Data collection

Rigaku AFC-5 diffractometer	$R_{int} = 0.024$
	$\theta_{max} = 27.51°$
ω scans	$h = -12 \rightarrow 12$
Absorption correction: none	$k = -12 \rightarrow 2$
3704 measured reflections	$l = -11 \rightarrow 11$
1821 independent reflections	3 standard reflections
1571 reflections with	every 200 reflections
$I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.036$	$\Delta\rho_{max} = 0.302$ e Å^{-3}
$wR(F^2) = 0.100$	$\Delta\rho_{min} = -0.206$ e Å^{-3}
$S = 1.113$	Extinction correction: none
1821 reflections	Scattering factors from
128 parameters	*International Tables for*
All H atoms refined	*Crystallography* (Vol. C)
$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 0.1496P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. *Hydrogen-bonding geometry (Å, °)*

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$S^i	0.83 (3)	2.69 (2)	3.481 (2)	162 (1)
N2—H22$\cdots$S^{ii}	0.85 (3)	2.50 (2)	3.337 (2)	169 (1)
N2—H21$\cdots$C5^{iii}	0.83 (3)	2.74 (3)	3.318 (3)	128 (2)

Symmetry codes: (i) $x, \frac{1}{2}-y, z-\frac{1}{2}$; (ii) $x, \frac{1}{2}-y, \frac{1}{2}+z$; (iii) $x, -\frac{1}{2}-y, \frac{1}{2}+z$.

Program(s) used to solve structure: *SHELXS*96 (Sheldrick, 1996a). Program(s) used to refine structure: *SHELXL*96 (Sheldrick, 1996b). Molecular graphics: *PLUTON* (Spek, 1990). Software used to prepare material for publication: *SHELXL*96.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1278). Services for accessing these data are described at the back of the journal.

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2',4'-Dihydroxy-2-[3-(4-methylphenyl)-isoxazol-5-yl]acetophenone†

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Abstract

The synthesis and structure of the title compound, $C_{18}H_{15}NO_4$, are reported. The dihydroxyphenyl and methylphenyl groups are inclined at angles of 84.98 (4) and 9.18 (10)°, respectively, to the plane of the isoxazole ring. Both intra- and intermolecular hydrogen-bonding interactions are identified.

Comment

Due to the known various biological activities of isoxazoles (an important example being antispasmodic; Naruto *et al.*, 1982, 1983), we have synthesized a series of isoxazoles for further testing. Recently, we reported (Kumar *et al.*, 1996) the synthesis and X-ray structure of [3-(4-methylphenyl)-5-isoxazolyl]acetonitrile, (I); in the process of its derivatization (for structure–activity studies), we condensed it with resorcinol under Hoesch reaction conditions to produce the corresponding β-resacetophenone derivative (II). Such compounds have not been reported previously. This X-ray investigation was undertaken in order to unambiguously assign its constitution.

(I)

(II)
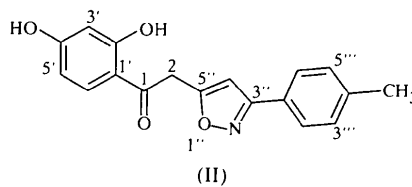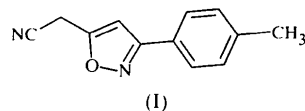
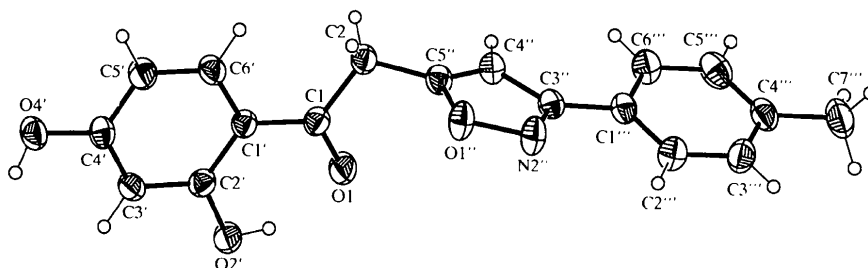† IUPAC name: 2,4-dihydroxyphenyl 3-(4-methylphenyl)isoxazol-5-ylmethyl ketone.

Fig. 1. View of the title molecule showing the atomic numbering. Displacement ellipsoids are drawn at the 50% probability level.

The structure of the title compound is illustrated in Fig. 1. The bond lengths and angles are unexceptional (Allen *et al.*, 1987). There is intramolecular hydrogen bonding between atoms O1 and O2′ [O1···O2′ 2.5493 (14) Å], and this interaction is presumably responsible for the approximately planar alignment of the O1—C1—C2 unit with respect to the plane of the dihydroxyphenyl group [O1—C1—C1′—C6′ −178.22 (14)°]. The isoxazole ring is essentially flat, with an r.m.s. deviation of 0.0017 Å. The dihydroxyphenyl and methylphenyl groups are inclined at angles of 84.98 (4) and 9.18 (10)°, respectively, to the mean plane through the isoxazole group. Structural stability is enhanced by intermolecular hydrogen bonding between the O4′ and N2″(−x, y + ½, −z + ½) atoms [N···O 2.8124 (16) Å].

Experimental

Crystal data

C₁₈H₁₅NO₄
M_r = 309.31
Monoclinic
$P2_1/c$
a = 7.9188 (14) Å
b = 15.010 (3) Å
c = 13.422 (2) Å
β = 101.064 (3)°
V = 1565.7 (5) Å³
Z = 4
D_x = 1.312 Mg m⁻³
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 4695 reflections
θ = 2.06–28.53°
μ = 0.093 mm⁻¹
T = 180 (2) K
Block
0.50 × 0.24 × 0.20 mm
Colourless

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
9463 measured reflections
3667 independent reflections
2462 reflections with
$I > 2\sigma(I)$

R_{int} = 0.028
θ_{max} = 28.53°
h = −9 → 10
k = −19 → 14
l = −17 → 17
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.111
S = 0.990
3667 reflections
213 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0581P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.211 e Å⁻³
$\Delta\rho_{min}$ = −0.202 e Å⁻³
Extinction correction: none
Scattering factors from
International Tables for Crystallography (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1″—C5″	1.3530 (16)	N2″—C3″	1.3160 (17)
O1″—N2″	1.4064 (15)		
N2″—C3″—C4″	110.52 (12)	C5″—C4″—C3″	105.35 (12)
N2″—C3″—C1‴	119.85 (12)	C4″—C5″—O1″	109.71 (12)
O1—C1—C1′—C6′	−178.22 (14)	C4″—C3″—C1‴—C6‴	8.4 (2)
C2—C1—C1′—C2′	−177.51 (13)	N2″—C3″—C1‴—C2‴	9.4 (2)

The temperature of the crystal was controlled using an Oxford Cryosystems Cryostream Cooler (Cosier & Glazer, 1986). Data were collected over a hemisphere of reciprocal space, by a combination of three sets of exposures. Each set had

a different φ angle for the crystal and each exposure of 10 s covered 0.3° in ω. The crystal-to-detector distance was 5.01 cm. Coverage of the unique set was over 91% complete to at least 28° in θ. Crystal decay was monitored by repeating the initial frames at the end of the data collection and analysing the duplicate reflections, and was found to be negligible. H atoms were added at calculated positions and refined using a riding model. Anisotropic displacement parameters were used for all non-H atoms; for H atoms, $U(H) = 1.2U_{eq}(C$ or O) or $1.5U_{eq}(C)$ for methyl groups.

Data collection: *SMART* (Siemens, 1994*a*). Cell refinement: *SAINT* (Siemens, 1995). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL/PC* (Siemens, 1994*b*). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXTL/PC*.

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9,10-Dihydro-1,4-dimethoxy-5,8-dimethyl-*cis*-9,10-diphenyl-9,10-anthracenediol, a Crowded Planar 9,10-Dihydroanthracene

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Abstract

The crystal structure determination of the title compound, $C_{30}H_{28}O_4$, a key intermediate in the preparation of highly functionalized anthracenes, establishes the geometry at the central 9,10-positions to be *cis*. Owing to the buttressing effect of the methoxy and methyl groups in the *peri*-1,4,5,8-positions on the central 9,10-substituents, the 9,10-dihydroanthracene framework adopts a planar arrangement and the *cis*-9,10-phenyl groups assume a perpendicular conformation with regard to this plane.

Comment

The title compound, (1), and the corresponding *trans* isomer, which exhibit different reactivities, are key intermediates in the preparation of specifically functionalized anthracenes (Wartini, 1997). Therefore, an unambiguous elucidation of their geometry was required.

(1)
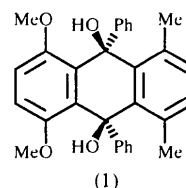
The crystal structure determination establishes a *cis* geometry for (1), and reveals that the tricyclic skeleton is essentially planar (Fig. 1). This is substantiated by the interplanar angle of 172.7 (5)° between the C1–C4, C9a and C5–C8, C8a, C10a arene planes, and by the relevant C10—C4a—C9a—C9 and C9a—C4a—C10—C10a torsion angles of 0.7 (2) and −1.9 (2)°, respectively. In the constrained almost-planar 'twisted pseudo-boat'-shaped 1,4-cyclohexadiene ring, the bridging C9 and C10 atoms deviate from the C4a, C10a, C8a, C9a plane by only 0.025 (1) and 0.016 (1) Å, respectively. The angles at the central ring C9 and C10 atoms are distorted from a tetrahedral arrangement, *e.g.* at C9, C8a—