refined}}$:	272
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

<https://doi.org/10.1515/ncrs-2020-0278>

Received June 9, 2020; accepted June 24, 2020; available online July 3, 2020

Abstract

C₂₄H₁₇NO₅, triclinic, $P\bar{1}$ (no. 2), $a = 8.5858(2)$ Å, $b = 9.2397(4)$ Å, $c = 13.7831(8)$ Å, $\alpha = 97.11(3)^\circ$, $\beta = 103.23(3)^\circ$, $\gamma = 113.33(3)^\circ$, $V = 948.8(2)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0545$, $wR_{\text{ref}}(F^2) = 0.1669$, $T = 296(2)$ K.

CCDC no.: 2012794

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.6272(3)	0.6579(2)	0.86948(14)	0.0776(7)
O2	0.9171(3)	1.28216(19)	1.08863(13)	0.0677(6)
O3	0.9255(3)	0.8626(2)	0.64640(17)	0.0910(8)
O4	0.5448(3)	0.8779(3)	0.61771(16)	0.0891(7)
O5	0.2699(3)	0.6798(4)	0.57571(19)	0.1241(10)
N1	0.9611(3)	1.1060(2)	0.79999(15)	0.0490(5)
C1	0.6391(3)	0.8460(3)	1.00742(17)	0.0449(6)
C2	0.5175(3)	0.7240(3)	1.03674(19)	0.0547(7)
H2	0.468475	0.617460	0.998578	0.066*
C3	0.4672(4)	0.7572(3)	1.1216(2)	0.0625(7)
H3	0.385410	0.673327	1.140561	0.075*
C4	0.5385(4)	0.9153(3)	1.1787(2)	0.0613(7)
H4	0.502999	0.938538	1.235258	0.074*
C5	0.6622(4)	1.0379(3)	1.15125(19)	0.0545(7)
H5	0.712506	1.143584	1.190906	0.065*
C6	0.7134(3)	1.0069(3)	1.06544(17)	0.0448(6)
C7	0.8429(3)	1.1426(3)	1.03530(18)	0.0489(6)
C8	0.8713(3)	1.0982(3)	0.93797(18)	0.0454(6)
C9	0.7959(3)	0.9371(3)	0.87682(17)	0.0441(6)
C10	0.6839(3)	0.8013(3)	0.91217(18)	0.0492(6)
C11	0.8536(3)	0.9435(3)	0.79034(18)	0.0458(6)
C12	0.9724(3)	1.1990(3)	0.88761(19)	0.0498(6)
C13	1.0858(4)	1.3733(3)	0.8977(2)	0.0692(8)

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H13A	1.192215	1.415036	0.956237	0.083*
H13B	1.021135	1.437300	0.905470	0.083*
C14	1.1326(4)	1.3758(3)	0.7973(2)	0.0776(9)
H14A	1.260456	1.434166	0.811340	0.093*
H14B	1.075983	1.429673	0.756142	0.093*
C15	1.0659(4)	1.1990(3)	0.7394(2)	0.0664(8)
H15A	0.992588	1.177981	0.669574	0.080*
H15B	1.164620	1.173201	0.738012	0.080*
C16	0.8274(4)	0.8245(3)	0.6993(2)	0.0572(7)
C17	0.6854(4)	0.6530(3)	0.67201(19)	0.0547(7)
C18	0.7432(5)	0.5330(4)	0.6841(2)	0.0717(8)
H18	0.863773	0.562387	0.712611	0.086*
C19	0.6240(7)	0.3728(4)	0.6542(3)	0.0949(12)
H19	0.663755	0.293621	0.662074	0.114*
C20	0.4458(7)	0.3284(5)	0.6126(3)	0.1085(16)
H20	0.364497	0.219369	0.593698	0.130*
C21	0.3874(5)	0.4465(5)	0.5989(2)	0.0961(13)
H21	0.266723	0.415697	0.569507	0.115*
C22	0.5058(4)	0.6098(4)	0.62825(19)	0.0628(8)
C23	0.4476(4)	0.7394(5)	0.6088(2)	0.0738(9)
C24	0.1989(6)	0.7923(7)	0.5445(4)	0.166(2)
H24A	0.242607	0.832933	0.490478	0.250*
H24B	0.236265	0.881184	0.602127	0.250*
H24C	0.071198	0.736764	0.520386	0.250*

Source of material

To a solution of naphthalene-1,4-dione (1.58 g, 10.0 mmol) in methanol (20 mL) was added ninhydrin hydrate (1.78 g, 10.0 mmol) and *L*-proline (1.15 g, 10.0 mmol) at room temperature. The mixture was stirred at 60 degree for 2 h. After completion the mixture was concentrated to remove all solvent, then extracted by dichloromethane, washed with saturated sodium bicarbonate solution and brine. The organic phase was dried by anhydrous sodium sulfate, concentrated and purified by column chromatography over silica gel eluting with a gradient of ethyl acetate/hexane (v/v, 0–30%) to give the yellow solid. Yield, 2.16 g, 54%. The title compound was dissolved in ethyl acetate/hexane (v/v, 1/3). After four days, orange crystals were obtained.

Experimental details

The structure was determined by the intrinsic phasing routines in the SHELXT program [2] and refined by SHELXL [3] by using Olex2 [4]. All of the hydrogen atoms were placed in the calculated positions.

Comment

Fused ring compounds play an important and basic role in chemistry, medicine and materials science [5, 6]. Therefore, the synthesis of fused ring compounds with high efficiency has always been the concern of synthetic chemists. However, it is difficult to synthesize fused ring compounds with complicated structures through simple methods [7]. For example, the

syntheses of compounds with 2,3-dihydro-1*H*-benzo[*f*]pyrrolo [2,1-*a*] isoindole-6,11-dione skeleton are rarely reported [8]. Moreover, current synthetic methods and reaction conditions of such compounds are relatively intricate, and most of them need multiple steps [9, 10]. Herein, we report a simple method for the synthesis of 2,3-dihydro-1*H*-benzo[*f*]pyrrolo [2,1-*a*]isoindole-6,11-dione derivative. Specifically, the title compound was synthesized using easily available commercial materials (naphthalene-1,4-dione, ninhydrin hydrate and *L*-proline) in methanol in one-pot under mild reaction condition, and then purified by column chromatography and crystallized from ethyl acetate-hexane solvents. There is one molecule in the asymmetric unit (see the Figure).

The bond lengths and angles are in the normal ranges [11]. The title compound was synthesized in one step by easily available commercial materials under mild reaction condition, which opens up a new method for the synthesis of such complicated ring compounds.

Acknowledgements: This work was supported by the National Natural Science Foundation of China (Grant No. 21901028), Natural Science Foundation of Chongqing (Grant Nos. cstc2019jcyj-msxmX0202 and cstc2019jcyj-msxmX0012), Science and Technology Research Program of Chongqing Municipal Education Commission (Grant Nos. KJQN201801305 and KJQN201801321) and Chongqing University of Arts and Sciences (Grant Nos. 2017RBX08 and R2015CH12).

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