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Crystal structure of 1-(2-hydroxyphenyl)-3-(4-nitrophenyl)-1,3-propanedioxime, $C_{15}H_{13}N_3O_5$

N. Galitsky[I], V. Cody[I] and J.-C. Wallet[II]

[I] Hauptman-Woodward Medical Research Institute, 73, High Street, Buffalo, NY 14203-1196, USA
[II] Université de Saint-Jerome, Faculte des Sciences et Techniques, Laboratoire de Phytochimie, Case 531, 13397 Marseille Cedex 20, France

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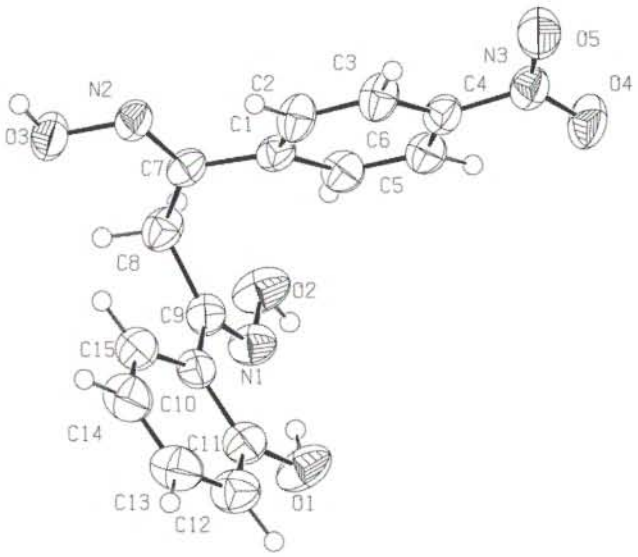

Abstract

$C_{15}H_{13}N_3O_5$, triclinic, $P\bar{1}$ (No. 2), a = 6.979(1) Å, b = 8.309(1) Å, c = 13.128(1) Å, α = 105.566(9)°, β = 105.287(8)°, γ = 93.15(1)°, V = 700.9 Å^3, Z = 2, $R_{gt}(F)$ = 0.043, $wR_{ref}(F)$ = 0.055, T = 292 K.

Source of material

The compound was synthezised by boiling at reflux the 4′-nitroflavone (2.67 g, 10^{-2} mol) in pyridin (50 ml) with hydroxylamine chlorhydrate (1.40 g, $2 \cdot 10^{-2}$ mol) for 4 hours. The mixture was cooled at room temperature and poured into iced water. The precipitate was filtered and dried. It was recrystallized in ethanol giving two compounds: yellow crystals (the title compound, mp = 483 K), and another yellow crystals which are now under investigation.

Discussion

The molecules are not planar. The geometry of the molecule is determined by the sp^3 carbon C8. The two phenyl rings are not in the C7C8C9 plane. An intra molecular hydrogen bond is formed between the phenolic hydroxyl O1 and the neighbouring oxime group. Each six-membered ring is involved in a ten membered ring formed with a neighbouring molecule via intermolecular hydrogen bonds. Molecules are also bound by classical six-atoms rings involving O—H···N hydrogen bridges [1] and form polymeric hydrogen bonded chains.

Table 1. Data collection and handling.

Crystal:	yellow, prismatic, size 0.15 × 0.30 × 0.50 mm
Wavelength:	Cu K_α radiation (1.54178 Å)
μ:	9.71 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, ω/2θ
$2\theta_{max}$:	119.88°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2052, 2052
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 3\ \sigma(I_{obs})$, 1980
$N(param)_{refined}$:	260
Program:	teXsan [2]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(2)	2*i*	1.191(3)	0.705(3)	0.899(2)	0.064(6)
H(3)	2*i*	0.985(3)	0.909(3)	0.909(2)	0.061(6)
H(5)	2*i*	0.496(3)	0.555(3)	0.805(2)	0.063(6)
H(6)	2*i*	0.712(3)	0.344(2)	0.804(2)	0.051(6)
H(81)	2*i*	0.954(3)	0.143(3)	0.828(2)	0.063(6)
H(82)	2*i*	1.154(3)	0.130(3)	0.789(2)	0.053(6)
H(15)	2*i*	1.284(3)	0.270(3)	0.690(2)	0.061(6)
H(14)	2*i*	1.434(4)	0.372(3)	0.576(2)	0.096(9)
H(12)	2*i*	0.873(4)	0.270(3)	0.342(2)	0.087(8)
H(1′)	2*i*	0.660(4)	0.112(4)	0.507(2)	0.092(9)
H(2′)	2*i*	0.544(5)	−0.018(4)	0.666(3)	0.11(1)
H(3′)	2*i*	1.525(5)	0.345(5)	0.960(3)	0.14(1)
H(13)	2*i*	1.225(4)	0.368(3)	0.398(2)	0.097(9)

* Correspondence author
(e-mail: jean-claude.wallet@iut-chimie.u-3mrs.fr)

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	2*i*	0.6819(2)	0.1546(2)	0.4495(1)	0.0406(8)	0.086(1)	0.0518(9)	–0.0164(7)	–0.0025(7)	0.0217(8)
O(2)	2*i*	0.6571(2)	0.0347(2)	0.7079(1)	0.0507(9)	0.072(1)	0.0508(9)	–0.0214(8)	0.0047(7)	0.0179(8)
O(3)	2*i*	1.4133(2)	0.2996(2)	0.9170(1)	0.0391(8)	0.0523(9)	0.0611(9)	0.0121(7)	0.0028(7)	0.0147(7)
O(4)	2*i*	0.4202(2)	0.8473(2)	0.8229(1)	0.0311(8)	0.068(1)	0.077(1)	0.0066(7)	0.0042(7)	0.0192(8)
O(5)	2*i*	0.6772(2)	1.0220(2)	0.9324(1)	0.0510(9)	0.0501(9)	0.074(1)	0.0087(7)	0.0030(8)	0.0019(8)
N(1)	2*i*	0.7326(2)	0.0832(2)	0.6303(1)	0.0437(9)	0.047(1)	0.045(1)	–0.0087(7)	0.0075(7)	0.0074(8)
N(2)	2*i*	1.2882(2)	0.4257(2)	0.9169(1)	0.0346(8)	0.0462(9)	0.0436(9)	0.0052(7)	0.0049(7)	0.0108(7)
N(3)	2*i*	0.5988(2)	0.8824(2)	0.8721(1)	0.0367(9)	0.053(1)	0.049(1)	0.0060(8)	0.0086(7)	0.0161(8)
C(1)	2*i*	0.9740(2)	0.5001(2)	0.8492(1)	0.0316(9)	0.042(1)	0.0339(9)	–0.0016(8)	0.0045(7)	0.0084(8)
C(2)	2*i*	1.0521(3)	0.6696(2)	0.8784(2)	0.0267(9)	0.045(1)	0.059(1)	–0.0028(8)	0.0047(8)	0.0175(9)
C(3)	2*i*	0.9312(3)	0.7938(3)	0.8853(2)	0.034(1)	0.040(1)	0.060(1)	–0.0017(8)	0.0049(9)	0.0168(9)
C(4)	2*i*	0.7268(3)	0.7483(2)	0.8587(1)	0.034(1)	0.047(1)	0.040(1)	0.0046(8)	0.0072(8)	0.0141(8)
C(5)	2*i*	0.6423(3)	0.5831(2)	0.8238(2)	0.0268(9)	0.050(1)	0.045(1)	–0.0046(8)	0.0053(8)	0.0058(9)
C(6)	2*i*	0.7658(3)	0.4587(2)	0.8201(2)	0.034(1)	0.042(1)	0.047(1)	–0.0061(8)	0.0078(8)	0.0060(9)
C(7)	2*i*	1.1096(2)	0.3701(2)	0.8517(1)	0.0319(9)	0.042(1)	0.036(1)	–0.0027(7)	0.0035(7)	0.0120(8)
C(8)	2*i*	1.0367(3)	0.1882(2)	0.7879(2)	0.042(1)	0.040(1)	0.050(1)	0.0007(9)	0.0016(9)	0.0127(9)
C(9)	2*i*	0.9136(3)	0.1588(2)	0.6695(2)	0.037(1)	0.033(1)	0.048(1)	–0.0006(8)	0.0057(8)	0.0042(8)
C(10)	2*i*	0.9988(3)	0.2156(2)	0.5914(2)	0.036(1)	0.034(1)	0.044(1)	0.0005(8)	0.0076(8)	0.0007(8)
C(11)	2*i*	0.8818(3)	0.2123(2)	0.4856(2)	0.039(1)	0.043(1)	0.048(1)	–0.0037(8)	0.0080(9)	0.0042(9)
C(12)	2*i*	0.9648(3)	0.2700(3)	0.4151(2)	0.052(1)	0.060(1)	0.052(1)	–0.002(1)	0.013(1)	0.011(1)
C(13)	2*i*	1.1672(3)	0.3292(3)	0.4480(2)	0.057(1)	0.064(1)	0.058(1)	–0.006(1)	0.023(1)	0.006(1)
C(14)	2*i*	1.2858(3)	0.3312(3)	0.5495(2)	0.041(1)	0.068(2)	0.066(2)	–0.006(1)	0.020(1)	0.002(1)
C(15)	2*i*	1.2033(3)	0.2746(3)	0.6203(2)	0.036(1)	0.054(1)	0.052(1)	–0.0007(9)	0.008(1)	0.002(1)

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