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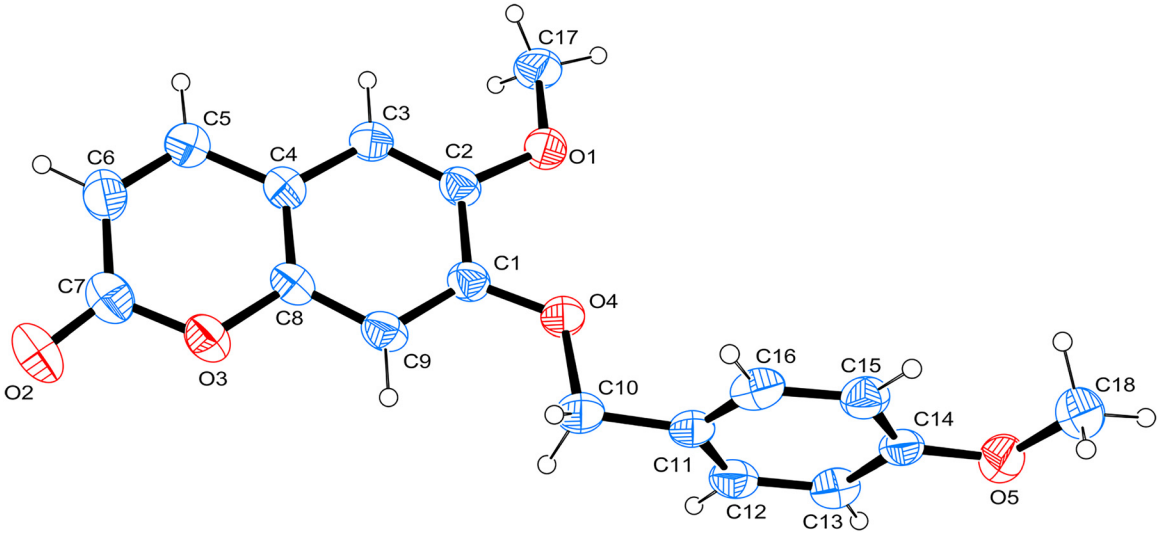
Synthesis and crystal structure of 6-methoxy-7-[(4-methoxyphenyl)methoxy]-2H-1-benzopyran-2-one, $C_{18}H_{16}O_5$



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Abstract

$C_{18}H_{16}O_5$, Triclinic, $P\bar{1}$ (no. 2), a = 7.6389(9) Å, b = 9.1335(11) Å, c = 10.8946(13) Å, α = 90.983(2)°, β = 90.398(2)°, γ = 93.522(2)°, V = 758.54(16) Å^3, Z = 2, $R_{gt}(F)$ = 0.0433, w$R_{ref}(F^2)$ = 0.1169, T = 296(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data. The list of the atoms including atomic coordinates and displacement parameters can be found in the cif-file attached to this article.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.24 × 0.18 × 0.11 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω scans
θ_{max}, completeness:	25.7°, 100 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8452, 2872, 0.029
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 1,603
$N(param)_{refined}$:	210
Programs:	Bruker,[1] SHELX[2,3], WinGx[4]

1 Source of material

All chemicals were purchased from commercial sources and used as received without further purification. The scopoletin (1.0 mmol, 0.192 g), 4-methoxybenzylchloride (3.0 mmol, 0.469 g), potassium carbonate (2 mmol, 0.276 g), and sodium iodide (0.1 mmol, 0.015 g) was suspended in anhydrous *N*,*N*-dimethylformamide (30 mL) in a dry round bottom flask at room temperature. The mixture was warmed to 60 °C and stirred for 12 h under N_2. All solvents were removed by vacuum distillation to obtain a crude product. The residue was purified by column chromatography on silica gel to yield the

pure title compound. The crystal was obtained by slow evaporating chloroform at room temperature.

2 Experimental details

All H-atoms bonded to C atoms were placed geometrically and refined using a riding model with common isotropic displacement factors U_{iso}(H) = 1.2 or 1.5 U_{eq} (parent C-atom).

3 Comment

Natural products have always been an important source of medicines for the treatment of various diseases. Among the various natural product scaffolds, benzopyrone compounds play an important role in biologically active compounds.[5] Scopoletin is a natural benzopyrone compound extracted from the ethnopharmacology *Prismatomeris tetrandra* (Prismatomerisconnata Y. Z. Ruan), which has various biological activities.[6–8] Currently, the benzopyrone class of compounds has shown great potential in drug development. Therefore, the synthesis and crystal structure of the benzopyrone derivative are important significance for the study of the application.

Single-crystal structure analysis revealed that the title compound crystallized in the triclinic space group $P\bar{1}$. The asymmetric unit of the title compound 6-methoxy-7-[(4-methoxyphenyl)methoxy]-*2H*-1-benzopyran-2-one molecule. The ORTEP diagram is presented in figure. Bond lengths and angles are all in the expected ranges. All atoms on the benzopyrone scaffold structure are in the same plane, and the bond lengths of C7=O2, C7=O3 and C8=O3 are 1.204(2) Å, 1.380(3) Å and 1.378(2) Å, respectively, they are consistent with the results reported for similar compounds in the literature.[9]

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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