

symmetry operations relating the ribbons. This vacant space is just enough for the hexamethyleneimine ring, but too wide for the pyrrolidine or piperidine ring. Thus, the ribbons for the five- or six-membered ring, if any, should be formed by a different mode of O—H...O hydrogen bonding.

The crystal of PYH is isostructural with that of PIH (Fukuyama *et al.*, 1973). The benzoate ions related by an *n*-glide plane perpendicular to **b** are held together by an intermolecular O—H...O hydrogen bond to form a ribbon along [101]. The ribbons related by a *c*-glide plane are held together by the cations through two kinds of N—H...O hydrogen bonds, resulting in a three-dimensional network.

A common feature of the four crystals is the formation of the ribbons of benzoate ions *via* O—H...O hydrogen bonds. However, for PYH and PIH the benzoate ions in the ribbons are related by the glide plane in place of the twofold screw axis. The repetition period of the ribbons in PYH and PIH is the length of the [101] axis. The period is 11.30 (2) Å for PYH and 11.59 (1) Å for PIH. The values are almost equal irrespective of the difference in the size of pyrrolidine and piperidine rings, and smaller by a factor of about 0.78 than the values of *b* for the dimorphic forms of HMI. Thus, the glide operation results in ribbons with smaller period and larger cross section compared to the 2<sub>1</sub> operation. The vacant space among the ribbons thus formed is suitable for the packing of the smaller size ring such as the piperidine or pyrrolidine ring.

The dihedral angle between the planes of the benzene ring and the carboxylate group is 23.7 (3)° in PYH and 16.6 (2)° in PIH, and 4.9 (1) and 6.0 (1)° in the orthorhombic and monoclinic forms of HMI, respectively, reflecting the difference in the mode of O—H...O hydrogen bonding. However, the O...O distances in the four crystals remain in a smaller range of values, 2.60

to 2.63 Å, than the N...O distances, 2.67 to 2.83 Å. For all these crystals the C(7)—O(9) bond is shorter than C(7)—O(8), probably because O(8) accepts N—H and O—H hydrogen bonds, while O(9) accepts only one N—H hydrogen bond.

The authors thank the Crystallographic Research Center, Institute for Protein Research, Osaka University, for the use of the facility.

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*Acta Cryst.* (1987). **C43**, 158–160

## Structure of 2',4'-Dihydroxychalcone

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(Received 1 May 1986; accepted 14 August 1986)

**Abstract.** 1-(2,4-Dihydroxyphenyl)-3-phenyl-2-propen-1-one,  $C_{15}H_{12}O_3$ ,  $M_r = 240.3$ , orthorhombic, *Pbca*,  $a = 7.3653$  (6),  $b = 23.667$  (2),  $c = 14.054$  (2) Å,  $V = 2449.8$  (8) Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 1.303$  g cm<sup>-3</sup>,  $Mo K\alpha$ ,  $\lambda = 0.71073$  Å,  $\mu = 0.85$  cm<sup>-1</sup>,  $F(000) = 1008$ ,  $T =$

297 K,  $R = 0.035$  for 1385 observations (of 2153 unique data). The compound was isolated from Florida false rosemary (*Ceratiola ericoides*; Empetraceae). The carbonyl function is oriented *s-cis* to the *trans* C=C double bond and *syn* to a phenolic OH group, forming

0108-2701/87/010158-03\$01.50

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an intramolecular hydrogen bond of length 2.575 (1) Å. The carbonyl O atom also accepts an intermolecular hydrogen bond from phenolic OH of length 2.759 (1) Å, forming chains along the *c* direction.

**Introduction.** *Ceratiola ericoides* (false rosemary) of the family Empetraceae is an endemic, dominant shrub of the Florida scrub. Preliminary investigations had shown that *C. ericoides* releases allelopathic constituents which inhibit the germination and growth of plants of the Florida sandhill community (Richardson, 1985). Therefore, a chemical analysis of *C. ericoides* was performed which led to isolation of a number of flavonoids. The title compound (Adityachaudhury, Kirtaniya & Mukherjee, 1971) exhibits mild germination inhibition on *Lactuca sativa* (lettuce). Its structure is described here.

**Experimental.** Yellow rectangular prisms, dimensions 0.32 × 0.36 × 0.52 mm. Space-group determination by systematic absences *Ok*l with *k* odd, *h*0l with *l* odd, *hk*0 with *h* odd. Enraf-Nonius CAD-4 diffractometer with graphite monochromator, cell dimensions from setting angles of 25 reflections having 13 < θ < 14°. Data collection by ω-2θ scans designed for *I* = 50σ(*I*), max. scan time 120 s, scan rates varied 0.45–4.0° min<sup>-1</sup>. Reflections having 1 < θ < 25°, 0 ≤ *h* ≤ 8, 0 ≤ *k* ≤ 28, 0 ≤ *l* ≤ 16 measured, corrected for background, Lorentz, polarization, no absorption correction, 2153 unique data, no redundant data. Standard reflections 200, 040, 002, ±2.0% maximum random variation. Structure solved using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), refinement by full-matrix least squares based on *F* with weights  $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$  with 1385 data for which *I* > 3σ(*I*) (768 unobserved reflections), using Enraf-Nonius *SDP* (Frenz & Okaya, 1980). Atomic scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974) for C and O. Non-H atoms refined anisotropically, H atoms located from Δ*F* map (0.31–0.50 e Å<sup>-3</sup>), refined isotropically. Final *R* = 0.035 (0.078 all data), *wR* = 0.037, *S* = 1.858 for 212 variables. Largest Δ/σ = 0.02 on final cycle, max. residual density 0.13, min. -0.15 e Å<sup>-3</sup>, extinction coefficient 4.9 (3) × 10<sup>-7</sup>. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.\*

**Discussion.** The structure of the title compound is illustrated in Fig. 1. The carbonyl is *syn* to hydroxyl

\* Lists of H-atom coordinates, anisotropic thermal parameters, deviations from least-squares planes and structure factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43331 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

group O(17), forming an intramolecular hydrogen bond, and is also *s-cis* to the *trans* double bond. The deviations of the molecule from overall planarity can be described in terms of dihedral angles between three planar subunits, the trisubstituted phenyl ring *P*1, the monosubstituted phenyl ring *P*2, and the four carbon atoms comprising the central double-bond plane, *P*3. Average and maximum deviations from best planes for these three subunits are: *P*1 0.005 (3), 0.009 (2) Å; *P*2 0.004 (3), 0.009 (2) Å; *P*3 0.002 (<1), 0.002 (2) Å. The dihedral angles are *P*1/*P*2 29.2 (2), *P*1/*P*3 19.7 (2) and *P*2/*P*3 9.5 (2)°.

Bond distances and angles are listed in Table 2, and are normal. The C(2)=C(3) double-bond length is typical of those observed in stilbenes [Tirado-Rives, Oliver, Fronczek & Gandour (1984) and references cited therein] and agrees well with the distance of 1.328 (2) Å observed in a structurally similar chalcone with a similar conformation (Tomita, 1982).

The pattern of bond lengths within the trisubstituted phenyl ring exhibits a pronounced shortening of C(8)–C(9) versus expected values, as well as a lengthening of C(5)–C(10) and, to a lesser extent, C(9)–C(10). This same pattern has previously been

Table 1. Coordinates and equivalent isotropic thermal parameters

$$B_{eq} = \frac{1}{3}(a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33}).$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> <sub>eq</sub>
O(1)	0.6028 (2)	0.17641 (4)	-0.27092 (7)	4.67 (3)
C(2)	0.5175 (3)	0.06935 (7)	-0.19046 (11)	4.08 (4)
C(3)	0.4810 (3)	0.11859 (7)	-0.15072 (11)	3.91 (4)
C(4)	0.5456 (3)	0.17282 (6)	-0.18677 (11)	3.61 (4)
C(5)	0.6023 (2)	0.27585 (7)	-0.15984 (11)	3.55 (4)
C(6)	0.6014 (3)	0.32285 (6)	-0.10152 (11)	3.77 (4)
C(7)	0.5482 (2)	0.31780 (7)	-0.00799 (12)	3.61 (4)
C(8)	0.4912 (3)	0.26590 (7)	0.02776 (11)	3.75 (4)
C(9)	0.4895 (2)	0.22000 (6)	-0.03046 (11)	3.48 (4)
C(10)	0.5448 (2)	0.22256 (6)	-0.12584 (11)	3.24 (4)
C(11)	0.4560 (3)	0.01395 (6)	-0.15816 (11)	3.70 (4)
C(12)	0.3261 (3)	0.00782 (7)	-0.08725 (12)	4.48 (4)
C(13)	0.2689 (3)	-0.04557 (7)	-0.06010 (13)	5.30 (5)
C(14)	0.3385 (3)	-0.09279 (7)	-0.10363 (14)	5.47 (5)
C(15)	0.4683 (3)	-0.08769 (7)	-0.17247 (14)	5.34 (5)
C(16)	0.5261 (3)	-0.03432 (7)	-0.20026 (13)	4.83 (5)
O(17)	0.6642 (2)	0.28278 (4)	-0.24966 (9)	5.04 (3)
O(18)	0.5562 (2)	0.36499 (4)	0.04719 (8)	4.85 (3)

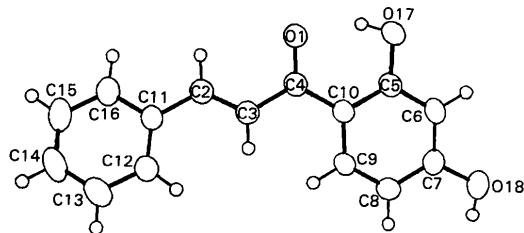


Fig. 1. Numbering scheme and thermal ellipsoids drawn at the 40% probability level. Hydrogen atoms have arbitrary radius.

observed in compounds containing the same substitution on a benzene ring. Liebich (1979) has commented on this pattern in 2,4-dihydroxybenzophenone (HHB) and Schlemper (1982) on an identical pattern in 2,2',4,4'-tetrahydroxybenzophenone (THB).

Table 2. Bond distances (Å) and angles (°)

O(1)—C(4)	1.258 (1)	C(6)—C(7)	1.377 (2)
O(17)—C(5)	1.352 (2)	C(7)—C(8)	1.392 (2)
O(17)—H(17)	0.879 (16)	C(8)—C(9)	1.360 (2)
O(18)—C(7)	1.361 (1)	C(9)—C(10)	1.402 (2)
O(18)—H(18)	0.892 (16)	C(11)—C(12)	1.389 (2)
C(2)—C(3)	1.320 (2)	C(11)—C(16)	1.386 (2)
C(2)—C(11)	1.459 (2)	C(12)—C(13)	1.386 (2)
C(3)—C(4)	1.460 (2)	C(13)—C(14)	1.373 (2)
C(4)—C(10)	1.456 (2)	C(14)—C(15)	1.366 (2)
C(5)—C(6)	1.382 (2)	C(15)—C(16)	1.389 (2)
C(5)—C(10)	1.414 (2)	C—H range:	0.932 (13)—0.973 (15)
C(3)—C(2)—C(11)	126.77 (15)	C(4)—C(10)—C(5)	121.4 2(12)
C(2)—C(3)—C(4)	124.29 (14)	C(4)—C(10)—C(9)	121.91 (12)
O(1)—C(4)—C(3)	119.64 (13)	C(5)—C(10)—C(9)	116.67 (12)
O(1)—C(4)—C(10)	119.98 (12)	C(2)—C(11)—C(12)	122.03 (13)
C(3)—C(4)—C(10)	120.37 (12)	C(2)—C(11)—C(16)	119.49 (12)
O(17)—C(5)—C(6)	117.23 (12)	C(12)—C(11)—C(16)	118.47 (14)
O(17)—C(5)—C(10)	121.67 (12)	C(11)—C(12)—C(13)	120.15 (16)
C(6)—C(5)—C(10)	121.08 (12)	C(12)—C(13)—C(14)	120.40 (18)
C(5)—C(6)—C(7)	119.86 (13)	C(13)—C(14)—C(15)	120.33 (16)
O(18)—C(7)—C(6)	117.42 (13)	C(14)—C(15)—C(16)	119.63 (17)
O(18)—C(7)—C(8)	122.11 (12)	C(11)—C(16)—C(15)	121.01 (18)
C(6)—C(7)—C(8)	120.47 (13)	C(5)—O(17)—H(17)	103.7 (11)
C(7)—C(8)—C(9)	119.38 (13)	C(7)—O(18)—H(18)	111.1 (12)
C(8)—C(9)—C(10)	122.53 (12)		

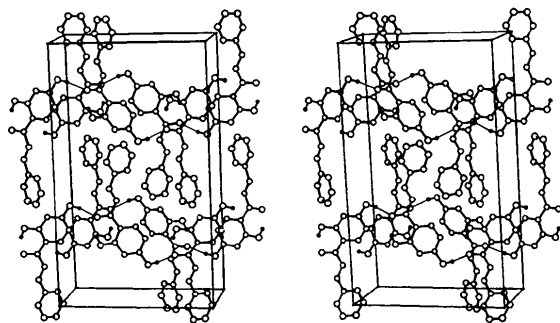


Fig. 2. Stereoscopic representation of the unit cell, viewed slightly oblique to the a axis, with c horizontal. H atoms other than in OH are not shown.

Both intramolecular and intermolecular hydrogen bonding are present. The intramolecular bond O(17)—H...O(1) has length 2.575 (1) Å, H...O distance 1.76 (2) Å, and angle at H 154 (2)°. The intramolecular bond length in similar compounds is 2.533 (2) Å in the Tomita (1982) chalcone, 2.550 (4) Å in HHB (Liebich, 1979), 2.502 (3) Å in  $\alpha$ -zearalenol (Watson, Zabel, Mirocha & Pathre, 1982), and ranges 2.516 (2)—2.716 (2) Å for four independent values in THB (Schlemper, 1982). The intermolecular hydrogen bond involves hydroxy group O(18) as donor and carbonyl O(1) at  $x, \frac{1}{2}-y, \frac{1}{2}+z$  as acceptor. Parameters are O...O 2.759 (1), H...O 1.90 (2) Å, and angle 162 (2)°. These hydrogen bonds thus form chains along the c direction, as shown in Fig. 2.

This material is based (in part) upon work supported by the US Department of Agriculture, Competitive Research Grants Program for Forest and Rangeland Renewable Resources under agreement No. 85-FSTY-9-0139.

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