C(31)	−0.1974 (3)	0.1037 (2)	0.1366 (2)	0.0387 (6)
N(1)	−0.3920 (2)	−0.2713 (1)	0.2785 (1)	0.0394 (5)
O(1)	−0.4579 (2)	−0.3821 (1)	0.3204 (1)	0.0580 (6)
C(1)	−0.2798 (3)	−0.1712 (2)	0.3666 (1)	0.0416 (5)
C(11)	−0.4169 (6)	−0.1776 (3)	0.4764 (2)	0.0671 (11)
C(12)	−0.0631 (4)	−0.2012 (2)	0.4020 (2)	0.0567 (8)
C(2)	−0.2585 (3)	−0.0394 (2)	0.3128 (2)	0.0396 (6)
C(3)	−0.1860 (3)	−0.0282 (1)	0.1848 (1)	0.0340 (5)
O(3)	0.0253 (2)	−0.0500 (1)	0.1736 (1)	0.0488 (5)
C(4)	−0.3407 (3)	−0.1279 (2)	0.1084 (2)	0.0380 (6)
C(5)	−0.3674 (3)	−0.2660 (2)	0.1466 (2)	0.0385 (6)
C(51)	−0.1821 (4)	−0.3263 (2)	0.1106 (2)	0.0547 (8)
C(52)	−0.5746 (4)	−0.3431 (2)	0.0873 (2)	0.0611 (8)

Table 2. *Bond lengths* (Å) *and angles* (°)

N(100)—O(100)	1.322 (2)	C(1)—C(11)	1.522 (3)
N(100)—C(200)	1.368 (2)	C(1)—C(12)	1.535 (3)
N(100)—C(600)	1.358 (2)	C(1)—C(2)	1.538 (3)
C(200)—C(300)	1.380 (3)	C(2)—C(3)	1.518 (3)
C(200)—C(31)	1.500 (2)	C(3)—O(3)	1.428 (2)
C(300)—C(400)	1.385 (3)	C(3)—C(4)	1.525 (2)
C(400)—C(500)	1.373 (3)	C(4)—C(5)	1.535 (3)
C(500)—C(600)	1.363 (3)	C(5)—C(51)	1.524 (3)
C(31)—C(3)	1.549 (3)	C(5)—C(52)	1.533 (3)
N(1)—O(1)	1.284 (2)	O(3)—O(100)	2.673 (2)
N(1)—C(1)	1.494 (2)	O(3)—H(3)	0.90 (2)
N(1)—C(5)	1.491 (3)	O(100)—H(3)	1.79 (3)
C(200)—N(100)—C(600)	120.7 (2)	C(11)—C(1)—C(2)	109.0 (2)
O(100)—N(100)—C(600)	118.7 (2)	C(11)—C(1)—C(12)	109.1 (2)
O(100)—N(100)—C(200)	120.6 (1)	C(1)—C(2)—C(3)	116.1 (2)
N(100)—C(200)—C(31)	119.3 (2)	C(31)—C(3)—C(2)	110.9 (1)
N(100)—C(200)—C(300)	117.9 (1)	C(2)—C(3)—C(4)	107.7 (1)
C(300)—C(200)—C(31)	122.7 (2)	C(2)—C(3)—O(3)	111.8 (1)
C(200)—C(300)—C(400)	121.7 (2)	C(31)—C(3)—C(4)	107.4 (1)
C(300)—C(400)—C(500)	118.5 (2)	C(31)—C(3)—O(3)	110.0 (1)
C(400)—C(500)—C(600)	119.7 (2)	O(3)—C(3)—C(4)	109.0 (1)
N(100)—C(600)—C(500)	121.3 (2)	C(3)—C(4)—C(5)	116.2 (2)
C(200)—C(31)—C(3)	118.1 (2)	N(1)—C(5)—C(4)	109.5 (2)
C(1)—N(1)—C(5)	124.6 (1)	C(4)—C(5)—C(52)	107.9 (2)
O(1)—N(1)—C(5)	116.4 (1)	C(4)—C(5)—C(51)	113.1 (2)
O(1)—N(1)—C(1)	115.6 (1)	N(1)—C(5)—C(52)	107.6 (2)
N(1)—C(1)—C(2)	109.6 (1)	N(1)—C(5)—C(51)	109.8 (2)
N(1)—C(1)—C(12)	108.6 (1)	C(51)—C(5)—C(52)	108.9 (2)
N(1)—C(1)—C(11)	107.5 (2)	C(3)—O(3)—H(3)	104 (2)
C(12)—C(1)—C(2)	112.9 (2)	O(3)—H(3)—O(100)	167 (3)

The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985). The first *E* map revealed positions of all non-H atoms. Least-squares refinement used *SHELX*76 (Sheldrick, 1976) and the *CRYSRULER* package (Rizzoli, Sangermano, Calestani & Andreetti, 1986), isotropic and then anisotropic displacement factors. H atoms were located on a difference Fourier map.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the IUCr (Reference: KA1059). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2′-Hydroxy-2-methoxychalcone

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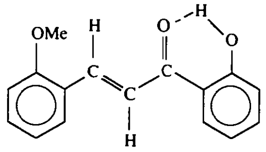
Abstract

In 1-(2-hydroxyphenyl)-3-(2-methoxyphenyl)-2-propen-1-one, $C_{16}H_{14}O_3$, the methoxylated phenyl ring and the benzoyl group are *trans* with respect to the ethylenic bond. The phenolic OH group forms an intramolecular hydrogen bond with the carbonyl O atom. The torsion angle O9—C9—C8—C7 of the enone moiety is −11.0 (2)°. The phenolic ring is almost coplanar with the carbonyl group: O9—C9—C1′—C2′ = −3.0 (2)°. The torsion angle of the methoxyphenyl group C5—C6—C7—C8 = −15.7 (2)°.

Comment

Natural and synthetic analogues of hydroxy and/or methoxychalcones have biological properties (Ischitsuka, Ninomiya, Ohsawa, Fujiu & Suhura, 1982; Ninomiya, Ohsawa, Aoyama, Umeda, Suhura & Ischitsuka, 1984; Ramanathan, Das & Tan, 1993; Batt, Goodman, Jones, Kerr, Mantegna, McAllister, Newton, Nurnberg, Welch & Covington, 1993). More recently it has been noted that chalcones frequently crystallize in non-centrosymmetric space groups, which is a favorable criterion for non-linear optical properties. In order to find relationships between hydrogen bonding and non-linear optical properties, a series of substituted chalcones have been synthesized.

2′-Hydroxychalcones are synthetic precursors for preparation of the corresponding flavonols (Wallet, Gaydou, Molins & Miravitlles, 1994). In the title chalcone (1) an intramolecular hydrogen bond, O9···H2′—O2′, contributes to the planarity of the benzoyl moiety.



(1)

The methoxyphenyl group is slightly more twisted with C5—C6—C7—C8 = −15.7 (2)°. The crystal packing is formed by discrete molecules separated by normal van der Waals interactions. The chalcone described here crystallizes in a centrosymmetric space group. Recent structures of chalcones solved for their optical properties are 4-bromochalcone (Zhengdong, Fen & Genbo, 1992), 4′-methoxychalcone (Zhengdong, Liangren & Genbo, 1992), thiophene chalcone (Zhengdong & Genbo, 1993) and 4-chlorochalcone (Zhengdong & Genbo, 1994). The labelling sequence and a view of the molecule are presented in Fig. 1.

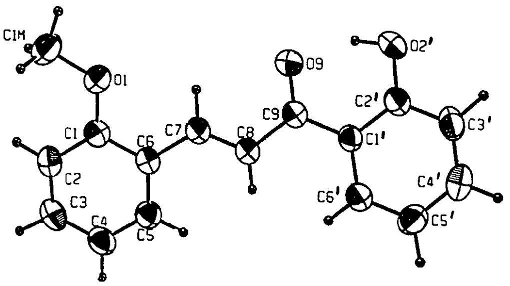


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule with the atom-labeling scheme. The displacement ellipsoids are drawn at 50% probability.

Experimental

Crystal data

$C_{16}H_{14}O_3$
$M_r = 254.29$
Monoclinic
$P2_1/n$
$a = 3.992$ (1) Å
$b = 24.356$ (2) Å
$c = 13.071$ (1) Å
$\beta = 90.55$ (1)°
$V = 1270.9$ (2) Å^3
$Z = 4$
$D_x = 1.328$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 1–25°$
$\mu = 0.08$ mm^{-1}
$T = 294$ K
Prismatic
0.68 × 0.26 × 0.13 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega–2\theta$ scans
Absorption correction: empirical
$T_{\min} = 0.9781$, $T_{\max} = 0.9999$
5025 measured reflections
4008 independent reflections

2014 observed reflections [$F_o > 3.0\sigma(F_o)$]
$R_{\rm int} = 0.020$
$\theta_{\max} = 30.45°$
$h = 0 \rightarrow 5$
$k = -34 \rightarrow 0$
$l = -18 \rightarrow 18$
3 standard reflections
frequency: 60 min
intensity variation: 0.4%

Refinement

Refinement on F
$R = 0.043$
$wR = 0.055$

$(\Delta/\sigma)_{\max} = 0.01$
$\Delta\rho_{\max} = 0.19$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}

$S = 1.80$
2014 reflections
228 parameters
Only H-atom U's refined
$w = 4F_o/\sigma(F_o)$

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{\rm eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	$B_{\rm eq}$
O1	0.7846 (3)	0.00206 (5)	0.61359 (8)	4.43 (3)
O2′	0.2986 (4)	0.26565 (5)	0.42282 (9)	5.50 (3)
O9	0.5629 (4)	0.17902 (5)	0.49489 (9)	5.35 (3)
C1	0.9127 (4)	0.01692 (6)	0.7066 (1)	3.16 (3)
C1*M*	0.7839 (5)	−0.05434 (7)	0.5862 (1)	4.82 (4)
C2	1.0446 (4)	−0.02037 (6)	0.7770 (1)	3.64 (3)
C3	1.1633 (4)	−0.00150 (7)	0.8698 (1)	4.08 (3)
C4	1.1515 (4)	0.05326 (7)	0.8941 (1)	4.06 (4)
C5	1.0243 (4)	0.09025 (6)	0.8245 (1)	3.56 (3)
C6	0.9015 (4)	0.07334 (6)	0.7294 (1)	2.91 (3)
C7	0.7670 (4)	0.11179 (6)	0.6539 (1)	3.30 (3)
C8	0.6740 (4)	0.16292 (6)	0.6685 (1)	3.39 (3)
C9	0.5409 (4)	0.19616 (6)	0.5837 (1)	3.40 (3)
C1′	0.3767 (4)	0.24953 (6)	0.6041 (1)	3.02 (3)
C2′	0.2610 (4)	0.28166 (6)	0.5213 (1)	3.60 (3)
C3′	0.1023 (4)	0.33128 (7)	0.5390 (1)	4.14 (4)
C4′	0.0576 (4)	0.34956 (6)	0.6369 (1)	4.21 (4)
C5′	0.1724 (5)	0.31915 (7)	0.7197 (1)	4.16 (4)
C6′	0.3298 (4)	0.26968 (6)	0.7028 (1)	3.54 (3)

Table 2. *Selected geometric parameters* (Å, °)

O1—C1	1.363 (2)	C6—C7	1.459 (2)
O1—C1*M*	1.419 (2)	C7—C8	1.314 (2)
O2′—C2′	1.354 (2)	C8—C9	1.468 (2)
O9—C9	1.238 (2)	C9—C1′	1.481 (2)
C1—C2	1.393 (2)	C1′—C2′	1.410 (2)
C1—C6	1.407 (2)	C1′—C6′	1.395 (2)
C2—C3	1.377 (2)	C2′—C3′	1.385 (2)
C3—C4	1.372 (2)	C3′—C4′	1.368 (2)
C4—C5	1.373 (2)	C4′—C5′	1.386 (2)
C5—C6	1.395 (2)	C5′—C6′	1.378 (2)
C1—O1—C1*M*	118.8 (1)	O9—C9—C1′	120.0 (1)
O1—C1—C2	123.5 (1)	C8—C9—C1′	120.4 (1)
O1—C1—C6	115.8 (1)	C9—C1′—C2′	119.4 (1)
C2—C1—C6	120.6 (1)	C9—C1′—C6′	122.6 (1)
C1—C2—C3	119.3 (1)	C2′—C1′—C6′	118.0 (1)
C2—C3—C4	121.0 (2)	O2′—C2′—C1′	122.1 (1)
C3—C4—C5	119.9 (1)	O2′—C2′—C3′	117.7 (1)
C4—C5—C6	121.4 (1)	C1′—C2′—C3′	120.2 (1)
C1—C6—C5	117.8 (1)	C2′—C3′—C4′	120.3 (2)
C1—C6—C7	119.7 (1)	C3′—C4′—C5′	120.8 (2)
C5—C6—C7	122.5 (1)	C4′—C5′—C6′	119.4 (2)
C6—C7—C8	127.8 (1)	C1′—C6′—C5′	121.4 (1)
C7—C8—C9	120.9 (1)	C2′—O2′—H2′	104. (1)
O9—C9—C8	119.6 (1)		

The structure was solved by direct methods with a straightforward run of *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Full-matrix refinement on F was carried out using the *MolEN* (Fair, 1990) program. Molecular graphics were drawn using *ORTEP* (Johnson, 1965).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1116). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Cyclohexanecarbohydroxamic Acid

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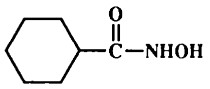
Abstract

The conformation of the hydroxamic acid moiety, O═C—N—O, of cyclohexanecarbohydroxamic acid ($C_7H_{13}NO_2$, *N*-hydroxycyclohexanecarboxamide) is synperiplanar with a torsion angle of 4.5 (2)°. The cyclohexyl ring adopts a chair conformation with the planar hydroxamic acid moiety in an equatorial position. The plane of the hydroxamic acid moiety is almost perpendicular to the cyclohexyl ring. The hydrogen bonds in the crystal form ladders of molecules which extend along the *c* axis.

Comment

Hydroxyurea is an inhibitor of the enzyme ribonucleotide reductase (RNR), as are some other hydroxamic acids (Larsen, Sjöberg & Thelander, 1982). RNR catalyses the conversion of ribonucleotides into the corresponding deoxyribonucleotides. This is an essential step in DNA synthesis for all living cells, hence inhibitors of RNR are of interest as anticancer or antiviral drugs. The RNR enzyme of *E. coli* consists of two proteins, R1 and R2. Hydroxyurea and analogues act at the smaller R2 protein by reducing a tyrosyl free radical, which initiates the reduction process (Atkin, Thelander, Reichard & Lang, 1973; Stubbe, Ator & Krinitsky, 1983; Larsson & Sjöberg, 1986). It has been proposed that hydroxamic acids gain their activity from their ability to undergo one-electron oxidation, and that an almost planar conformation is advantageous due to steric demands at the active site (Larsen *et al.*, 1982; Lam, Fortier, Thomson & Sykes, 1990; Atta *et al.*, 1993). The X-ray structure of R2 shows that the tyrosyl radical is buried in the protein *ca* 10 Å from the nearest surface (Nordlund, Sjöberg & Eklund, 1990), and it is not yet known whether deactivators are able to penetrate to the tyrosyl radical or react *via* long-range electron transfer (Nordlund & Eklund, 1993). Cyclohexanecarbohydroxamic acid (CYCHA), with a bulky cyclohexyl substituent directly attached to the CO-NHOH group, has no inhibitory effect on R2 of *E. coli*, although its reducing effect is the same as that of, *e.g.* *n*-hexanohydroxamic acid, a weak deactivator of R2 (Larsen *et al.*, 1982). Hydroxamic acids may adopt either a synperiplanar (*sp*, O═C—N—O torsion angle *ca* 0°) or an antiperiplanar (*ap*, O═C—N—O torsion angle *ca* 180°) conformation, with the *sp* conformation favoured by hydroxamic acids with a bulky substituent attached to the carbonyl group (Larsen, 1988). The hydroxamic acid moiety in CYCHA is in an equatorial position and is almost planar. The O═C—N—O torsion angle is 4.5 (2)°, the average deviation from the least-squares plane through C1, C7, O7, N8 and O9 is 0.028 Å, with a maximum deviation of 0.041 (1) Å (N8). The molecule as a whole is far from being planar. The cyclohexyl ring is in a chair conformation (*cf.* torsion angles in Table 2), and the plane of the hydroxamic acid moiety is almost perpendicular to the plane through the cyclohexyl ring atoms constituting the seat of the chair, the angle between the planes being 82.6 (1)°. Each molecule in the crystal is involved in four hydrogen bonds to three neighbouring molecules. The cyclohexyl rings are stacked in columns in the *c*



CYCHA

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