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Synthesis and Spatial Structure of (*E*)-1-(2-(4-Bromobutoxy)-6-Hydroxy-4-Methoxyphenyl-3-Phenylprop-2-en-1-one

(*E*)-1-(2-(4-bromobutoxy)-6-hydroxy-4-methoxyphenyl-3-phenylprop-2-en-1-one was synthesized from the pinostrobin molecule. The tetrahydropyran cycle was opened in acetone under heating (50–60 °C) mixtures of components for 16 hours in the presence of 3 moles of K_2CO_3 and 1,4-dibromobutane. The resulting substance is a yellow powder of the composition $C_{20}H_{21}BrO_4$, mp 83.7–86.6 °C. The structure of the obtained compound was established on the basis of the data of elemental analysis, IR and NMR spectra. As a result of X-ray diffraction analysis, it was found that the hydrogen atoms in the C8=C9 bond take the trans-conformation. The rotation of the C1…C6 phenyl ring (flat with an accuracy of ±0.008 Å) relative to the C10…C15 cycle (flat with an accuracy of ±0.004 Å) is 14.3°. In the crystal, the molecules are linked by an intramolecular hydrogen bond O4-H....O1 (distances O-H 0.95(8) Å, O···O 2.469(6) Å, H···O 1.58 (8) Å, angle O-H···O 153(7)°). The formation of (*E*)-1-(2-(4-bromobutoxy)-6-hydroxy-4-methoxy-1-phenylprop-2-en-1-one can be explained by the ease of the retro-Michael reaction of the pyran ring and the subsequent O-alkylation of the resulting chalcone. Carrying out the reaction in the presence of other bases (cesium carbonate, triethylamine) did not lead to success. The starting substance (pinostrobin) was completely converted into oligomeric compounds.

Keywords: NMR spectroscopy, IR spectroscopy, X-ray analysis, (*E*)-1-(2-(4-bromobutoxy)-6-hydroxy-4-methoxyphenyl-3-phenylprop-2-en-1-one, chalcone, flavone, phenolic compounds.

Introduction

Compounds of a number of chalcones are of interest due to the simplicity of their synthesis, as well as the possibility of using them as synthons to obtain various heterocyclic compounds with high biological activity, in particular, pyrazolines and flavones [1, 2]. Compounds containing a chalcone skeleton exhibit significant antibacterial, antiviral, antihyperglycemic, antifungal, anti-inflammatory, antimalarial, antitumor, and immunomodulatory activities, as well as chemoprotective and antioxidant properties [3–8]. In this regard, the synthesis of new chalcones seems to be an important task.

The flavonoid pinostrobin (**1**) is of particular interest for chemical modification. The availability of pinostrobin, previously isolated from the extract of the buds of *Populus balsamifera* L. [9, 10], and the analysis of its structure leads to the conclusion that its synthetic transformations are promising to obtain compounds structurally similar to a number of natural metabolites and other practically significant substances.

Experimental

The melting point was determined on a Boetius heating table. Elemental analysis was carried out on a Eurovector 3000 analyzer. TLC analysis was performed on Sorbfil plates, visualization with iodine vapor.

The IR spectrum was recorded on an Avatar 360 instrument (Thermo Nicolet) in KBr tablets.

^{1}H NMR spectra were recorded on a Bruker AV-400 spectrometer with a frequency of 400 MHz. ^{13}C NMR spectra were recorded on a Bruker AV-400 spectrometer with a frequency of 100 MHz. Solvent — $CDCl_3$, standard — TMS. The X-ray diffraction experiment was carried out on a Bruker APEX-II CCD diffractometer.

Synthesis of (E)-1-(2-(4-bromobutoxy)-6-hydroxy-4-methoxyphenyl -3-phenylprop-2-en-1-one (**2**) a yellow powder, composition $C_{20}H_{21}BrO_4$, mp 87–89 °C. To a solution of 0.5 g (1.85 mmol) of pinostrobin in 10 ml of acetone, 0.37 g (2.7 mmol) of calcined K_2CO_3 was added, and after 10 min, 1.2 equivalents (2.2 mmol) of 1,4-dibromobutane were added. The reaction mixture was stirred for 7 hours at 50 °C, 10 ml of acetone were added and stirred for another 7 hours at 50 °C. After cooling, the precipitate was filtered off, washed with acetone (3×5 ml), the combined filtrates were evaporated, the residue was recrystallized from diethyl ether, compound 2 was isolated. Yield is 72 %, mp is 87–89 °C (from ether). HPLC purity is 98.72 %. Control over the course of reactions and the purity of the obtained product were carried out by TLC on Sorbfil plates using the system petroleum ether-ethyl acetate, 2:1, R_f=0.52.

The addition of an excess (1.2 equiv.) of dibromide contributed to increasing the yield of the target product **2**. The formation of compound **2** can be explained by the ease of the retro-Michael reaction of the pyran ring and the subsequent O-alkylation of the resulting chalcone. It should be noted that the result of the conversion of pinostrobin significantly depends on the nature of the dibromoalkane. Carrying out the reaction in the presence of other bases (cesium carbonate, triethylamine) also did not lead to success. The starting substance (pinostrobin) was completely converted into oligomeric compounds.

Elemental analysis: found, %: C 61.43, H 5.02, Br 18.96. calculated, %: C 59.25, H 5.18, Br 19.73.

IR spectrum (v, cm^{-1}, KBr): 3053 (OH), 2924 (OCH_3), 1631 (C=O), 1581 (C=C), 1495, 1444, 1346 (CH_3), 1112 (C-O-S), 1033 (Arom), 866, 693, 624.

X-ray diffraction study of compound **2**. Determination of unit cell parameters and intensity of 17139 diffracted reflections (3472 independent, R_{int} 0.0696) were measured on a Bruker Kappa APEX II CCD diffractometer (MoK_α, graphite monochromator, ω-scan, 2.635° ≤ θ ≤ 25.817°) at 200 K. Monoclinic crystals, space group P21/n, *a*=6.9892(12), *b*=14.249(2), *c*=18.413(3) Å, β=92.222(6)°, *V*=1832.3(5) Å^3, *Z*=4 ($C_{20}H_{21}BrO_4$), calculated density *d*=1.469 g/cm^3, absorption coefficient μ=2.264 mm^{-1}. The experimental array of reflections was corrected for absorption. Takeover accounting was carried out using SAINT [11] and SADABS [12] programs (multi-scan method, $T_{min.}$ 0.812, $T_{max.}$ 0.922).

The structure was deciphered by the method of direct phase determination. The coordinates of non-hydrogen atoms were refined taking into account the anisotropic approximation of thermal vibrations by the full-matrix least-squares method. The positions of hydrogen atoms, with the exception of hydroxyl ones, were calculated geometrically and refined in the isotropic approximation of thermal vibrations (rider model). The H atoms of the hydroxyl groups were revealed from the difference synthesis, and their positions were refined in the isotropic approximation. The structure was deciphered and the coordinates were refined using the SHELXS software package [13] and the SHELKS-2018/3 software [14]. 2268 independent reflections with I ≥ 2σ(I) were used in the calculations; the number of refined parameters was 231.

The divergence coefficients were R_1=0.0736, $_wR_2$=0.1961 (for reflections with I≥2σ(I)), R_1=0.1162, $_wR_2$=0.2139 (for all reflections), GooF=1.126. Maximum and minimum residual density: Δρ=1.522 and -0.527 e/Å^3. The coordinates of atoms in the crystal are presented in Table 1.

Table 1

Coordinates of the atoms in the fractions of the cell (x10^4, Hx10^3) and equivalent thermal parameters (Å^2, x10^3) in the structure 2

Atom	*x*	*y*	*z*	$U_{equiv.}$
Br1	8262(2)	9067(1)	6987(1)	68(1)
O1	7920(7)	5134(3)	3379(2)	44(1)
O2	7286(6)	5664(3)	5568(2)	34(1)
O3	7257(7)	2440(3)	6089(2)	42(1)
O4	7969(6)	3437(3)	3652(2)	40(1)

Continuation of Table 1

Atom	*x*	*y*	*z*	$U_{equiv.}$
C1	7675(8)	4616(4)	4586(3)	28(1)
C2	7382(8)	4750(4)	5345(3)	29(1)
C3	7228(8)	4016(4)	5816(3)	31(1)
C4	7377(8)	3104(4)	5563(4)	34(1)
C5	7652(8)	2915(4)	4839(3)	34(1)
C6	7754(8)	3658(4)	4358(3)	35(1)
C7	7844(8)	5352(4)	4039(3)	32(1)
C8	7869(9)	6360(4)	4219(3)	36(1)
C9	7437(8)	7005(4)	3734(3)	34(1)
C10	7408(9)	8017(5)	3864(3)	36(2)
C11	7229(9)	8649(5)	3285(4)	40(2)
C12	7233(12)	9608(5)	3393(4)	53(2)
C13	7458(10)	9965(5)	4090(4)	49(2)
C14	7631(10)	9365(5)	4673(4)	46(2)
C15	7621(9)	8399(5)	4572(3)	36(1)
C15	7085(9)	5861(4)	6333(3)	34(1)
C17	6989(9)	6918(4)	6388(3)	32(1)
C18	6678(10)	7217(5)	7166(4)	40(2)
C19	6166(10)	8237(5)	7232(4)	50(2)
C20	7385(10)	1472(4)	5879(4)	44(2)
H04	807(11)	405(5)	341(4)	5(2)

Results and Discussions

Based on pinostrobin (**1**), (*E*)-1-(2-(4- bromobutoxy)-6-hydroxy-4-methoxyphenyl-3-phenylprop-2-en-1-one (**2**) was synthesized by opening the tetrahydropyran cycle (Fig. 1). The synthesis of compound **2** was carried out by the interaction of pinostrobin with 1,4-dibrombutane in acetone in the presence of K_2CO_3.

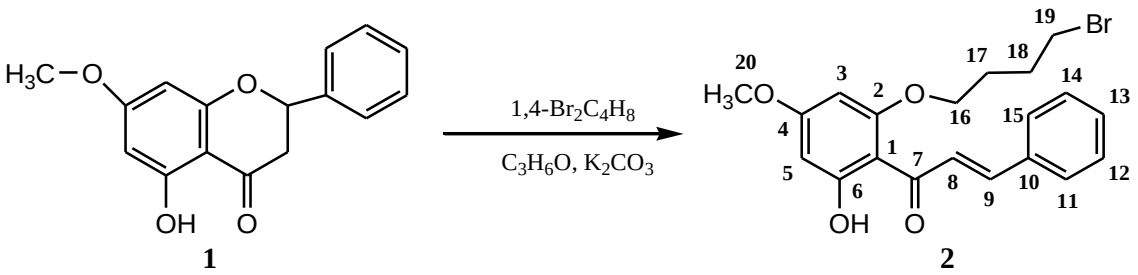

Figure 1. Synthesis scheme of (*E*)-1-(2-(4-bromobutoxy)-6-hydroxy-4-methoxyphenyl-3-phenylprop-2-en-1-one **(2)**

Structure of the synthesized compound **2** was proved by IR, ^{1}H and ^{13}C NMR spectroscopy [15].

The IR spectrum contains bands corresponding to the stretching vibrations of the OH group involved in the hydrogen bond (3053 cm^{-1}), OCH_3, C=O, C=C (2924, 1631, 1581 cm^{-1}, respectively).

The ^{1}H NMR spectrum of molecule **2** is characterized by the presence of signals of protons of substituted phenyl rings at 6.10–7.90 ppm and signal of proton of the hydroxyl group at 12.17 ppm. The aliphatic protons are observed in the area of 2.02–4.07 ppm. The protons of the methoxy group appear at 1.83 ppm as a singlet.

In the ^{13}C NMR spectrum of compound **2**, the signals of carbons of phenyl rings are observed in the range of 91.85–135.44 ppm. Carbon atoms C=C appear at 127.85 and 130.12 ppm, C=O — at 192.72 ppm. Aliphatic carbon atoms resonate in the region of 28.04-67.96 ppm and the methoxy carbon at 55.62 ppm.

Detailed assignment of ^{1}H and ^{13}C NMR signals in molecule **2** is given in Table 2.

Table 2

^{1}H and ^{13}C NMR data (400 and 100 MHz, CDCl3, δ, ppm, J/Hz) for molecule 2

Atom	δH	δC	Atom	δH	δC
1	–	106.37, s	12	7.39, m (1H)	128.95, d
2	–	161.65, s	13	7.25, m (1H)	135.44, s
3	6.10, d (1H, J=2,5)	91.85, d	14	7.41, m (1H)	128.95, d
4	–	168.23, s	15	7.25, m (1H)	128.20, d
5	5.93, d (1H, J=2,5)	93.87, d	16	4.07, m (2H)	67.96, t
6	–	166.19, s	17	2.03, m (2H)	29.28, t
7	–	192.72, s	18	2.02, m (2H)	28.04, t
8	7.90, d (1H, J=15.6)	130.12, d	19	3.30, m (2H)	38.20, t
9	7.57, d (1H, J=15.6)	127.85, d	OH	12.17, s (1H)	–
10	–	130.12, d	OCH3	3.83, s (3H)	55.62, q
11	7.56, m (1H)	128.20, d			

Based on the data of elemental analysis and IR, NMR spectra, the resulting substance was identified as (*E*)-1-(2-(4-bromobutoxy)-6-hydroxy-4-methoxyphenyl-3-phenylprop-2-en-1-one. The spatial structure of compound **2** was determined by X-ray diffraction.

The general view of compound **2** is shown in Figure 2. It follows from the data obtained that the bond lengths and valence angles in compounds **2** are close to the usual ones [16].

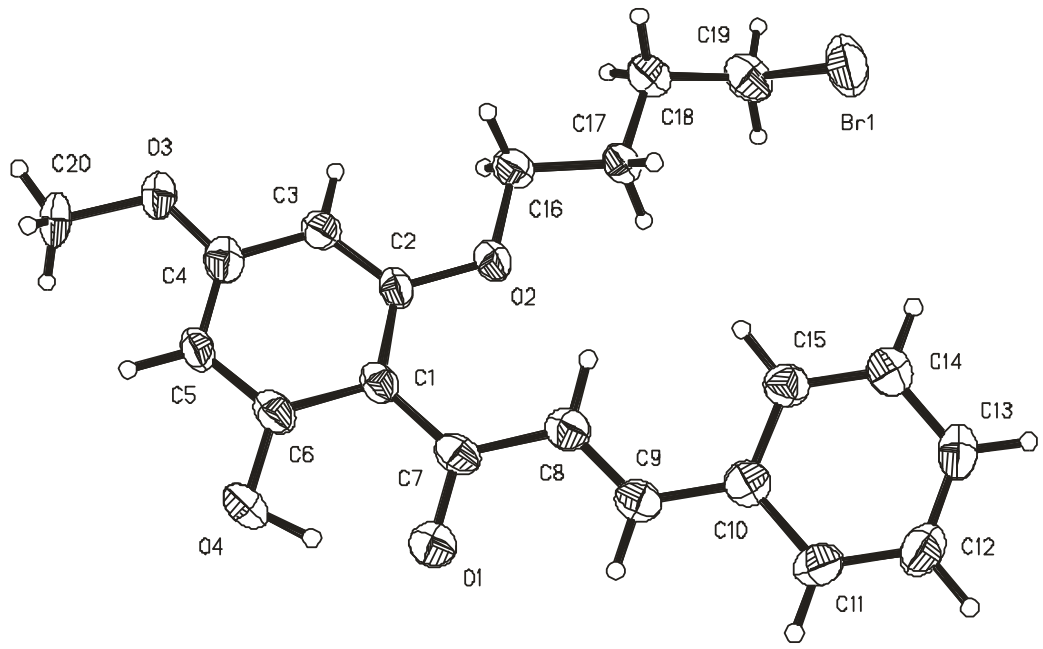

Figure 2. Crystal structural of (*E*)-1-(2-(4-bromobutoxy)-6-hydroxy-4-methoxyphenyl-3-phenylprop-2-en-1-one **(2)** (thermal vibration ellipsoids shown with a probability of 50 %)

Table 3

Bond lengths (d, Å) in the structure 2

Bond	*d*	Bond	*d*
Br1-C19	1.949(7)	C5-C6	1.385(9)
O1-C7	1.258(7)	C7-C8	1.474(9)
O2-C2	1.368(7)	C8-C9	1.309(9)
O2-C16	1.448(7)	C9-C10	1.461(9)
O3-C4	1.359(8)	C10-C11	1.398(9)
O3-C20	1.436(7)	C10-C15	1.415(9)
O4-C6	1.353(7)	C11-C12	1.381(10)

Continuation of Table 3

Bond	*d*	Bond	*d*
C1-C6	1.429(9)	C12-C13	1.384(11)
C1-C2	1.432(8)	C13-C14	1.375(10)
C1-C7	1.462(9)	C14-C15	1.389(10)
C2-C3	1.365(8)	C16-C17	1.511(8)
C3-C4	1.386(8)	C17-C18	1.519(9)
C4-C5	1.380(9)	C18-C19	1.502(10)

Table 4

Valent angles (ω, deg.) in the structure 2

Angle	ω	Angle	ω
C2-O2-C16	119.0(5)	O1-C7-C8	117.2(6)
C4-O3-C20	118.1(5)	C1-C7-C8	123.0(5)
C6-C1-C2	115.0(5)	C9-C8-C7	122.1(6)
C6-C1-C7	118.5(5)	C8-C9-C10	125.8(6)
C2-C1-C7	126.5(5)	C11-C10-C15	117.2(6)
C3-C2-O2	122.1(5)	C11-C10-C9	120.8(6)
C3-C2-C1	122.4(5)	C15-C10-C9	122.0(6)
O2-C2-C1	115.5(5)	C12-C11-C10	121.9(6)
C2-C3-C4	119.7(6)	C11-C12-C13	119.8(7)
O3-C4-C5	124.5(6)	C14-C13-C12	119.9(7)
O3-C4-C3	114.0(6)	C13-C14-C15	120.8(7)
C5-C4-C3	121.5(6)	C14-C15-C10	120.4(6)
C4-C5-C6	118.7(6)	O2-C6-C17	105.4(5)
O4-C6-C5	116.6(6)	C16-C17-C18	110.5(5)
O4-C6-C1	120.8(6)	C19-C18-C17	113.0(6)
C5-C6-C1	122.6(5)	C18-C19-Br1	112.6(5)
O1-C7-C1	119.8(5)		

Hydrogen atoms in the C8=C9 bond have a trans-conformation. The atom of the O1 ketone group is practically located in the plane of the 1,2,4,6-substituted phenyl cycle (torsion angle C6-C1-C7-O1 4.8(8)°). The keto group and the double bond C8=C9 deviate significantly from the coplanarity (torsion angle O1-C7-C8-C9 19.4(9)°). The double bond C8=C9 and C10-substituted phenyl cycle are also somewhat less conjugated (torsion angle C8-C9-C10-C15 -8.4(9)°). In general, the reversal of the phenyl cycle C1... C6 (flat with an accuracy of ± 0.008 Å) relative to the cycle C10...C15 (flat with an accuracy of ± 0.004 Å) is 14.3°.

In the crystal, the molecules are linked by an intramolecular hydrogen bond O4-H....O1 (x, y, z) (distances O-H 0.95(8) Å, O···O 2.469(6) Å, H···O 1.58 (8) Å, angle O-H···O 153(7)°) (Fig. 3).

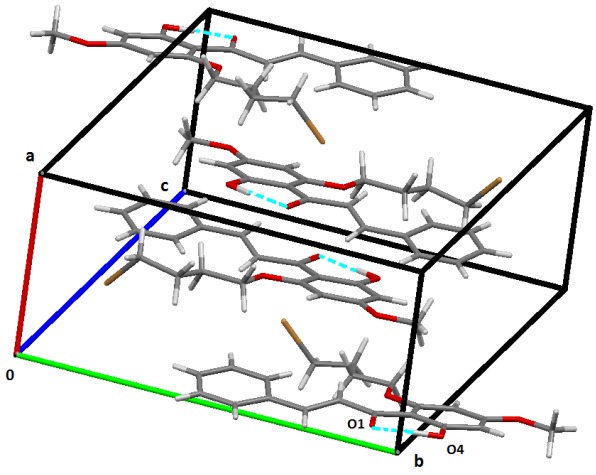

Figure 3. Crystal packing of molecules (*E*)-1-(2-(4-bromobutoxy)-6-hydroxy-4-methoxyphenyl-3-phenylprop-2-en-1-one (**2**)

Conclusions

(*E*)-1-(2-(4-bromobutoxy)-6-hydroxy-4-methoxyphenyl-3-phenylprop-2-en-1-one was synthesized from the pinostrobin molecule by opening the tetrahydropyran ring. Synthesis of (*E*)-1- (2-(4-bromobutoxy)-6-hydroxy-4-methoxyphenyl-1-phenylprop-2-en-1-one was carried out by the interaction of pinostrobin with 1,4-dibromobutane in acetone in the presence of K_2CO_3. The structure of the obtained substance was established on the basis of elemental analysis data and IR and NMR spectra. It follows from the constant of the vicinal spin-spin interaction of H atoms at C8 and C9 that they must have cis conformations. Such a conformation would lead to a significant thermal stress between the O1 atom of the keto group and the phenyl ring. As a result of X-ray diffraction analysis, it was found that hydrogen atoms in the C8=C9 bond have a trans-conformation. The reversal of the cycle C1...C6 (flat with an accuracy of ± 0.008 Å) relative to the cycle C10...C15 (flat with an accuracy of ± 0.004 Å) is 14.3°. In the crystal, the molecules are bound by an intramolecular hydrogen bond O4-H....O1.

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