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Crystal structure of phenyl(1,3,4*a*-triphenyl-4*a*,5,6,10*b*-tetrahydro-1*H*-[1,4]oxazino[2,3-*c*]quinolin-5-yl)methanone, C₃₆H₂₈N₂O₂

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.23 × 0.20 × 0.18 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.08 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	9573, 4777, 0.028
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3228
$N(\text{param})_{\text{refined}}$:	361
Programs:	Bruker [1], SHELX [2, 3]

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.

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 0.50 g (1.61 mmol) of 2,4,6-triphenyl-1,4-oxazine was placed in a quartz tube with acetone (20 mL), and irradiated by a blue LED (410 nm) performing a photochemical reaction for 8 hours. The product was separated by column chromatography, and the elution solvent was petroleum ether and ethyl acetate 20:1. Finally, the obtained solution was evaporated to dryness in vacuo and recrystallized from methanol and dichloromethane to obtain the title compound as colorless block crystals.

Experimental details

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

Comment

Photochemical reactions have been widely used in various synthetic reactions [5, 6]. In particular, six-membered rings

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Abstract

C₃₆H₂₈N₂O₂, triclinic, $P\bar{1}$ (no. 2), $a = 9.6248(18)$ Å, $b = 10.848(2)$ Å, $c = 14.099(3)$ Å, $\alpha = 95.822(2)^\circ$, $\beta = 105.907(2)^\circ$, $\gamma = 102.404(2)^\circ$, $V = 1362.4(5)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0497$, $wR_{\text{ref}}(F^2) = 0.1205$, $T = 296$ K.

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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>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1	1.0418(2)	1.15123(17)	0.30480(14)	0.0359(5)
C2	1.0279(2)	1.23880(19)	0.23980(15)	0.0463(5)
H2	1.046052	1.223238	0.178775	0.056*
C3	0.9874(3)	1.3486(2)	0.26547(18)	0.0582(6)
H3	0.978626	1.407067	0.221616	0.070*
C4	0.9596(3)	1.3730(2)	0.35519(19)	0.0626(7)
H4	0.930984	1.446918	0.371662	0.075*
C5	0.9744(3)	1.2877(2)	0.42005(17)	0.0582(6)
H5	0.957256	1.304588	0.481272	0.070*
C6	1.0145(2)	1.1765(2)	0.39554(15)	0.0467(5)
H6	1.023263	1.118688	0.439883	0.056*
C7	1.1596(2)	0.97874(18)	0.34960(14)	0.0369(5)
H7	1.218909	1.028042	0.410983	0.044*
C8	1.15207(19)	0.85346(17)	0.33514(13)	0.0331(4)
C9	0.93683(19)	0.82526(17)	0.19523(13)	0.0330(4)
C10	1.0112(2)	0.95972(17)	0.17917(13)	0.0346(4)
H10	0.933415	0.996761	0.141375	0.041*
C11	1.2436(2)	0.78735(18)	0.40250(14)	0.0364(5)
C12	1.2355(2)	0.6590(2)	0.37511(17)	0.0535(6)
H12	1.172505	0.615042	0.312852	0.064*
C13	1.3198(3)	0.5954(2)	0.4390(2)	0.0741(8)
H13	1.312173	0.509125	0.419541	0.089*
C14	1.4135(3)	0.6576(3)	0.5298(2)	0.0742(8)
H14	1.470081	0.614092	0.572151	0.089*
C15	1.4245(3)	0.7847(3)	0.55912(17)	0.0651(7)
H15	1.488732	0.827492	0.621391	0.078*
C16	1.3406(2)	0.8490(2)	0.49634(15)	0.0511(6)
H16	1.348696	0.935141	0.516954	0.061*
C17	0.8248(2)	0.83210(18)	0.25186(13)	0.0379(5)
C18	0.7252(2)	0.9084(2)	0.22879(16)	0.0508(6)
H18	0.732003	0.961531	0.181570	0.061*
C19	0.6157(3)	0.9056(3)	0.2759(2)	0.0683(7)
H19	0.548975	0.956397	0.259877	0.082*
C20	0.6059(3)	0.8280(3)	0.3463(2)	0.0733(8)
H20	0.531987	0.825872	0.377411	0.088*
C21	0.7043(3)	0.7542(3)	0.37060(17)	0.0657(7)
H21	0.697948	0.702403	0.418739	0.079*
C22	0.8135(2)	0.7559(2)	0.32397(15)	0.0487(5)
H22	0.880182	0.705366	0.341186	0.058*
C23	1.0974(2)	0.83675(18)	0.05483(14)	0.0379(5)
C24	1.2031(2)	0.8255(2)	0.00618(15)	0.0491(5)
H24	1.191612	0.749100	−0.035064	0.059*
C25	1.3242(3)	0.9265(2)	0.01866(19)	0.0647(7)
H25	1.393638	0.918177	−0.014680	0.078*
C26	1.3435(3)	1.0401(2)	0.08032(19)	0.0672(7)
H26	1.423817	1.109200	0.087124	0.081*
C27	1.2429(2)	1.0498(2)	0.13144(16)	0.0504(6)
H27	1.257756	1.125328	0.174715	0.060*
C28	1.1196(2)	0.94990(18)	0.12024(14)	0.0368(5)
C29	0.8661(2)	0.73639(17)	0.09136(13)	0.0359(4)
H29	0.784955	0.768831	0.052087	0.043*
C30	0.8005(2)	0.59670(19)	0.09921(14)	0.0410(5)
C31	0.6562(2)	0.55696(19)	0.12158(14)	0.0441(5)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C32	0.6301(3)	0.4458(2)	0.16224(17)	0.0587(6)
H32	0.701031	0.398341	0.173511	0.070*
C33	0.4998(3)	0.4057(3)	0.1859(2)	0.0751(8)
H33	0.483577	0.331616	0.213484	0.090*
C34	0.3935(3)	0.4748(3)	0.1690(2)	0.0793(8)
H34	0.306045	0.447665	0.185511	0.095*
C35	0.4170(3)	0.5839(3)	0.12767(18)	0.0665(7)
H35	0.345064	0.630358	0.115871	0.080*
C36	0.5475(2)	0.6248(2)	0.10359(15)	0.0518(6)
H36	0.562457	0.698343	0.075133	0.062*
N1	1.08341(17)	1.03895(14)	0.27766(11)	0.0371(4)
N2	0.97297(17)	0.73581(15)	0.03688(11)	0.0432(4)
H2A	0.958820	0.670732	−0.008101	0.052*
O1	0.86915(16)	0.51749(13)	0.08555(11)	0.0539(4)
O2	1.05331(13)	0.77224(11)	0.24903(9)	0.0354(3)

with symmetrical double bond structure are often used as starting materials for various photochemical reactions; for example, 1,4-dihydropyridine [7], 1,4-dihydropyrazine [8] and 1,4-oxazine. Earlier, we used 1,4-oxazine as starting material to obtain 2-oxa-5-azabicyclo compound [9]. This time, a new oxazinoquinoline compound was obtained by irradiation using different solvents.

In the molecules forming the title crystal structure, the tricyclic structure formed by oxazinoquinoline structure is not in one plane. The dihedral angle of the phenyl ring formed by C17–22 and the phenyl ring formed by C31–36 is 23.7°. The dihedral angle between the phenyl ring formed by C11–16 and the phenyl ring formed by C23–28 is 95.2°. Several important bond angle data are involved as follows C7–N1–C10 = 114.87(15) Å, C8–O2–C9 = 114.09(13) Å, C23–N2–C29 = 121.98(15) Å. The bond lengths and angles are in the expected ranges.

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