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SINGLE CRYSTAL X-RAY DIFFRACTION ANALYSIS OF THE STRUCTURE OF METHYL 4-METHYL-3,5-DIOXO-1-PHENYL-2-OXASPIRO[5.5]UNDECANE-4-CARBOXYLATE

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A single crystal X-ray diffraction study of the crystals of methyl 4-methyl-3,5-dioxo-1-phenyl-2-oxaspiro[5.5]undecane-4-carboxylate shows that phenyl and methoxycarbonyl substituents are in the equatorial position on the one side of the dioxopyran ring with *twist boat* conformation.

Keywords: dioxopyran, spiroheterocycles, zinc enolates, single crystal X-ray diffraction analysis .

The interaction of dimethyl 2-(1-bromocyclohexylcarbonyl)-2-methylmalonate with zinc and benzaldehyde has lead to the formation of methyl 4-methyl-3,5-dioxo-1-phenyl-2-oxaspiro- [5.5]undecane-4-carboxylate isolated as one isomer.

In order to study the spatial structure of the synthesized compound, we have performed the single crystal X-ray diffraction analysis. Colorless faceted $C_{19}H_{22}O_5$ crystals of the tetrahedral prismatic shape belong to the triclinic crystal system: $a = 6.530(1)$ Å, $b = 11.106(1)$ Å, $c = 12.526(1)$ Å, $\alpha = 71.94(1)°$, $\beta = 88.03(1)°$, $\gamma = 86.50(1)°$, $V = 861.9(2)$ Å^3, $M = 330.37$, $d_x = 1.273$ g/cm^3, $Z = 2$, P-1 space group. A set of experimental reflections was obtained using and automated four-circle KM-4 (KUMA DIFFRACTION) diffractometer with χ-geometry by $\omega/2\theta$-scanning on monochromated MoK_α radiation ($2\theta \leq 50°$). In total, 3578 reflections were measured, out of which 2720 were independent ($R(\text{int}) = 0.0310$). Absorption was not taken into account ($\mu = 0.092$ mm^{-1}). The structure was solved by the direct method using the SIR92 program [4] with subsequent calculations of the electron density maps. Hydrogen atom were set geometrically. Full-matrix anisotropic refinement (non-hydrogen atoms) by the least squares method was performed using the SHELXL-97 program package [5] and was completed when $R_1 = 0.0443$ and $wR2 = 0.1261$ for 2383 reflections with $I \geq 2\sigma(I)$ and $R_1 = 0.0497$ for all 3578 reflections. 217 parameters were refined; $\Delta\rho_{\max} = 0.208$ Å^{-3}, $\Delta\rho_{\min} = -0.267$ Å^{-3}; *GOOF* 1.044.

The general view of the molecule is shown in Fig. 1. The pyran ring has the *twist boat* conformation. Phenyl and methoxycarbonyl substituents are in the equatorial position. The methoxycarbonyl group plane is bisectorial against the heterocycle. The phenyl ring plane forms an angle of 72.3° with the O1, C4, and C5 atom plane; the O1C5C6C7 torsion angle is 138.9°. All bond lengths and bond angles are within the range of standard values for the corresponding atoms [1]. They also comply with the results of the studied dioxopyrans of a different structure [2, 3]. Table 1 lists the bond lengths in the pyran heterocycle. No short intermolecular contacts were found in the crystal.

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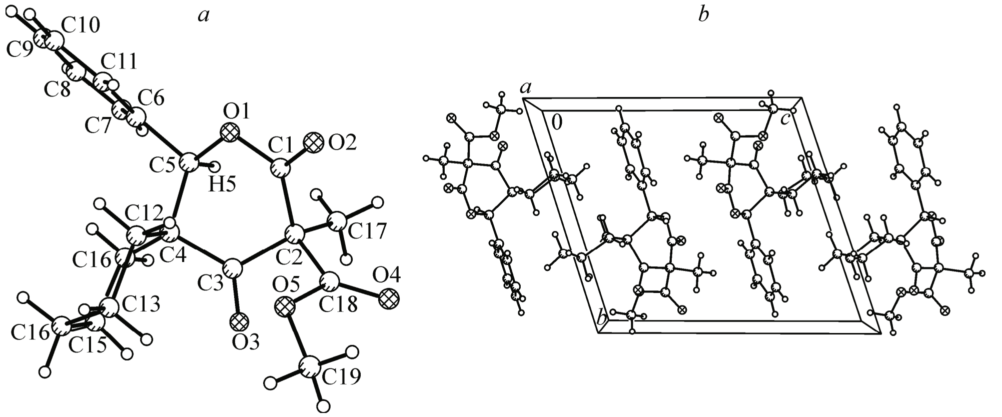

Fig. 1. General view of the molecule.

TABLE 1. Bond Lengths in the Pyran Ring and Its Substituents, *d* (Å)

Bond	*d*	Bond	*d*	Bond	*d*	Bond	*d*
O(1)–C(1)	1.343(2)	C(2)–C(18)	1.525(2)	O(1)–C(5)	1.461(2)	C(2)–C(3)	1.537(2)
O(2)–C(1)	1.197(2)	C(2)–C(17)	1.543(2)	O(3)–C(3)	1.206(2)	C(3)–C(4)	1.519(2)
O(4)–C(18)	1.193(2)	C(4)–C(16)	1.535(2)	C(4)–C(12)	1.546(2)	C(4)–C(5)	1.559(2)
C(1)–C(2)	1.525(2)	C(5)–C(6)	1.509(2)				

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