definite. Therefore we concluded that the structure determination in *Pna*2$_1$ appears to be more sensible.

The final atomic parameters are given in Table 1 and interatomic distances and interbond angles with e.s.d.'s are listed in Table 2, along with torsion angles.* A perspective view of the molecule with the atom-numbering scheme is presented in Fig. 1.

Related literature. The title compound belongs to a series of substituted hydrazones synthesized and tested from a pharmacological point of view by the late Professor Silvano Rossi (Rossi, 1985). The hydrazone portion of the molecule, *i.e.* from C(1) to C(8) through N(1), N(2) and C(7), is of special interest. The geometry here as it pertains to the hydrazone–azo question has been discussed in detail by Pendergrass, Paul & Curtin (1972); they list literature values of bond lengths expected for the two tautomers: N—N 1.33–1.38, N=N 1.23–1.28; N—C(amide) 1.30–1.41; N=C 1.27–1.29 Å. The important point is that our distances fit into the pattern for the azo tautomer. Our N—C distance is on the low end of the range quoted and our N=N is slightly larger than observed in some related compounds, suggesting perhaps that there might be some small contribution present from the hydrazone tautomer.

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* Lists of structure factors, anisotropic thermal parameters, least-squares-planes data, H-atom coordinates and bond distances involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55676 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1003]

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Structure of a 2′-Oxo-8.1′-neolignan

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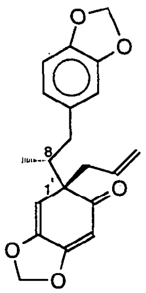
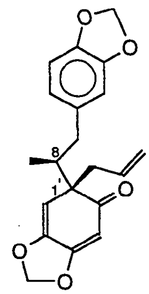
Abstract. Δ^8-1′,2′-Dihydro-3,4,3′,4′-bismethylenedioxy-2′-oxo-8.1-neolignan [6-allyl-6-(1,3-benzodioxol-5-ylpropylene)-1,3-benzodioxol-5(6*H*)-one], $C_{20}H_{20}O_5$, $M_r = 340.38$, orthorhombic, $P2_12_12_1$, $a = 6.628\ (4)$, $b = 12.604\ (6)$, $c = 20.774\ (11)$ Å, $V = 1735\ (3)$ Å^3, $Z = 4$, $D_x = 1.308$ g cm^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 0.876$, $F(000) = 720$, $T = 295$ K, $R = 0.032$, $wR = 0.041$ for 972 unique reflections. The analysis confirmed the structure of this new 2′-oxo-8.1′-neolignan, originally proposed on the basis of NMR data, and established the relative stereochemistry. No hydrogen bonding was observed; the closest intermolecular contact was between O2 and O4 (O atoms in the 1-position on each 1,3-benzodioxole) at 2.99 Å. The molecule has a bent conformation with a dihedral angle of 51.2 (1)° between the two planes defined by the aromatic rings.

Experimental. The title compound (1) and a diastereomer (2) were isolated from separate fractions of an extract of *Piper capense* roots (Green & Wiemer, 1991). A single crystal of the title compound was obtained from methanol by slow evaporation. The crystal, with approximate dimensions 0.6 × 0.5 × 0.4 mm, was mounted on an Enraf–Nonius



(1)



(2)

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Table 1. *Positional parameters and equivalent isotropic thermal parameters* (Å^2)

$$B_{\rm eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	$B_{\rm eq}$
O1	−0.2492 (4)	−0.1883 (2)	0.7045 (1)	5.33 (6)
O2	0.0116 (4)	−0.2209 (2)	0.7746 (1)	4.85 (6)
O3	0.6881 (4)	−0.0018 (2)	0.4921 (1)	5.14 (6)
O4	0.4103 (5)	0.3118 (2)	0.4058 (1)	5.20 (6)
O5	0.1180 (4)	0.2307 (2)	0.3737 (1)	5.02 (6)
C1	0.1507 (5)	−0.0614 (3)	0.6076 (2)	3.35 (7)
C1′	0.3750 (5)	−0.0158 (2)	0.4360 (1)	3.20 (7)
C2	−0.0435 (6)	−0.0941 (3)	0.6223 (2)	3.62 (7)
C2′	0.5600 (5)	0.0463 (3)	0.4620 (1)	3.46 (7)
C3	−0.0724 (5)	−0.1469 (3)	0.6796 (2)	3.52 (7)
C3′	0.5781 (6)	0.1595 (3)	0.4493 (2)	4.06 (8)
C4′	0.4252 (6)	0.2076 (2)	0.4200 (2)	3.46 (7)
C4	0.0822 (6)	−0.1667 (3)	0.7215 (1)	3.65 (7)
C5′	0.2445 (5)	0.1553 (3)	0.3998 (1)	3.44 (7)
C5	0.2737 (6)	−0.1351 (3)	0.7090 (2)	4.74 (9)
C6′	0.2149 (6)	0.0517 (2)	0.4059 (2)	3.68 (7)
C6	0.3049 (6)	−0.0812 (3)	0.6514 (2)	4.62 (8)
C7′	0.4631 (6)	−0.0935 (3)	0.3843 (2)	4.21 (8)
C7	0.1959 (6)	−0.0069 (3)	0.5443 (2)	3.76 (8)
C8	0.2873 (6)	−0.0812 (2)	0.4933 (2)	3.58 (7)
C8′	0.5794 (8)	−0.0404 (3)	0.3311 (2)	5.3 (1)
C9	0.1330 (7)	−0.1648 (3)	0.4731 (2)	4.98 (9)
C9′	0.7617 (7)	−0.0645 (3)	0.3126 (2)	6.2 (1)
C10	−0.1948 (7)	−0.2391 (4)	0.7635 (2)	5.42 (9)
C10′	0.2215 (7)	0.3299 (3)	0.3751 (2)	5.19 (9)

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

O1—C3	1.383 (4)	C1′—C7′	1.564 (5)
O1—C10	1.427 (5)	C1′—C8	1.557 (4)
O2—C4	1.377 (4)	C2—C3	1.374 (4)
O2—C10	1.408 (5)	C2′—C3′	1.452 (4)
O3—C2′	1.216 (4)	C3—C4	1.366 (5)
O4—C4′	1.345 (4)	C3′—C4′	1.328 (5)
O4—C10′	1.424 (5)	C4′—C5′	1.430 (5)
O5—C5′	1.376 (5)	C4—C5	1.356 (5)
O5—C10′	1.424 (5)	C5′—C6′	1.323 (5)
C1—C2	1.386 (5)	C5—C6	1.388 (5)
C1—C6	1.389 (5)	C7′—C8′	1.502 (5)
C1—C7	1.510 (5)	C7—C8	1.535 (5)
C1′—C2′	1.551 (5)	C8—C9	1.525 (5)
C1′—C6′	1.496 (5)	C8′—C9′	1.305 (6)
C3—O1—C10	105.8 (3)	O4—C4′—C3′	126.8 (3)
C4—O2—C10	106.3 (3)	O4—C4′—C5′	108.8 (3)
C4′—O4—C10′	108.5 (3)	C3′—C4′—C5′	124.4 (3)
C5′—O5—C10′	107.5 (3)	O2—C4—C3	110.0 (3)
C2—C1—C6	119.2 (3)	O2—C4—C5	128.0 (3)
C2—C1—C7	120.6 (3)	C3—C4—C5	122.0 (3)
C6—C1—C7	120.1 (3)	O5—C5′—C4′	108.1 (3)
C2′—C1′—C6′	114.9 (2)	O5—C5′—C6′	128.6 (4)
C2′—C1′—C7′	104.9 (3)	C4′—C5′—C6′	123.3 (4)
C2′—C1′—C8	107.3 (2)	C4—C5—C6	116.5 (3)
C6′—C1′—C7′	109.5 (2)	C1′—C6′—C5′	119.5 (3)
C6′—C1′—C8	110.6 (3)	C1—C6—C5	122.6 (3)
C7′—C1′—C8	109.4 (2)	C1′—C7′—C8′	114.7 (3)
C1—C2—C3	117.6 (3)	C1—C7—C8	113.6 (2)
O3—C2′—C1′	118.7 (3)	C1′—C8—C7	110.5 (2)
O3—C2′—C3′	121.5 (3)	C1′—C8—C9	113.9 (2)
C1′—C2′—C3′	119.7 (3)	C7—C8—C9	110.1 (3)
O1—C3—C2	128.5 (3)	C7′—C8′—C9′	126.0 (4)
O1—C3—C4	109.4 (3)	O1—C10—O2	108.3 (3)
C2—C3—C4	122.1 (3)	O4—C10′—O5	107.1 (3)
C2′—C3′—C4′	117.8 (3)		

CAD-4 diffractometer that used Mo $K\alpha$ radiation and a graphite monochromator. Unit-cell parameters were refined from the setting angles of 23 reflections having $8.4 < \theta < 24.0°$. 7194 reflections were measured over $1 < \theta < 20°$, and for index ranges $-8 \leq h \leq 8$, $-15 \leq k \leq 15$, $-25 \leq l \leq 25$, using 2θ–θ scans of width $(0.8 \pm 0.35\tan\theta)°$. The collection of data was halted at $\theta = 20°$ because of limited diffractometer time. Three standard reflections were measured every hour; a 0.2% intensity variation was observed, therefore a decay correction was not made. Lorentz and polarization corrections were applied. An absorption correction on F was made ranging from 0.97 to 1.0 using the empirical ψ-scan method. After averaging 7194 reflections, 972 unique reflections were obtained; average agreement of equivalent reflections was ±2% on F. The *SDP* package of programs provided by Enraf–Nonius (1979) was used for data reduction. The structure was solved using direct-methods computer programs *MULTAN* (Germain, Main & Woolfson, 1971) and *NQEST* (De Titta, Edmonds, Langs & Hauptman, 1975) and refined using the *SDP* package. All non-H atoms were found from the first E map. Although a few H atoms were found in a difference electron density map, all H atoms were placed in calculated theoretical positions (C—H bond distance 0.95 Å). Methyl H atoms were placed in a staggered conformation. The H-atom thermal parameters, equal to 1.3 times the value of $B_{\rm eq}$ of the attached heavy atom, were also fixed. A total of 883 reflections [$I > 3\sigma(I)$] were used in the full-matrix refinement on F with anisotropic thermal parameters for non-H atoms; total number of parameters was 226. (The ratio of parameters to data is lower than usual but the unique reflections were obtained after averaging eight equivalent reflections.) All atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final $R = 0.032$ and $wR = 0.041$. {Weight $w = 1/[\sigma F^2 + ({\rm PWT} \times F)^2 + {\rm QWT}]$ (Killean & Lawrence, 1969) where PWT = 0.02 and QWT = 0.00}. Maximum peak in the final difference electron density map = 0.173 e Å^{-3}; standard deviation of unit weight = 1.554; parameter shift/e.s.d. < 0.04. Atomic coordinates for all non-H atoms are given in Table 1 and interatomic distances and angles are given in Table 2.* The molecular structure and atomic numbering

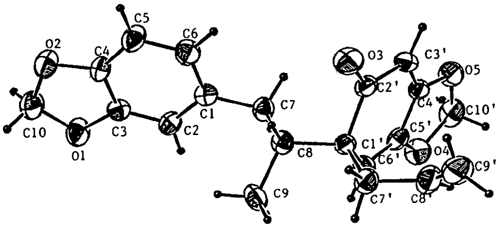

Fig. 1. *ORTEP*II (Johnson, 1976) view showing the relative stereochemistry of the molecule. Thermal ellipsoids are drawn at 35% probability.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55682 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GR0196]

scheme are shown in the *ORTEP*II (Johnson, 1976) drawing in Fig. 1.

Related literature. The isolation of Δ^8-1',2'-dihydro-3,4,3',4'-bismethylenedioxy-2'-oxo-8.1-neolignan has been reported, along with a partial structure assignment based on NMR data (Green & Wiemer, 1991). For a description of other work on the chemistry of *Piper capense* roots, see Green, Galinis & Wiemer (1991).

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1,1,4-Trimethylsilacyclohexan-4-ol

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Abstract. $C_8H_{18}OSi$, $M_r = 158.32$, monoclinic, $C2/c$, $a = 40.88\,(1)$, $b = 6.733\,(4)$, $c = 30.16\,(1)$ Å, $\beta = 132.08\,(3)°$, $V = 6161$ Å^3, $Z = 24$, $D_x = 1.02$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 1.68$ cm^{-1}, $F(000) = 2112$, $T = 298$ K, $R = 0.059$, $wR = 0.049$ and $S = 1.71$ for 4194 observed reflections. The three independent molecules, two in the more stable conformation with the OH axial and the third with the OH equatorial, form an infinite hydrogen-bonded helix.

Experimental. A colorless prism, 0.20 × 0.25 × 0.30 mm, was obtained by slow evaporation from methanol, and used for data collection on an Enraf–Nonius CAD-4 diffractometer with Mo $K\alpha$ radiation and ω–2θ scans to $2\theta_{\max} = 53°$. 25 reflections with $20 < 2\theta < 26°$ were used for unit-cell determination. Space group was determined from systematic absences (hkl, $h + k$ odd; $h0l$, l odd) and successful refinement. Three standard reflections measured after every 7200 s of X-ray exposure showed no deterioration. For $-49 < h < 38$, $0 < k < 8$, $0 < l < 37$, no redundant data were collected; a total of 6368 unique reflections were measured with 4149 observed [$I > 2\sigma(I)$]. Data were corrected for Lorentz and polarization effects but not for absorption. The structure was solved by direct methods (Sheldrick, 1985). H atoms were located on difference Fourier maps and refined with isotropic thermal parameters; non-H atoms were refined with anisotropic thermal parameters. Refinement was on F; function minimized during refinement was $\sum w(|F_o| - |F_c|)^2$, with $w = 4F_o^2[\sigma^2(I) + (0.04F_o^2)^2]^{-1}$. $R = 0.059$, $wR = 0.049$ and $S = 1.71$ for the observed data and 487 parameters; $(\Delta/\sigma)_{\max}$ in final cycle was less than 2%; the final difference map showed maxima of ± 0.2 e Å^{-3}. Atomic scattering factors and anomalous-dispersion corrections were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were performed with *SHELX*76 (Sheldrick, 1976).

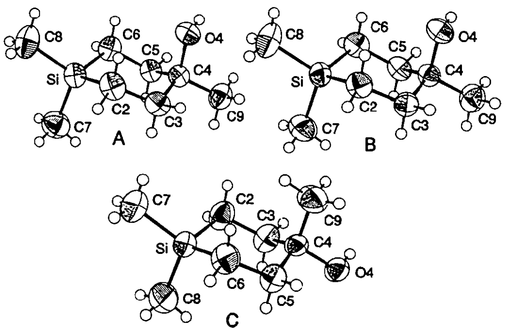

Fig. 1. Perspective view of the three independent molecules with numbering scheme. Thermal ellipsoids are drawn at the 50% probability level.

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