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Crystal structure of 1,3,5,7-tetraphenyl-8-(*N*-phenylformamido)-2-oxa-5-azabicyclo[4.2.0]oct-3-en-7-yl benzoate, C₄₄H₃₄N₂O₄

The title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.25 × 0.23 × 0.20 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.08 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	25°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8883, 6042, 0.020
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4316
$N(\text{param})_{\text{refined}}$:	452
Programs:	Bruker programs [1], SHELX [2, 3]

<https://doi.org/10.1515/ncrs-2019-0799>

Received October 28, 2019; accepted December 28, 2019; available online January 21, 2020

Abstract

C₄₄H₃₄N₂O₄, triclinic, $P\bar{1}$ (no. 2), $a = 10.4564(13)$ Å, $b = 11.3627(14)$ Å, $c = 15.3946(19)$ Å, $\alpha = 82.106(2)^\circ$, $\beta = 75.318(2)^\circ$, $\gamma = 77.704(2)^\circ$, $V = 1722.0(4)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0420$, $wR_{\text{ref}}(F^2) = 0.1086$, $T = 296(2)$ K.

CCDC no.: 1956451

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Source of material

A mixture of 1.55 g (4.7 mmol) of diphenacylaniline and 0.87 mL (9.4 mmol) of POCl₃ in 30 mL of pyridine (dried over CaH₂) was heated with occasional swirling at 373 K for 45 min. The deep red solution was poured onto 50 mL of crushed ice and the resulting solid was filtered. Two recrystallizations from isopropyl alcohol gave 0.81 g (55%) of red needles (2,4,6-triphenyl-1,4-oxazine) [4]. The oxazine was placed in a quartz tube with an equal proportion of methanol and tetrahydrofuran (30 mL), and irradiated by a blue LED for light reaction for 5 hours. The product was obtained by column chromatography, and the developing solvent was petroleum ether and ethyl acetate 30:1. Finally, the resulting solution was evaporated to dryness in vacuo to give the title compound as a colorless crystal.

Experimental details

All hydrogen atoms were placed in the calculated positions and all the non-hydrogen atoms were refined anisotropically.

Discussion

Photochemical reaction is a widely used method in organic synthesis. Many photosensitive compounds such as

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
O1	0.14510(14)	1.08305(11)	0.15685(9)	0.0523(4)
O2	0.22295(11)	0.68436(10)	0.26358(7)	0.0337(3)
O3	0.57113(15)	0.76609(15)	0.39095(11)	0.0676(5)
N1	0.02029(13)	0.89295(12)	0.30322(10)	0.0347(3)
N2	0.48491(13)	0.74951(13)	0.27164(10)	0.0361(4)
C1	−0.09376(17)	0.97957(16)	0.34078(12)	0.0375(4)
C2	−0.0796(2)	1.09594(18)	0.34658(15)	0.0527(5)
H2	0.004499	1.117788	0.326743	0.063*
C3	−0.1910(2)	1.1802(2)	0.38200(17)	0.0669(7)
H3	−0.181068	1.258571	0.385965	0.080*
C4	−0.3156(2)	1.1493(3)	0.41128(17)	0.0737(8)
H4	−0.389863	1.206292	0.435316	0.088*
C5	−0.3301(2)	1.0343(3)	0.40492(16)	0.0699(7)
H5	−0.414874	1.013635	0.423444	0.084*
C6	−0.21958(18)	0.9484(2)	0.37116(14)	0.0511(5)
H6	−0.229817	0.869693	0.368853	0.061*
C7	0.00295(17)	0.79503(15)	0.26562(11)	0.0337(4)
H7	−0.080419	0.798096	0.253410	0.040*
C8	0.09685(16)	0.69670(15)	0.24562(11)	0.0317(4)
C9	0.22425(16)	0.74766(14)	0.33813(11)	0.0309(4)
C10	0.14236(15)	0.87938(14)	0.33365(11)	0.0311(4)
H10	0.121074	0.910066	0.393116	0.037*
C11	0.07760(17)	0.59383(15)	0.20555(11)	0.0347(4)
C12	−0.0167(2)	0.60757(19)	0.15341(14)	0.0531(5)
H12	−0.061947	0.684521	0.138496	0.064*
C13	−0.0436(2)	0.5083(2)	0.12364(16)	0.0668(7)
H13	−0.107962	0.518885	0.089778	0.080*
C14	0.0239(2)	0.3938(2)	0.14367(16)	0.0633(6)
H14	0.004791	0.326894	0.124239	0.076*
C15	0.1196(2)	0.37971(18)	0.19252(14)	0.0514(5)
H15	0.166651	0.302715	0.205304	0.062*
C16	0.14716(18)	0.47753(16)	0.22298(12)	0.0409(4)
H16	0.213124	0.465952	0.255642	0.049*
C17	0.19536(18)	0.66654(15)	0.42331(12)	0.0363(4)
C18	0.0679(2)	0.6761(2)	0.47902(13)	0.0527(5)
H18	−0.001432	0.735998	0.464831	0.063*
C19	0.0423(3)	0.5979(2)	0.55531(15)	0.0710(7)
H19	−0.043758	0.605984	0.592559	0.085*
C20	0.1418(3)	0.5092(2)	0.57654(16)	0.0725(7)
H20	0.123990	0.456473	0.628086	0.087*
C21	0.2689(3)	0.4974(2)	0.52167(16)	0.0732(7)
H21	0.337200	0.436316	0.535800	0.088*
C22	0.2956(2)	0.57640(18)	0.44533(14)	0.0569(6)
H22	0.382005	0.568438	0.408617	0.068*
C23	0.52364(16)	0.70527(16)	0.18346(12)	0.0394(4)
C24	0.5078(2)	0.59169(19)	0.17261(15)	0.0542(6)
H24	0.464234	0.545404	0.221192	0.065*
C25	0.5568(2)	0.5464(2)	0.08941(18)	0.0714(7)
H25	0.544939	0.469909	0.081960	0.086*
C26	0.6222(2)	0.6127(3)	0.01820(17)	0.0778(8)
H26	0.656047	0.581068	−0.037448	0.093*
C27	0.6379(2)	0.7256(3)	0.02858(16)	0.0745(7)
H27	0.682287	0.771074	−0.020228	0.089*
C28	0.5885(2)	0.7730(2)	0.11104(14)	0.0560(6)
H28	0.598981	0.850269	0.117708	0.067*
O4	0.28409(11)	0.90589(10)	0.18084(7)	0.0347(3)
C29	0.27291(16)	0.92363(15)	0.27345(11)	0.0318(4)
C30	0.58476(19)	0.73755(19)	0.31617(16)	0.0507(5)
H30	0.671261	0.704341	0.286125	0.061*
C31	0.20647(17)	0.98676(16)	0.13248(12)	0.0371(4)
C32	0.20940(17)	0.94044(17)	0.04661(12)	0.0395(4)

Table 2 (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C33	0.2455(2)	0.81899(19)	0.03327(14)	0.0527(5)
H33	0.277083	0.763641	0.076944	0.063*
C34	0.2344(3)	0.7798(2)	−0.04537(16)	0.0720(7)
H34	0.255236	0.697697	−0.053288	0.086*
C35	0.1929(3)	0.8612(3)	−0.11157(16)	0.0767(8)
H35	0.187892	0.834554	−0.164867	0.092*
C36	0.1592(3)	0.9808(3)	−0.09926(16)	0.0782(8)
H36	0.131908	1.035990	−0.144529	0.094*
C37	0.1651(2)	1.0213(2)	−0.01999(14)	0.0599(6)
H37	0.139197	1.103210	−0.011419	0.072*
C38	0.31286(17)	1.03919(15)	0.28575(13)	0.0367(4)
C39	0.2720(2)	1.08800(18)	0.36819(14)	0.0495(5)
H39	0.214272	1.051849	0.415563	0.059*
C40	0.3155(2)	1.1897(2)	0.38131(18)	0.0623(6)
H40	0.286343	1.221398	0.437068	0.075*
C41	0.4006(2)	1.2435(2)	0.3132(2)	0.0677(7)
H41	0.428691	1.312457	0.321831	0.081*
C42	0.4446(2)	1.1952(2)	0.23170(19)	0.0685(7)
H42	0.504073	1.230951	0.185321	0.082*
C43	0.4016(2)	1.09361(18)	0.21746(15)	0.0519(5)
H43	0.432516	1.061837	0.161762	0.062*
C44	0.35038(15)	0.80638(14)	0.31557(11)	0.0316(4)
H44	0.358459	0.827205	0.373407	0.038*

*Occupancy: 0.854(7).

1,4-dihydropyridines [5], 1,4-dihydropyrazines [6] and 1,4-oxazines are used as raw materials for [2 + 2] Photocyclization. The 1,4-oxazine is among the simplest heterocyclic compounds to be so far unknown. A fair number of substituted and ring-fused 1,4-oxazines have been prepared [7], which have abundant pharmacological activities and used as central nervous system agonists [8] and selective serotonin reuptake inhibitors [9].

In the molecules forming the title crystal structure, the rings of oxazine and four membered ring are not planar due to the steric hindrance of phenyl substituents (see the figure). In the four membered ring, the C9–C10, C10–C29, C29–C44 and C44–C9 bond lengths are of 1.560(2), 1.581(2), 1.556(2) and 1.544(2) Å, respectively. Several important bond angle data are involved as follows C7–N1–C10 = 116.67(13)°, C8–O2–C9 = 114.37(12)°, C30–N2–C44 = 117.94(16)°. The bond lengths and angles are in the expected ranges.

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