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# PLANT COUMARINS. 2. BECKMANN REARRANGEMENT OF OREOSELONE *E*- AND *Z*-OXIMES

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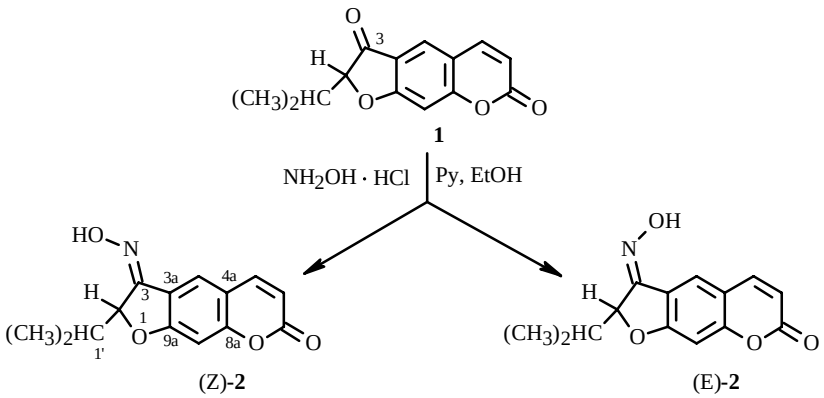
*Oximation of oreoselone to produce a mixture the E- and Z-oximes was investigated. The crystal and molecular structures of oreoselone Z-oxime and the Beckmann rearrangement product of oreoselone E- or Z-oximes and $PCl_5$, 7-(1-chloro-2-methylpropoxy)-2-oxo-2H-1-benzopyran-6-carbonitrile, were established by XSA. Hydrolysis of the latter produced 7-hydroxy-2-oxo-2H-1-benzopyran-6-carbonitrile.*

**Key words:** coumarins, oreoselone, oxime, 7-(1-chloro-2-methylpropoxy)-2-oxo-2H-1-benzopyran-6-carbonitrile, 7-hydroxy-2-oxo-2H-1-benzopyran-6-carbonitrile, XSA, NMR spectroscopy.

In the previous report [1] we prepared a series of peucedanin derivatives by modifying the furan ring. In order to expand the number of derivatives, which are potentially promising as synthons for preparing antiviral (anti-HIV) [2] and antitumor [3] agents, we attempted to prepare derivatives from racemic oximes of oreoselone (**1**), which is easily prepared by hydrolysis of peucedanin.

The synthesis of racemic oreoselone oxime {3-hydroxyimino-2-(1-methylethyl)-7*H*-furo[3,2-*g*][1]benzopyran-7-one} of undetermined configuration with mp 200-202°C (dec., from EtOH) was previously reported [4]. Classical oximation [5] of **1** by a solution of the free base produced by alkaline neutralization of an aqueous alcohol solution of hydroxylamine hydrochloride (the excess of base was 3.2 mole relative to oreoselone) was used. We found by PMR that the product produced this way [4] was a mixture of the two isomeric oreoselone oximes, 3*Z*- and 3*E*-hydroxyimino-2-(1-methylethyl)-7*H*-furo[3,2-*g*][1]-benzopyran-7-one [(*Z*)-**2** and (*E*)-**2**].

In order to avoid the large excess of hydroxylamine, we investigated oximation of racemic **1** by boiling with an alcoholic pyridine solution of hydroxylamine hydrochloride (mole ratio **1**:hydroxylamine hydrochloride:pyridine 1.00:1.05:2.00). According to PMR spectra, the reaction produced in 52% yield a crystalline mixture of (*Z*)-**2** and (*E*)-**2** with an *E*:*Z* ratio of about 1:0.7. The low yield of the mixture of oxime isomers under these conditions is apparently due to the formation of side products via reaction of hydroxylamine with the lactone ring of **1** and (or) its hydroxyimino derivatives (cf. the reaction of coumarin and hydroxylamine [6, 7]).




(Z)-**2** (E)-**2**

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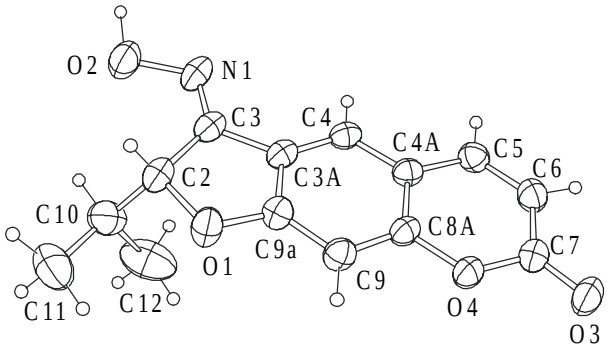



Fig. 1

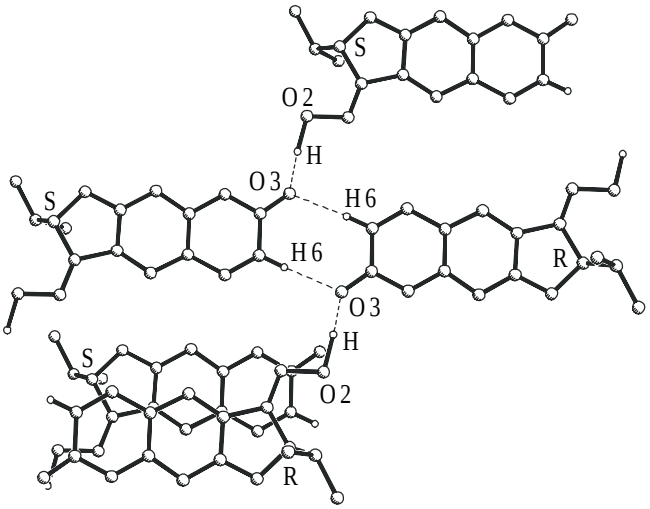



Fig. 2

Fig. 1. Molecular structure of (*Z*)-**2** and selected bond lengths from the XSA: O(1)–C(9A) 1.353(2), O(1)–C(2) 1.467(2), C(3)–N(1) 1.290(3), O(2)–N(1) 1.403(2), C(7)–O(3) 1.208(2), C(7)–O(4) 1.375(2), O(4)–C(8A) 1.377(2) Å.
Fig. 2. Crystal packing of (*Z*)-**2** [configurations of the C2 chiral centers (*R* or *S*) are shown].

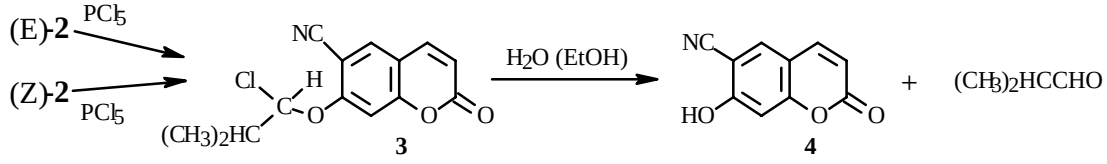
Fractional crystallization of the mixture of oximes prepared in the alcoholic pyridine solution isolated both isomers. The *Z*-configuration was established by an x-ray structure analysis (XSA) for a single crystal of the pure compound with mp 242-244°C (dec.).

Figure 1 shows the molecular structure of (*Z*)-**2** and selected bond lengths from the XSA. All atoms of the framework of (*Z*)-**2** lie in a single plane [mean-square deviation 0.016 Å; maximum deviation from the plane, 0.045(2) Å for C2]. The oxime group also lies practically in this same plane. Atoms N1 and O2 deviate from the plane by 0.114 and 0.149 Å, respectively. The torsion angle C2C3N1O2 is 0.3(3)°. Thus, the oxime group has the *E*-configuration. The bond lengths and angles in (*Z*)-**2** are within 3σ of the statistical average values [8]. It should be noted only that the O1–C9A bond is shorter [1.353(2) Å] than the statistical average value of 1.374(13) Å. Among the 40 structures with a linear furocoumarin framework in the Cambridge crystallographic database [9], none were found with an exocyclic double bond in the 3-position.

Figure 2 shows a portion of the crystal packing of (*Z*)-**2**. The molecules form infinite chains through H-bonds. The coordinates of the OH hydrogen shifted upon refinement rather significantly toward the ketone in the neighboring molecule: O(2)–H 1.28(5), O(2)...O(3) 2.715(2) Å, H...O(3) 1.46(5) Å, and O(2)–H...O(3) 164(4)°. These chains, in turn, are bonded to each other through short intermolecular O...H contacts: O(3)...H(6) 2.47(2) Å. Pairs of neighboring molecules can be found in the crystal packing of (*Z*)-**2** that form head—tail dimers through π-stacking interactions of the aryl rings (interplanar distance 3.49, distance between aryl-ring centers 3.86 Å). These dimers bind the double chains pairwise to each other to form a one-dimensional supramolecular motif in the crystal. It should be noted that weak nonbonded interactions are at present of great interest owing to their important role in the regulation of the packing of organic molecules in crystalline solids.

The structure of the isomeric oxime (*E*)-**2** [mp 193-195°C (dec.)] was established by comparing the PMR and $^{13}C$ NMR spectra of the isomers and by elemental analysis.

We attempted to perform a Beckmann rearrangement (BR) on the prepared oximes using in the first step $PCl_5$. The predominant crystalline product (70-75% yield) from reaction of (*E*)-**2** or (*Z*)-**2** with $PCl_5$ was 7-(1-chloro-2-methylpropoxy)-2-oxo-2*H*-1-benzopyran-6-carbonitrile (**3**), the formation of which can be represented as a type II BR [10], which assumes cleavage of the $C^2$–$C^3$ bond in the intermediates formed from (*E*)-**2** or (*Z*)-**2**.



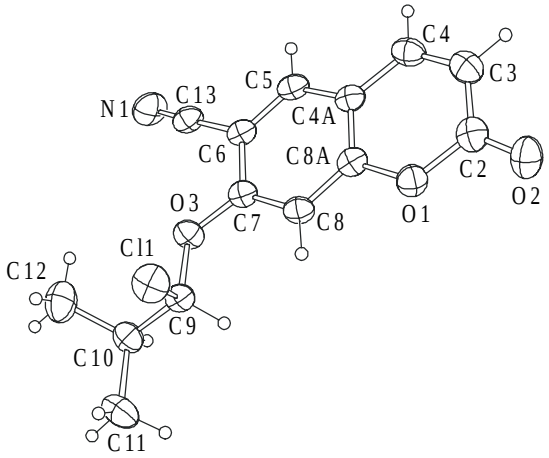



Fig. 3

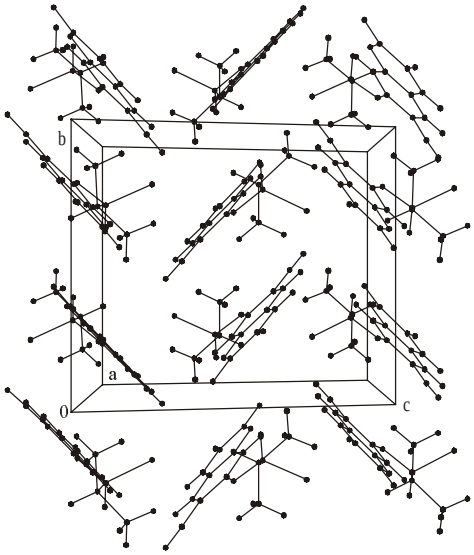



Fig. 4

Fig. 3. Molecular structure of **3** and selected bond lengths (Å) and torsion angles (°) from the XSA: O(1)–C(8A) 1.371(3), O(1)–C(2) 1.386(3), C(2)–O(2) 1.202(3), C(7)–O(3) 1.356(3), O(3)–C(9) 1.410(3), Cl(1)–C(9) 1.808(2), C(6)–C(13) 1.441(4), C(13)–N(1) 1.133(3), C(7)O(3)C(9)Cl(1) 72.4(2), C(7)O(3)C(9)C(10) -166.1(2), O(3)C(9)C(10)C(12) -57.0(3).
Fig. 4. Crystal packing of **3** along the *a* axis.

The structure of **3** was established by a single-crystal XSA. Figure 3 shows the molecular structure of **3** and selected bond lengths from the XSA. All atoms of the bicyclic framework of **3** lie in a single plane with a mean-square deviation of 0.008 Å. Atoms O(2), O(3), and the cyano group C(13)≡N(1) are practically in this same plane with deviations from the plane of 0.026(3), 0.035(2), 0.020(3), and 0.069(4) Å, respectively. The chlorobutyl group eclipses C(8) with a C(9)O(3)C(7)C(8) torsion angle of 5.5(3)°. The ethyl group in 7-ethoxycoumarin has a similar orientation [11]. The bond lengths and angles in **3** are within 3σ of the statistical average values [12].

The crystal packing of **3** (Fig. 4) shows centrosymmetric pairs of molecules that form head—tail dimers through π-stacking interactions of the aryl rings [interplanar distance 3.55; distance between centers, 3.861(1) Å].

Brief (0.5 h) boiling in aqueous alcohol cleaves **3** to form 7-hydroxy-2-oxo-2*H*-1-benzopyran-6-carbonitrile (**4**) and isobutyraldehyde.

Thus, the *E*- and *Z*-oximes of **1** were prepared. The crystal and molecular structures of the *Z*-oxime and of the BR product of the *E*- or *Z*-oxime and $PCl_5$ -[7-(1-chloro-2-methylpropoxy)-2-oxo-2*H*-1-benzopyran-6-carbonitrile] were established by XSA. Hydrolysis of the latter gave 7-hydroxy-2-oxo-2*H*-1-benzopyran-6-carbonitrile.

## EXPERIMENTAL

We used freshly distilled solvents and pure-grade reagents. Recemic **1** was prepared by hydrolysis of peucedanin by the literature method [13] with a modification [1]. Melting points were determined on a Kofler apparatus. IR spectra were recorded on a Vector 22 spectrometer in KBr disks; UV spectra, on a Specord UV—Vis spectrophotometer in ethanol ($c$ $10^{-4}$ M). Molecular weights and elemental composition of new compounds were determined using a high-resolution mass spectrometer (Finnigan MAT, model 8200, EI, 70 eV). NMR spectra were obtained on Bruker AC 200 (working frequency $^1$H 200.13 MHz, $^{13}$C 50.32 MHz), Bruker AM-400 ($^1$H 400.13 MHz, $^{13}$C 100.61 MHz), and Bruker DRX-500 ($^1$H 500.13 MHz, $^{13}$C 125.76 MHz) instruments using solutions (10%) at 25°C and resonances stabilized on the solvent D signal ($CDCl_3$ or DMSO-$d_6$). Chemical shifts (ppm) were measured relative to those of internal standards $CHCl_3$ ($\delta_H$ 7.24 ppm and $\delta_C$ 76.90 ppm) and DMSO ($\delta_H$ 2.50 ppm and $\delta_C$ 39.50 ppm). The multiplicity of signals in the $^{13}$C NMR spectra was determined by standard methods with J-modulation (JMOD) and with off-resonance irradiation of protons. Signals in the PMR and

$^{13}$C NMR of (*Z*)-**2** and (*E*)-**2** were assigned using data for the model compound **1** [1, 14]. Signals in $^{13}$C NMR spectra of **3** were assigned using various types of C—H correlation. Two-dimensional $^{13}$C—$^{1}$H spectra (COSY 125 Hz, COLOC 7-10 Hz) were recorded on a Bruker DRX-500 instrument using standard Bruker programs. Analytical TLC was performed on Sorbfil UV 254 plates (ZAO Sorbpolimer, Krasnodar, RF) with AcOEt:hexane (1:2 by vol) eluent. Mobilities ($R_f$) for **1**, (*E*)-**2** oxime, and (*Z*)-**2** oxime were 0.65, 0.55, and 0.30, respectively, with $R_f$ of (**3**) > $R_f$ of (*E*)-**2** oxime.

**X-ray structure analyses** were carried out on a Bruker P4 diffractometer (Mo Kα-radiation, graphite monochromator, 2θ/θ-scanning, 2θ < 52°). A light yellow crystalline prism of (*Z*)-**2** of dimensions 0.50 × 0.30 × 0.17 mm was selected. The crystal was triclinic and racemic, *a* = 5.9047(7), *b* = 10.015(1), *c* = 11.039(1) Å, α = 87.25(1)°, β = 78.777(8)°, γ = 81.500(9)°, V = 633.2(1) Å$^3$, space group *P*1, *Z* = 2, $C_{14}H_{13}NO_4$, $d_{calc}$ = 1.360 g/cm$^3$, μ = 0.101 mm$^{-1}$.

Intensities of 2212 independent reflections were measured. Absorption corrections were applied using the crystal faces (transmission 0.97-0.98). For **3**, a colorless crystalline plate of dimensions 0.60 × 0.30 × 0.1 mm was selected. The crystal was monoclinic, *a* = 12.762(2), *b* = 9.697(1), *c* = 11.331(2) Å, β = 105.27(1)°, V = 1352.7(4) Å$^3$, space group $P2_1/c$, *Z* = 4, $C_{14}H_{12}ClNO_3$, $d_{calc}$ = 1.364 g/cm$^3$, μ = 0.285 mm$^{-1}$.

Intensities of 2650 independent reflections were measured. Absorption corrections were not applied. The structures were solved by direct methods using the SHELXS-97 program [15]. Structure factors were refined by full-matrix anisotropic least-squares techniques using the SHELXL-97 program [15]. Factors for H atoms were calculated in each refinement cycle using coordinates of the corresponding C atoms. Final refinement of 172 parameters for the structure of (*Z*)-**2** over all F$^2$ gave $wR_2$ = 0.1321 and S = 1.002 (R = 0.0463 for 1645 F > 4σ). Final refinement of 225 parameters for the structure of **3** over all F$^2$ gave $wR_2$ = 0.1349 and S = 1.051 (R = 0.0443 for 1693 F > 4σ).

Coordinates and temperature factors for the atoms were deposited in the Cambridge Crystallographic Database (registration numbers CCDC 271987 and CCDC 271988 for (*Z*)-**2** and **3**, respectively).

**Oximation of 1.** A stirred mixture of hydroxylamine hydrochloride (1.80 g, 25.9 mmol) and absolutee pyridine (4.0 mL) was treated with absolute alcohol (44 mL). The resulting solution was treated with **1** (6.00 g, 24.6 mmol), stirred, and refluxed for 5 h. **1** dissolved completely to give a green solution after 30 min from the start of refluxing. Then solvent was removed from the mixture by heating on a bath. Residual solvent was removed at 80°C/30 torr. The resinous solid was treated with a mixture of $CHCl_3$ (60 mL) and water (10 mL). The aqueous layer was removed. The organic layer was washed with additional water (10 mL) and evaporated. The resinous solid was dried at 80°C/30 torr and dissolved in ether (15 mL). The crystalline solid that formed on standing was filtered off, washed with ether, and dried to afford a product (3.31 g, 52%) that was a mixture of the *Z*- and *E*-oximes of **1** (*E*:*Z* = 1:0.7) according to PMR spectra.

The *Z*-oxime was isolated by treating the mixture with boiling $CHCl_3$ (2 × 10 mL). The insoluble solid was recrystallized from boiling alcohol to afford crystalline prisms of (*Z*)-**2**, mp 242-244°C (dec.).

PMR spectrum (200.13 MHz, DMSO-$d_6$, δ, ppm, J/Hz): 0.64 and 1.08 [both d, 3H each, J = 6.8, $(CH_3)_2C$], 2.75 (septet of doublets, 1H, J = 6.8, J = 2.4, H-1′), 5.44 (d, 1H, J = 2.4, H-2), 6.29 (d, 1H, J = 9.6, H-6), 6.98 (s, 1H, H-9), 7.81 (s, 1H, H-4), 7.99 (d, 1H, J = 9.6, H-5), 11.50 (s, 1H, NO<u>H</u>).

$^{13}$C NMR spectrum of the same solution (50.32 MHz, $\delta_C$, ppm): 14.8 and 19.3 [both q, $(\underline{C}H_3)_2C$], 28.5 (d, C-1′), 88.3 (d, C-2), 98.4 (d, C-9), 112.8 (d, C-6), 113.4 (s, C-3a), 119.4 (s, C-4a), 120.6 (d, C-4), 144.6 (d, C-5), 154.1 (s, C-9a), 157.2 (s, C-8a), 159.9 (s, C-3), 166.2 (s, C-7). Found, %: C 64.70, H 5.02, N 5.46. $C_{14}H_{13}NO_4$. Calc., %: C 64.86, H 5.05, N 5.40.

IR spectrum (ν, cm$^{-1}$): 3310, 2969 (N<u>O–H</u>), 1716 (C=O), 1653, 1621, 1569, 1481, 1465, 1396, 1362, 1299, 1199, 1143, 1112, 1056, 948, 925, 890, 836.

UV spectrum ($\lambda_{max}$, nm, log ε): 229 (4.15), 256 (4.43), 301 (3.83), 314 (3.86), 351 (4.28).

**3*E*-Hydroxyimino-2-(1-methylethyl)-7H-furo[3,2-*g*][1]benzopyran-7-one [(*E*)-2]** was isolated by fractional crystallization from $CHCl_3$ of the solid obtained after separation of the *Z*-oxime, mp 193-195°C (dec.).

PMR spectrum (200.13 MHz, DMSO-$d_6$, δ, ppm, J/Hz): 0.83 and 1.03 [both d, 3H each, J = 6.8, $(CH_3)_2C$], 2.11 (septet of doublets, 1H, J = 6.8, J = 3.4, H-1′), 5.16 (d, 1H, J = 3.4, H-2), 6.27 (d, 1H, J = 9.6, H-6), 6.96 (s, 1H, H-9), 8.03 (d, 1H, J = 9.6, H-5), 8.39 (s, 1H, H-4), 11.71 (s, 1H, NO<u>H</u>).

$^{13}$C NMR spectrum of the same solution (50.32 MHz, $\delta_C$, ppm): 15.1 and 17.8 [both q, $(\underline{C}H_3)_2C$], 32.7 (d, C-1′), 87.7 (d, C-2), 98.4 (d, C-9), 112.4 (d, C-6), 112.9 (s, C-3a), 117.1 (s, C-4a), 128.2 (d, C-4), 144.8 (d, C-5), 152.2 (s, C-9a), 157.4 (s, C-8a), 159.8 (s, C-3), 166.5 (s, C-7). Found, %: C 64.61, H 5.02, N 5.37. $C_{14}H_{13}NO_4$. Calc., %: C 64.86, H 5.05, N 5.40.

IR spectrum (ν, cm$^{-1}$): 3273, 2970, 2932 (N<u>O–H</u>), 1744, 1721, 1625, 1568, 1465, 1394, 1365, 1258, 1221, 1204, 1144, 1103, 973, 912, 824 (C=O).

UV spectrum ($\lambda_{max}$, nm, log ε): 229 (4.15), 256 (4.43), 301 (3.83), 314 (3.86), 351 (4.28).

(*Z*)-**2** and (*E*)-**2** were isolated analogously by oximation of **1** using another method [4].

**7-(1-Chloro-2-methylpropoxy)-2-oxo-2H-1-benzopyran-6-carbonitrile (3).** $PCl_5$ (574 mg, 2.76 mmol) was dissolved in refluxing dry benzene (11.3 mL). The solution was cooled to 20°C, treated in one portion with (*Z*)-**2** (714 mg, 2.76 mmol), stirred until the solution turned yellow (slightly exothermic reaction), boiled for 20 min, cooled to 10°C, stirred, and treated with icewater (11 mL) and ether (22 mL). The ether layer was separated. The aqueous layer was extracted with ether (2 × 5 mL). The combined organic layers were dried over $MgSO_4$. Solvent was removed. The solid was dried at 30°C/1 torr and treated with EtOH (4 mL) to afford **3** (547 mg, 71%), mp 150-151°C (hot aqueous 90% EtOH). High-resolution MS. Found, *m*/*z*: 277.05079. $C_{14}H_{12}ClNO_3$. Calc., *m*/*z*: 277.05056.

PMR spectrum (200.13 MHz, $CDCl_3$, δ, ppm, J/Hz): 1.17 and 1.19 [both d, 3H each, J = 6.8, $(CH_3)_2C$], 2.43 (septet of doublets, 1H, J = 6.8, J = 4.2, H-2′), 5.90 (d, 1H, J = 4.2, H-1′), 6.39 (d, 1H, J = 9.6, H-3), 7.21 (s, 1H, H-8), 7.65 (d, 1H, J = 9.6, H-4), 7.76 (s, 1H, H-5).

$^{13}C$ NMR spectrum of the same solution (125.77 MHz, $\delta_C$, ppm*): 16.97 and 17.01 [both q, $(\underline{C}H_3)_2C$], 36.00 (d, C-2′), 95.86 (d, C-1′), 100.07 (s, C-6)[a], 103.32 (d, C-8), 114.03 (s, C-4a)[a], 114.08 (s, CN), 115.84 (d, C-3), 133.36 (d, C-5), 141.64 (d, C-4), 157.60 (s, C-8a)[b], 158.79 (s, C-7)[b], 158.97 (s, C-2)[b].

IR spectrum (ν, $cm^{-1}$): 3058, 2977, 2883, 2232 (C≡N), 1739 (C=O), 1625, 1495, 1386, 1334, 1285, 1204, 1164, 1128, 1031, 919, 831, 737.

UV spectrum (EtOH, $\lambda_{max}$, nm, log ε): 210 (4.37), 237 (4.35), 294 (3.93), 320 (3.99).

Compound **3** was prepared analogously from (*E*)-**2** in 70% yield.

**7-Hydroxy-2-oxo-2H-1-benzopyran-6-carbonitrile (4).** A solution of **3** (115 mg) in aqueous alcohol (1.0 mL, 90% EtOH by vol) was refluxed for 0.5 h. The needlelike crystals that formed on cooling were separated, washed with $CHCl_3$, and dried to afford **4** (71 mg, 92%), mp 268-270°C (dec.).

High-resolution MS. Found, *m*/*z*: 187.02706. $C_{10}H_5NO_3$. Calc., *m*/*z*: 187.02694.

PMR spectrum (400.13 MHz, DMSO-$d_6$, δ, ppm, J/Hz): 6.29 (d, 1H, J = 9.6, H-3), 6.80 (s, 1H, H-8), 7.86 (d, 1H, J = 9.6, H-4), 8.00 (s, 1H, H-5).

$^{13}C$ NMR spectrum of the same solution (100.61 MHz, $\delta_C$, ppm*): 96.8 (s, C-6), 102.9 (d, C-8), 112.0 (s, C-4a), 113.4 (d, C-3), 115.8 (s, CN), 134.3 (d, C-5), 143.2 (d, C-4), 157.6 (s, C-8a)[a], 159.3 (s, C-7)[a], 162.6 (s, C-2). *Assignments denoted with the same superscript should possibly be switched.

IR spectrum (ν, $cm^{-1}$): 3487 and 3434 (OH), 3088, 3057, 2761, 2669, 2242 (C≡N), 1726 (C=O), 1707, 1627, 1515, 1446, 1405, 1329, 1295, 1272, 1142, 1100, 910, 856, 835, 672, 573, 522, 456.

UV spectrum (EtOH, $\lambda_{max}$, nm, log ε): 210 (4.37), 237 (4.35), 294 (3.93), 320 (3.99). Isobutyraldehyde was identified using the PMR spectrum in the mother liquor from **4**.

## ACKNOWLEDGMENT

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